Final report submitted to:

National Defense Center of Excellence for Research in Ocean Sciences (CEROS)
73-4460 Queen Ka'ahumanu Highway
Kailua Kona HI 96740 USA

CEROS Contract No. 58631
Award Amount: $382,542
Contract Period: October 1st, 2009 – September 30th, 2011
Date Submitted: December 30th, 2011

Improved Methods of Chemical Decontamination of Navy Assets and Infrastructure

Dr. Garry Edgington, PhD, Chief Scientist, CBI Polymers, Inc. and Principal Investigator

Dr. Andreas Mylonakis, Senior Scientist, CBI Polymers, Inc.

Mr. Michael Coy, Program Manager, CBI Polymers, Inc.

CBI Polymers, Inc.
1946 Young Street, Suite 288
Honolulu, Hawaii 96826
December 2011

REPORT DOCUMENTATION PAGE	Form Approved OMB NO. 0704-0188

Public Reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comment regarding this burden estimates or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188,) Washington, DC 20503.

1. AGENCY USE ONLY (Leave Blank)	2. REPORT DATE December 31, 2011	3. REPORT TYPE AND DATES COVERED Final Report, October 1st, 2009 – September 30th, 2011

4. TITLE AND SUBTITLE **Improved Methods of Chemical Decontamination of Navy Assets and Infrastructure**	5. FUNDING NUMBERS CEROS Contract No. 58631, Funding amount $382,542
6. AUTHOR(S) Dr. Garry Edgington, PhD, Principal Investigator; Dr. Andreas Mylonakis, Senior Scientist and Mr. Michael Coy, Program Manager	
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) CBI Polymers, Inc. 1946 Young Street, Suite 288 Honolulu, HI 96826	8. PERFORMING ORGANIZATION REPORT NUMBER
9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES) National Defense Center of Excellence for Research in Ocean Sciences (CEROS), 73-4460 Queen Ka'ahumanu Highway, Kailua Kona, HI 96740 USA	10. SPONSORING / MONITORING AGENCY REPORT NUMBER N/A

11. SUPPLEMENTARY NOTES The views, opinions and/or findings contained in this report are those of the author(s) and should not be construed as an official Department of the Army position, policy or decision, unless so designated by other documentation.

12 a. DISTRIBUTION / AVAILABILITY STATEMENT Approved for public release; distribution unlimited.	12 b. DISTRIBUTION CODE

13. ABSTRACT (Maximum 200 words)

The Principal Investigator was tasked with determining if a commercial product, designed for radioactive isotope decontamination, could be used to decontaminate a minimum of 25 general Toxic Industrial Chemicals (TICs) on various substrates, and use the information and results gleaned to solve 12 Navy specific decontamination needs, and validate, via field testing, the performance goal of >25% cost reduction for 3 of the 12 solutions for Navy specific decontamination needs.

14. SUBJECT TERMS DeconGel, Decontamination, CEROS, Contamination, Environmental, Pearl Harbor, Naval Shipyard, Intermediate Maintenance Facility	15. NUMBER OF PAGES 608
	16. PRICE CODE

17. SECURITY CLASSIFICATION OR REPORT **UNCLASSIFIED**	18. SECURITY CLASSIFICATION ON THIS PAGE **UNCLASSIFIED**	19. SECURITY CLASSIFICATION OF ABSTRACT **UNCLASSIFIED**	20. LIMITATION OF ABSTRACT **UL**

Approximate Conversions from Metric to U.S. Customary Measures

Length

Symbol	When You Know	Multiply By	To Find	Symbol
mm	millimeters	0.04	inches	in
cm	centimeters	0.4	inches	in
m	meters	3.3	feet	ft
m	meters	1.1	yards	yd
km	kilometers	0.6	miles	mi

Area

Symbol	When You Know	Multiply By	To Find	Symbol
cm^2	square centimeters	0.16	square inches	in^2
m^2	square meters	1.2	square yards	yd^2
km^2	square kilometers	0.4	square miles	mi^2
ha	Hectares (10,000 m^2)	2.5	acres	

Mass (weight)

Symbol	When You Know	Multiply By	To Find	Symbol
g	grams	0.035	ounces	oz
kg	kilograms	2.2	pounds	lb
	metric ton (1,000 kg)	1.1	short tons	

Volume

Symbol	When You Know	Multiply By	To Find	Symbol
mL	Milliliters	0.03	fluid ounces	fl oz
mL	Milliliters	0.06	cubic inches	in^3
L	Liters	2.1	pints	pt

L	Liters	1.06	quarts	qt
L	Liters	0.26	gallons	gal
m^3	cubic meters	35	cubic feet	ft^3
m^3	cubic meters	1.3	cubic yards	yd^3

Temperature (exact)

Symbol	When You Know	Multiply By	To Find	Symbol
°C	degrees Celsius	9/5, add 32	degrees Fahrenheit	°F
K	Kelvin	subtract 273.15	degrees Celsius	°C

Preface

This report provides data on a technology developed for a specific application and modifying it to meet another application. As background the Navy faces many environmental challenges due to the wide range of industrial operations that are performed within its sites. The diversity of combinations of contaminant, substrate type and substrate configuration result in numerous complex and difficult to decontaminate scenarios.

Many standard cleaning methods currently used by the Navy utilize power washing, vacuuming, water/detergent, solvents or hypochlorite solutions together with brushes, buckets, bags, swabs and rags. These cleaning methods often spread the contamination over a broader area. These commonly employed cleaning methods often require an extensive amount of time, labor and effort and are often inadequate to remove contamination located in cracks, crevices and porosity of common building materials such as concrete. Additionally the waste stream generated using standard techniques results in significant waste disposal costs for the Navy.

The Center of Excellence for Research in Ocean Sciences wanted to know if a commercialized radiological decontaminant solution could be used to mitigate the waste stream and impact on our water supplies and oceans by using this technology on a minimum of 25 general Toxic Industrial Chemicals (TICs) on various substrates. If successful, the technology was to be used to solve 12 Navy specific decontamination needs, and validate, via field testing, with the ultimate performance goal of a obtaining a >25% cost reduction for 3 of the 12 solutions for Navy specific decontamination needs.

The outcome of the tests indicated that significant cost savings, well in excess of the 25% goal desired. The testing indicated that significant savings can be obtained by adapting dual use technologies for purposes other than that which they were originally intended. The information presented shows that a commercial radiological decontaminant solution can be used as a cleaning mechanism for more general, yet vastly important, contamination problems affecting the Navy today. Adaption of this technology and used as illustrated can save the Department of Defense and Federal Government potentially hundreds of millions of dollars, while maintaining ecologically sound methods of decontamination and waste stream reduction.

Acknowledgements

The authors and the project team wish to acknowledge the following individuals and organizations for their assistance and encouragement in advancing the polymeric hydrogel technology described by this report.

In alphabetical order, and without academic credentials and/or military rank, they are listed below.

Defense Advanced Research Projects Agency (DARPA):
Duane Ashton, Andrew Coon, Deborah Furey, Ed Mihalak, and Larry Stotts.

National Defense Center of Excellence for Research in Ocean Sciences (CEROS):
Jacquie Brewbaker, Lee Fausak, Richard Hess, Alan Hilton, and Donna Mau.

U. S. Navy, Naval Sea Systems Command (NAVSEA):
William Bankhead, Kurt Doehnert and Jimmy Smith.

U. S. Navy, Naval Research Lab Center for Corrosion Science and Engineering:
Robert Brown and James Martin.

U. S. Navy, Pacific Fleet:
Michael Bresnan and R. D. Berkey.

U. S. Navy, Pearl Harbor Naval Shipyard and Intermediate Maintenance Facility (PHNSY & IMF):
Vicky Ah Quin, Nathan Lum, Randy Sawyer, Gail Shimazu, Robert Sonoda, and Vince Yokoyama.

Joint Program Executive Office for Chemical and Biological Defense; Joint Project Manager, Decontamination:

Mark Morgan, CSC Inc.

Table of Contents

List of Figures

List of Tables

Acronyms and Abbreviations

AAS	Atomic Absorbtion Spectroscopy
ASTM	American Society for Testing and Materials
BMP	Best Management Practices
CARC	Chemical Agent Resistance Coating
CBIP	CBI Polymers
CEES	Chloroethyl Ethyl Sulfide
CEPS	Chloroethyl Phenyl Sulfide
CEROS	National Defense Center of Excellence for Research in Ocean Sciences
CWC	Chemical Weapons Contamination
DARPA	Defense Advanced Research Projects Agency
DDT	Dichloro Diphenyltrichloroethane
DE	Decontamination Efficacy
DG	DeconGel
DOE	Department of Energy
EPA	US Environmental Protection Agency
FLAA	Flame Atomic Absorbtion
GC-MS	Gas Chromatography-Mass Spectroscopy
HPLC	High Performance Liquid Chromatography
ICP-OES	Inductively Coupled Plasma-Optical Emission Spectroscopy
JPEO-CBD	Joint Program Executive Office for Chemical and Biological Defense
LC-MS	Liquid Chromatography-Mass Spectroscopy
LOD	Limit of Detection
MBz	Methyl Benzoate
MS	Mass Spectrometry
N/A	Not Applicable
NAVSEA	Naval Sea Systems Command
ND	Not Determined
NPDES	National Pollutant Discharge Elimination System
OSHA	Occupational Safety and Health Administration
PAHS	Polyaromatic Hydrocarbons
PCBs	Polychlorinated Biphenyls
PEL	Permissible Exposure Limits
PHNSY	Pearl Harbor Naval Shipyard and Intermediate Maintenance Facility
PLM	Polarized Light Microscopy
PPB	Parts per Billion
PPM	Parts per Million
PPT	Parts per Trillion
RT	Room Temperature
SD	Standard Deviation
TATRC	Telemedicine and Advanced Technology Research Center
TBI	Tributyl Tin Compounds
TEM	Transmission Electron Microscopy
TIC	Toxic Industrial Chemical
TIM	Toxic Industrial Material
TS	Thermospray
TWA	Time Weighted Average

EXECUTIVE SUMMARY

The Navy faces many environmental challenges due to the wide range of industrial operations that are performed within its sites. During naval shipyard operations of vessel maintenance, repair and building, a wide variety of metallic, inorganic and organic toxic chemicals may be employed which can contaminate immediate work areas comprising a multitude of substrates, including concrete, metal, plastic, painted surfaces, etc., that come in different sizes, shapes and configurations. The diversity of combinations of contaminant, substrate type and substrate configuration result in numerous complex and difficult to decontaminate scenarios.

Many standard cleaning methods currently used by the Navy utilize power washing, vacuuming, water/detergent, solvents or hypochlorite solutions together with brushes, buckets, bags, swabs and rags. The detergent/water cleaning method, which is a broadly used cleaning method, utilizes significant amounts of water and absorbents. These cleaning methods often spread the contamination over a broader area. The large amount of waste water containing the toxic contaminants must be collected and disposed of properly, which incurs significant costs across the services, otherwise cross contamination and run off of toxins into surface water, ground water and soil may occur. These commonly employed cleaning methods often require an extensive amount of time, labor and effort and are often inadequate to remove contamination located in cracks, crevices and porosity of common building materials such as concrete.

CBI Polymers, Inc. (CBIP) has developed and commercialized a safe, non-caustic, user-friendly, polymer-based hydrogel that can be applied via spray, brush, or roller that dries to a tough film, allowing for peel-off removal of radioactive contamination from a broad spectrum of hard surfaces.

During this project, CBIP leveraged both its present intellectual property and the technical capability of DeconGel™ (DG) in the development of improved methods of decontamination that dissolve, incorporate and/or encapsulate the broad spectrum of organic, inorganic and metallic, hazardous TICs and particulates that are common contaminants in Navy assets and infrastructure.

The technical objectives of this program included:

1. Evaluate the decontamination efficacy of DG formulations on a minimum of 25 general TICs on various substrates.
2. Solve 12 Navy specific decontamination needs, i.e., 12 PHNSY/NAVSEA specific contaminant/substrate combinations.
3. Validate, via field testing, the performance goal of a >25% cost reduction for 3 of the 12 solutions for the Navy specific decontamination needs.

In this Project, CBIP developed decontamination methods for 37 TIC classes on multiple substrates, including 12 Toxic Industrial Chemical classes (TICs) selected by the Navy. 172 TIC/substrate combinations were evaluated with 145 TIC/substrate combinations having >95% decontamination efficacy (DE) values (% reduction in contaminant concentration).

These evaluations have been performed on a variety of substrates including industrial grade concrete, aluminum, stainless steel, carbon steel, linoleum tile, glass and painted surfaces. Decontamination efficacy values have been determined via both residue swipe analysis method (analysis and comparison of residue swipes before and after decontamination) and direct analysis of contaminants encapsulated in DG products or other hydrogel formulations.

Highlights of decontamination efficacy (DE) values (% reduction in contaminant concentration) by utilizing DG products for the wide range of TICs and chemicals classes of concern include: 99.8%+ efficacy against arsenic compounds, 97.6%+ efficacy against cadmium compounds, 98.2%+ efficacy against cyanide compounds, 99.4%+ efficacy against industrial solvents (m-cresol), 99.8%+ efficacy against mercury compounds, 99.5%+ efficacy against aromatic liquids (toluene), 99.2%+ efficacy against aromatic solids (naphthalene), 98.5%+ efficacy against iodine compounds and 100% efficacy against amine compounds (aniline) on a variety of substrates (including stainless steel, aluminum and industrial grade concrete) as determined by residue swipe analysis. Swipe sampling methods are used in most industrial standard practices for the evaluation of the contamination (powder, dust or liquid) level on a substrate.

99.8%+ efficacy against arsenic compounds, 97.6%+ efficacy against cadmium compounds, 98.2%+ efficacy against cyanide compounds, 99.4%+ efficacy against industrial solvents (m-cresol), 99.8%+ efficacy against mercury compounds, 99.5%+ efficacy against aromatic liquids (toluene), 99.2%+ efficacy against aromatic solids (naphthalene), 98.5%+ efficacy against iodine compounds and 100% efficacy against amine compounds (aniline)

A second major objective of this contract included the evaluation of the decontamination efficacy of DG products and other hydrogel formulations on 12 PHNSY/NAVSEA specific contaminant/substrate combinations. Highlights of the decontamination efficacy demonstrated by the use of the peelable hydrogels and methods developed under this contract against Navy priority contamination problems include DE values (%) against polychlorinated biphenyls (PCBs) oil (~100%), 99%+ efficacy against a variety of copper compounds, 98%+ efficacy against a variety of lead compounds, 100% efficacy against asbestos contamination (independent lab testing results), ~100% efficacy against paint dust containing tributyl tin compounds, 96.3-99.2% DE on metal against multiple chemical warfare agents simulants and 98%+ efficacy against chromium compounds on a variety of substrates.

The final objective included field testing and further validation of the decontamination methods developed under this contract with the objective of achieving ≥25% cost reduction for three (3) of the twelve (12) PHNSY/NAVSEA priority chemical contaminant/substrate combinations. When feasible, cost analysis comparing the current method utilized by the Navy and CBIP's hydrogel technology was performed by the PHNSY&IMF end-users evaluating the overall cost of the current Navy cleaning practices and the methods developed under this contract.

Outstanding efficacy against polychlorinated biphenyl (PCB) contamination was achieved in a field test aboard the USS Missouri. For all contaminated surfaces tested, EPA PCB limit standards (Regulations 761.79, 761.123) of ≤10ug/100cm^2 for unrestricted public access were achieved upon decontamination with DG 1102; this formulation was partially developed under a previous CEROS/DARPA contract and has been optimized for an affinity towards hydrophobic contaminants.

This cost model comparison estimated a 73% reduction in cost when DG is utilized to decontaminate surfaces contaminated with PCBs compared to current standard methods.

The second field test was performed at the PHNSY&IMF Building 6 Foundry Complex on a large open area composed of a variety of surfaces including a smelter, a control panel, bare and painted concrete, bare and painted metal and glass windows that were heavily contaminated with lead dust.

An estimated 73% reduction in cost can be obtained when DG is utilized to decontaminate surfaces contaminated with PCBs compared to current standard methods.

We found that while there are standards set by OSHA (Occupational Safety and Health Administration) for the amount of lead dust workers are allowed to be exposed to in the air, there are no legal standards for the lead content of surface dust in the workplace. However, OSHA, an agency that protects workers, requires that wipe samples collected on surfaces in eating areas in workplaces not exceed 200 micrograms/square foot (μg/ft^2). PHNSY&IMF agreed that this limit is more stringent than that required for remediation of Building 6 and that this limit could be used as a conservative estimated limit for the purpose of this field test intended to determine the suitability of using DG in the decontamination of lead dust in building 6.

DG formulations (both sprayable and non-sprayable versions were evaluated) achieved excellent surface decontamination efficacy against lead dust. Decontamination Efficacies (DEs) were ≥85% for most of the areas tested. With the exception of the smelting equipment that is planned to be removed from the building, all other areas decontaminated had average post decontamination lead levels below the 200 μg/ft^2 specification.

For the third field test, Chemical Warfare Agent (CWA) simulant decontamination testing performed in house under the direction of the Joint Program Executive Office for Chemical and Biological Defense (JPEO-CBD) resulted in the JPEO-CBD performing testing of DG on classified live CWAs at their facility at their own expense. JPEO-CBD informed us that DG performed exceptionally well as a physical decontamination method for classified emerging threat agents on sorbent substrates such as rubber and Chemical Agent Resistant Coating

(CARC). Although the efficacy of the current DG formulation was not sufficient to be a total decontamination solution, they recommended that we combine CWA neutralization technologies with the ability of DG to draw CWAs out of sorbent substrates and emulsify them, both of which increase the access of chemical neutralizers to the CWA.

Thirty-seven individual and detailed end-user reports presenting efficacy data, application instructions and tips for optimum decontamination efficacies have been prepared for all combinations of TICs and substrates evaluated in this project and are included in this report and available to PHNSY&IMF, NAVSEA and civilian end-users.

Recommendations and Conclusions

Because of the length of this report recommendations and conclusions will be presented here for readability. The project conclusively showed that the DeconGel technology can safely decontaminate radiological contamination, and clean hazardous and non-hazardous chemicals of concern.

In an era of drastically streamlined budgets and cuts in defense funding, one area for simple, but significant savings is in waste stream remediation and labor cost savings for ongoing and contemplated cleanup, or remediation/decontamination for base reduction and closure efforts. DeconGel has been shown, both in this study and in actual commercial use, to drastically lower total cost of ownership upwards of 70% and reduce waste stream generation upwards of 80%. The savings are immediate, easily recognizable, and defendable in budget hearings and contract negotiations. The technology requires absolute minimal investment in training and upkeep, and can be touted by the services as part of their responsibilities towards reducing the impacts to the environment in accordance with Executive Order 15341 dated October 2009. Therefore, the authors recommend that:

1. The Secretaries of the military services commission a study regarding concepts of operations scenarios using DeconGel that would reduce overall costs in environmental projects and programs within the services.

2. Maintain a supply of DeconGel onboard every naval vessel and military facility to mitigate common chemical spills to include radioactive isotope contamination as experienced by several ships that participated in Operation Tomodachi in Japan.

3. The military consider the use of the DeconGel technology in areas where military assets have been closed due to contamination such as lead and asbestos. An example would be Building 6 (closed Lead Foundary) at Pearl Harbor Naval Shipyard, Hawaii.

4. The DeconGel technology be considered for every spill and hazmat kit within the Department of Defense to mitigate human health hazards associated with unknown chemical spills.

5. Every DoD hospital in which radiation treatment is used should maintain a supply of DeconGel technology for radioactive, heavy metal or chemical decontamination.

1. INTRODUCTION

1.1 BACKGROUND

CBI Polymers, Inc. (CBIP) has developed, commercialized and is selling a safe, non-caustic, user-friendly, polymer-based hydrogel that can be applied via spray, brush, or roller, and dries to a tough film, allowing for peel-off removal of radioactive contamination from a broad spectrum of hard surfaces. Commercialization of DeconGel (DG) 1101 commenced in September 2007 with an initial market focus on Nuclear Decontamination and Decommissioning including DOE Super Fund sites, nuclear power plant maintenance, nuclear medicine decontamination as well as research and development laboratories. Several variations of the original DG products have since been developed including DG 1102 a formulation partially developed under a previous CEROS/DARPA contract that has been optimized for an affinity towards hydrophobic contaminants. CBIP has also set up warehousing, repackaging and distribution operations with TEC, Inc. in Avon, Ohio.

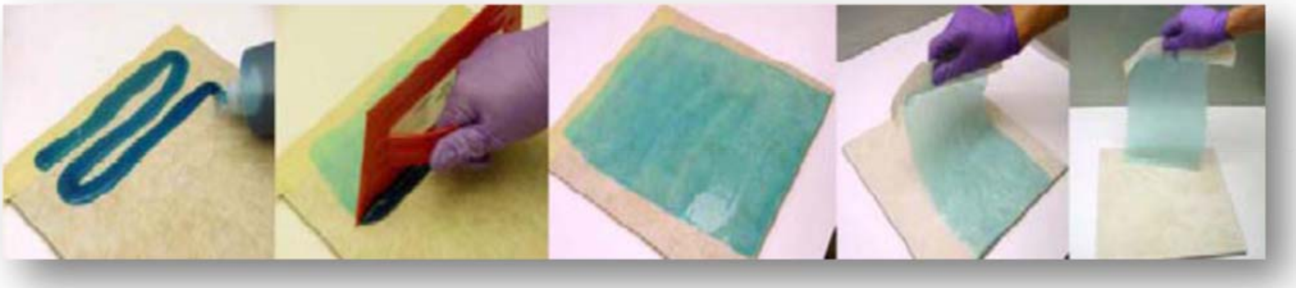


Figure 1. Hydrogel technology for decontamination: Apply, let Dry and Peel.

Pearl Harbor Naval Shipyard and Intermediate Maintenance Facility (PHNSY&IMF or PHNSY) is charged with fleet maintenance, repair, refurbishment, and decommissioning of Navy vessels. Navy shipyards and the Navy in general face many environmental challenges due to the wide range of industrial operations that are performed within their sites. During naval shipyard operations of vessel maintenance, repair and building, a wide variety of metallic, inorganic and organic toxic chemicals may be employed which can contaminate immediate work areas comprising a multitude of substrates, including concrete, metal, plastic, painted surfaces, etc., that come in different sizes, shapes and configurations.

Figure 2. Navy shipyards and the Navy face many environmental challenges due to the wide range of industrial operations that are performed within their sites. Note the discoloration of the concrete caused by long-term contamination.

The diversity of combinations of contaminant, substrate type and substrate configuration result in numerous complex and difficult to decontaminate scenarios. Potential toxic and hazardous substance spills, generation of hazardous wastes and air emission could result in adverse impacts on human health and the environment. Naval shipyards are in need of improved innovative methods of surface decontamination with a focus on a reduction in man hours, contaminated waste, and lower total costs of ownership.

Previous efficacy evaluations had been confined almost exclusively to radiological contaminants. However, we expected that DG would be shown effective in the decontamination of a variety of chemical types.

Figure 3. Decontamination field test at Alaron Nuclear Services with DG showing radioactive isotope removal from a nuclear fuel cask container and areas of a shop floor.

Under this Contract, CBIP was charged to develop improved methods of chemical decontamination of U.S. Navy assets and infrastructure, with a goal of achieving cost reductions of >25% compared to current practices. This effort entailed collaboration with PHNSY personnel to evaluate DG products on (1) general classes of toxic industrial chemicals (TICs), and (2) on specific combinations of TICs and substrates that are present in Naval Shipyards.

2 TECHNICAL OBJECTIVES

2.1 Decontamination of a Range of Toxic Industrial Chemicals/Chemicals of Concern

The first major objective of this contract included the evaluation of DG products and/or improved methods on a minimum of 25 general Toxic Industrial Chemicals (TICs) and chemicals of concern on various substrates. There is an almost an infinite number of toxic and hazardous chemicals and chemicals of concern/substrate combinations that are present in the industrial setting. This objective comprised an in-depth evaluation of the decontamination efficacy on a wide range of chemical classes that cover, to a large extent, the broad spectrum of chemical contaminant classes and chemical classes of concern that can be found in the industrial setting.

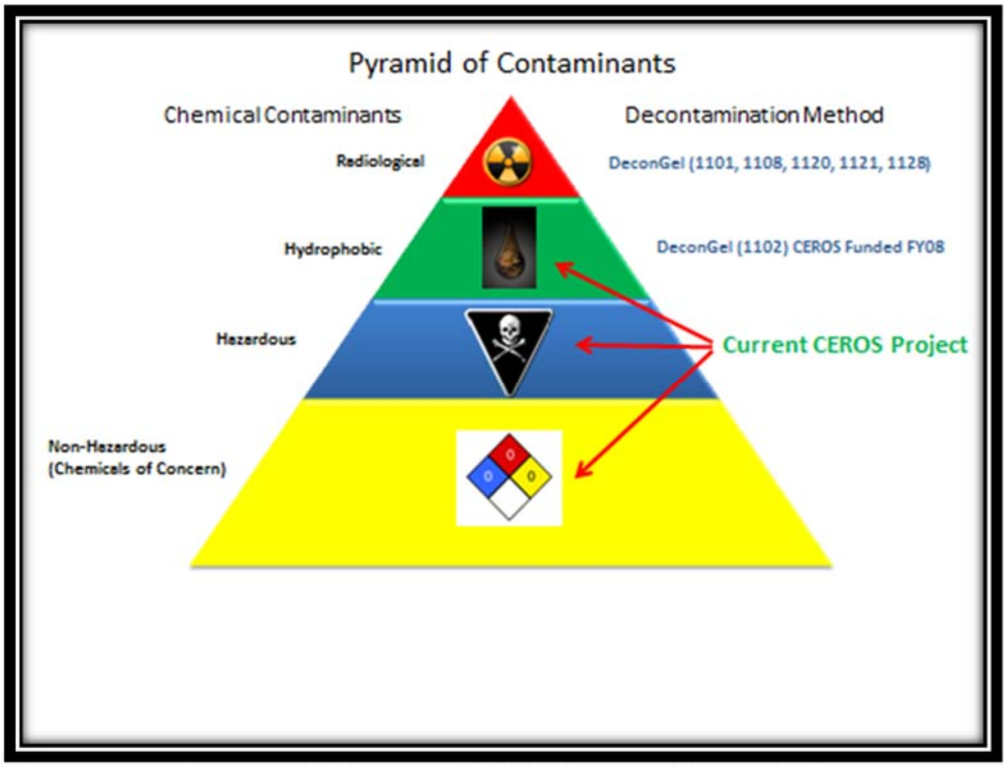


Figure 4. A broad range of Toxic Industrial Chemicals (TICs) and chemicals of concern that DG and/or improved methods have been evaluated on for their decontamination efficacy.

2.2 Decontamination of Navy Priority Contaminant/Substrate Combinations

The second major objective of this contract included the evaluation of DG products or other improved methods on 12 PHNSY/NAVSEA specific contaminant/substrate combinations. There are a significant number of toxic and hazardous chemicals and chemicals of concern/substrate combinations that are present in the naval setting. This objective comprised the evaluation of the decontamination efficacy on a relevant range of chemical classes that cover the spectrum of contaminant/substrate combinations commonly encountered in Navy shipyards, infrastructure and other areas (specifically identified by PHNSYandIMF and/or NAVSEA) and that required improved cost effective decontamination responses.

2.3 Testing and Further Validation of Performance Goals

The third major objective of this contract included field testing and further validation of the decontamination methods developed during the course of this contract towards the performance goal of ≥25% cost reduction for three (3) of the twelve (12) PHNSY/NAVSEA priority chemical contaminant/substrate combinations previously validated in laboratory testing.

3 METHODOLOGY

3.1 Laboratory Test Method

Detailed designs for in-house evaluations, on-site field testing, and independent laboratory testing employed during this project are presented in the sections below.

Analytical Instrumentation: Amongst others, analytical methods used to evaluate the efficacy of the formulations to remove the wide range of contaminants included Gas Chromatography-Mass Spectroscopy (GC-MS), Liquid Chromatography-Mass Spectroscopy (LC-MS), Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES), Mercury Vapor Analyzer, Transmission Electron Microscopy (TEM), Polarized Light Microscopy (PLM) and visual inspections/evaluations.

ICP-OES is used for qualitative and quantitative determination of metals and certain non-metals in solution. The liquid sample is nebulized into a strong magnetic field of plasma where the temperature is high enough to break chemical bonds, liberate elements present and transform them into the gaseous atomic state. Applications include environmental screening of water samples for contaminants such as lead, cadmium, mercury and zinc, analysis of biological samples for ions such as sodium and potassium and analysis of geological samples and minerals.

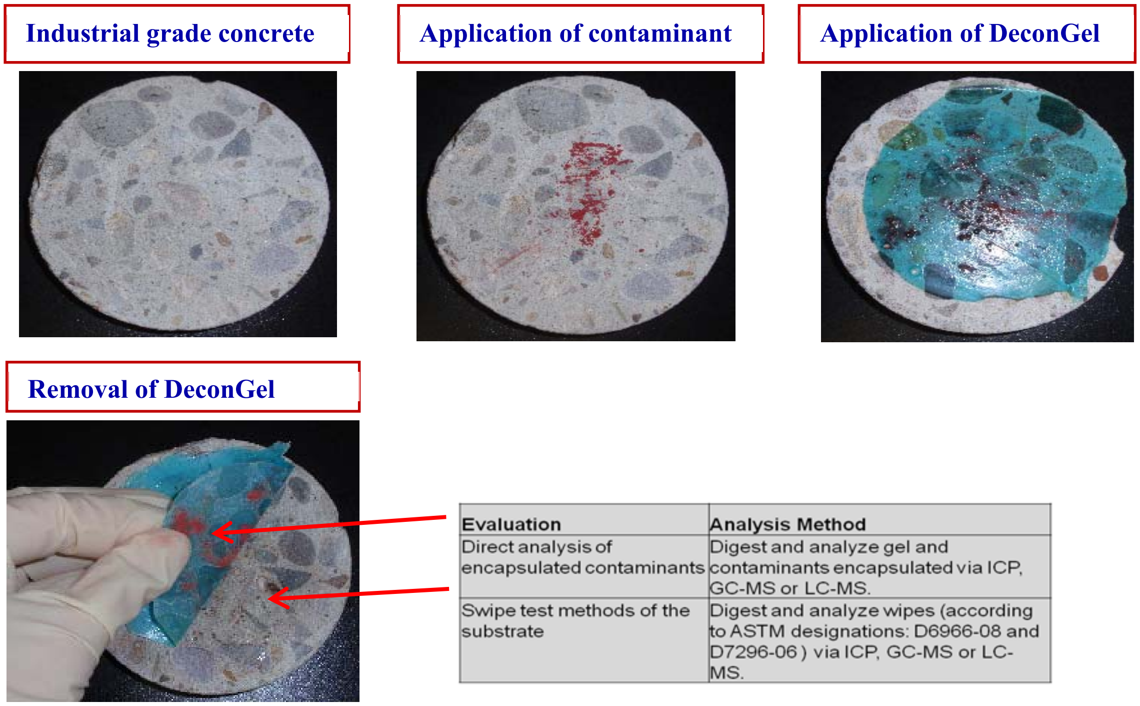
Gas chromatography-mass spectroscopy (GC-MS), as the name implies, is actually two techniques that are combined to form a single method of analyzing mixtures of chemicals. Gas chromatography separates the components of a mixture and mass spectroscopy characterizes each of the components individually. By combining the two techniques, an analytical chemist can both qualitatively and quantitatively evaluate a solution containing a wide variety of different chemicals. The uses for GC-MS are numerous in the medical, pharmacological, environmental, and law enforcement fields.

Liquid chromatography-mass spectrometry (LC-MS) is an analytical chemistry technique that combines the physical separation capabilities of liquid chromatography with the mass analysis capabilities of mass spectrometry. LC-MS is a powerful technique used for many applications that require high sensitivity and specificity.

This instrumentation has been acquired by CBI Polymers through DOE funding and was leveraged to complete this work without affecting the CEROS funding budget. Specific technical details are given for each individual contaminant analyzed via the ICP-OES, GC-MS or LC-MS.

Method of Application of the Contaminant: A known amount of a specific contaminant of interest was applied on a specific substrate (either by pouring or by brushing the contaminant). In some cases, depending on the contaminant, drops of methanol (or other solvent) were applied on top of the contamination and allowed to dry, in order to represent a contaminant deposit more likely to be found in a “real-world” contamination scenario.

For evaluation of DG products or improved methods against liquid contaminants, contaminants were applied on top of the contaminated surface with a brush. Application of a uniform thin layer of contaminant on the respective substrate facilitated an optimized interaction between contaminant and DG, and provided an accurate measure of DG's decontamination efficacy. Specific details on the application of the contamination and application/removal of the gel are given for each specific compound in the individual detailed technical reports that can be found in the Appendix sections of this report. Triplicate runs were performed for each substrate/contaminant combination.



Evaluation	Analysis Method
Direct analysis of encapsulated contaminants	Digest and analyze gel and contaminants encapsulated via ICP, GC-MS or LC-MS.
Swipe test methods of the substrate	Digest and analyze wipes (according to ASTM designations: D6966-08 and D7296-06) via ICP, GC-MS or LC-MS.

Figure 5. Application of contamination on the substrate of interest, application of gel, removal of the dried gel followed by the method of analysis.

Application of Hydrogel Formulation: A specific amount of DG or other hydrogel formulation was poured on top of the contamination.

Gel Drying Time: Unless specified the hydrogel formulations were left to dry for 24 hours.[1]

---

[1] Some contaminants might prolong the drying time of the gel.

Digestion of Samples: Samples that contain compounds that cannot be dissolved in a solvent (elemental metals or other metallic compounds) were digested according to EPA method-3010A (acid digestion of aqueous samples and extracts for total metals for analysis by FLAA or ICP spectroscopy) or a variation thereof. Variations may include a change in acid type and/or concentration as well as digestion temperature adjustments. For example, barium chloride (BaCl2) is soluble in water (as is DG) and requires only a small amount of acid to digest, which can occur at room temperature. Barium sulfate, on the other hand, in the presence of nitric acid, forms barium nitrate, which is insoluble. In this case, hydrochloric acid had to be used in place of nitric acid. Similar adjustments and variations of EPA method-3010A have been utilized for the analysis of other metallic compounds. To afford the complete digestion/dissolution of these inorganic contaminants a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC) was used. About 30 different digestion methods were developed throughout the duration of this contract for the analysis of the wide variety of metals, metalloids, inorganics, organo-metallics, organic compounds, pesticides and solvent classes that have been evaluated.

Substrates Evaluated: DG products and other hydrogel formulations were evaluated on a variety of substrates including: 1) commercial grade 304 stainless steel, 2) commercial grade carbon steel, 3) commercial grade aluminum, 4) commercial grade floor tile, and 5) industrial grade concrete. Unless specified, all substrates have been used as received with no further pretreatment before the application of the contamination. 1) commercial grade stainless steel (surface area: 56.3 cm^2), 2) commercial grade carbon steel (surface areas: 17.85 cm^2, 100 cm^2), 3) commercial grade aluminum (surface area: 56.3 cm^2), 4) commercial grade floor tile (surface area: 100 cm^2), 5) industrial grade concrete (surface area: 56.3 cm^2), 6) commercial grade composite tile (surface area: 48.8 cm^2), 7) commercial grade linoleum tile (surface areas: 10cm^2, 48.8 cm^2), 8) commercial grade porcelain tile (surface area: 48.8 cm^2), 9) painted drywall (surface area: 10cm^2), 10) cadmium-plated steel (surface area: 12.75 cm^2), 11) CARC (chemical agent resistant coating) (surface area: 19.63 cm^2), 12) industrial silicone rubber (surface area: 19.63 cm^2), 13) Lexan^{TM} polycarbonate (similar to plexi glass) and 14) industrial black rubber (surface area: 16.83 cm^2) coupons. Unless specified, all substrates were used as received with no further pretreatment before application of the contaminants.

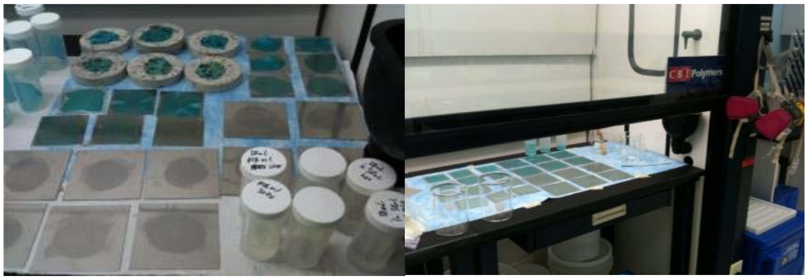


Figure 6. DG products and other hydrogel formulations have been evaluated on a variety of substrates.

Analytical Method Development: Analytical methods used to determine the decontamination efficacy of DG products or other hydrogel formulations on a wide range of TICs (or their simulants) and chemicals of concern included 1) Gas Chromatography-Mass Spectrometry (GC-MS), following standardized Environmental Protection Agency (EPA) SW-846 Analytical Method 8270C "Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) for organic TICs (or their simulants)/chemicals of concern, 2) Liquid Chromatography-Mass Spectrometry (LC-MS), following standardized EPA SW-846 Analytical Method 8321B "Solvent-Extractable Nonvolatile Compounds by High-Performance Liquid Chromatography/Thermospray/Mass Spectrometry (HPLC/TS/MS) or Ultraviolet (UV) Detection" for organic TICs (or their simulants)/chemicals of concern, and for inorganic TICs (or their simulants)/chemicals of concern, 3) Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES) following standardized EPA SW-846 Analytical Method 6010C "Inductively Coupled Plasma-Atomic Emission Spectrometry", 4) Mercury Vapor Analysis following Environmental Protection Agency (EPA) SW-846 Method 7471B: "Mercury in Solid or Semisolid Waste (Manual Cold-Vapor Technique)", and 5) Transmission Electron Microscopy following ASTM standard test method D6480-05 "Standard Test Method for Wipe Sampling of Surfaces, Indirect Preparation, and Analysis for Asbestos Structure Number Concentration by Transmission Electron Microscopy", and Polarized Light Microscopy following EPA/600/R-93/116 "Interim Method for the Determination of Asbestos in Bulk Insulation Sample".

Figure 7. DG products or other hydrogel formulations were evaluated against a wide range of toxic and hazardous industrial chemicals and chemicals of concern.

To ensure accurate determination of DG product's and other hydrogel formulation's decontamination efficacy against compounds that were to be analyzed via ICP-OES, calibration standards of the analyte of interest were prepared using either a sufficiently pure analyte or an appropriate ICP-MS Standard (Ricca Chemical Company; Arlington, TX); the respective standards were diluted to a known concentration (ppm) using the same digestion method as the one used for samples and controls. More details on the analytical method used for the evaluation of decontamination efficacy against metals and certain non-metals via ICP-OES are given in the detailed technical reports provided in the Appendices section.

To ensure accurate determination of DG product's or other hydrogel formulation's decontamination efficacies for organic contaminants via GC-MS and LC-MS, a standard curve of the analyte of interest was prepared using sufficiently pure analyte; the respective standards were diluted to a known concentration (ppm) using the same solvent as the one used for samples and controls.

3.2 Evaluation/Analysis of Contaminants

Method of Analysis: All samples and controls were run in triplicate. In a typical experimental procedure, contaminant was evenly applied using careful spreading with a spatula (solids) or brushing using a small paint brush (liquids) on coupons. Approximately 6.0 g of a DG (or other hydrogel formulation) was either poured (non-brushed) or brushed using a small paint brush onto the contaminated surface and let to dry for 24 h. Dried DG (or other formulation) samples were peeled off the contaminated surface, and the surface was swipe tested (see below). Swipe and gel samples were suspended in the appropriate solvent found to dissolve contaminants and gel film components for 24 h. Samples were then analyzed using the appropriate analytical method.

For Swipe Control samples, a respectable amount of contaminant was evenly applied via spreading or brushing and the surface was swipe tested using ASTM methods using GhostWipe™ swipes; depending on the type of contamination, one of the following ASTM standards were used: ASTM E1728-03: Standard Practice for Collection of Settled Dust Samples Using Wipe Sampling Methods for Subsequent Lead Determination or ASTM D6966-08: Standard practice for collection of settled dust samples using wipe sampling methods for subsequent determination of metals or ASTM 7296-06: Standard practice for collection of settled dust samples using dry wipe sampling methods for subsequent determination of beryllium and compounds or ASTM D6661-06: Standard Practice for Field Collection of Organic Compounds from Surfaces Using Wipe Sampling. Swipe controls were suspended in the appropriate solvent found to dissolve contaminants for 24 h. Controls were then analyzed using the appropriate analytical method.

For the hydrogel Control samples, a respectable amount of contaminant and approximately 6.0 g of dry hydrogel (pre-poured gel on the respective uncontaminated substrate and let to dry for 24 h) was suspended in the appropriate solvent found to dissolve contaminants and hydrogel film components for 24 h. Samples were then analyzed using the appropriate analytical method.

For organic contaminants, standardized EPA SW-846 Sampling Method 3500C "Organic Extraction and Sample Preparation" was used to prepare samples and controls. For inorganic contaminants, standardized EPA SW-846 Sampling Method 3005A, "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy" was used to prepare samples and controls. Additional details on the analytical method used for each contaminant class evaluated are provided in the individual technical reports for each chemical class.

Evaluation of Decontamination via Analysis of Swipe Samples: Wipe sampling methods are used in most industrial standard practices for the evaluation of the contamination (powder, dust or liquid) level on a substrate. This method of analysis presents the advantage of analyzing the contamination that is left on the substrate of interest directly after the removal of the gel, and thereby avoiding potential deviations when analyzing the gel with the encapsulated contaminants. This method consists of applying a specific amount of a contaminant on the substrate of interest, applying the gel on top, removing the gel, and swipe sampling the remaining contaminant on the surface via the standard swipe methods for metal sample collection.

A standardized swipe testing method (ASTM standard test method) was used to qualitatively and quantitatively analyze the residual surface contamination after decontamination of a contaminated surface by DG formulations. For solid contaminants, solvent-wetted GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes were used, whereas for liquid contaminants, air-dried GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes were utilized.

For analysis of the contaminant sampled, swipe samples and controls were suspended in the appropriate solvent that was to found to dissolve a specific contaminant usually for 24 hours. Samples and controls were then analyzed using the appropriate analytical method.

Decontamination Efficacy via swipe testing was calculated using the equation:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

Evaluation of Decontamination via Direct Analysis of Contaminant Encapsulated in Dried Gel: Direct chemical analysis of the dried peeled gel samples that contained the encapsulated contaminants was used to provide an improved understanding of decontamination efficacy. Dried DG films were digested utilizing an appropriate digestion method (refer to later sections of this report for more details) or dissolved in the appropriate solvent found to dissolve both contaminant and film components. This digestion/dissolution step would usually take 24 hours (unless otherwise specified). Samples were then analyzed using the appropriate analytical method.

Direct DG Decontamination Efficacy was calculated using the equation:

Decontamination Efficacy (DG Testing) =

(Contaminant (ppm) in DG Direct/Contaminant (ppm) in DG Control) x 100%

4 RESULTS AND DISCUSSION

4.1 Decontamination of a Range of Toxic Industrial Chemicals/Chemicals of Concern

The first major objective of this contract included the evaluation of DG products and other hydrogel formulations on a minimum of 25 general Toxic Industrial Chemicals (TICs) and chemicals of concern on various substrates.

A list of toxic and hazardous chemical classes that the gel has been evaluated on can be seen on Table 1. This table summarizes toxic, hazardous and general chemicals of concern that DG products and other hydrogel formulations have been evaluated on for their decontamination efficacy. These contaminants have a broad range of chemical and physical properties and are representative of the wide range of toxic and hazardous industrial chemicals. These chemicals present a contamination problem that requires an efficient and cost effective decontamination solution in the industrial setting. Due to shipping and handling regulations some of these chemicals presented some complexity to obtain for in-house evaluations. In addition, quantitative and qualitative evaluation for some of these chemicals revealed technical difficulties which restricted their further consideration for in-house evaluations. However, DG and other hydrogel formulations have been evaluated against more than the deliverable of 25 general TICs on various substrates.

Within the summarized list of toxic industrial chemicals and chemicals of concern below, PHNSYandIMF specifically identified some of these compounds as having an immediate need for an improved cost effective decontamination solution. Results on the evaluations of DG products against these classes of chemicals (specific PHNSY/NAVSEA contaminant substrate combinations) are given in later sections of this report.

Table 1. Broad range Toxic Industrial Chemicals (TICs) and chemicals of concern that DG products and other formulations have been evaluated on for their decontamination efficacy.

Chemical/ Contaminant Class[1]	Contaminant or Surrogate Tested[2]	Physical State	In-house Method of Analysis	OSHA Air Concentration Limits[3] Permissible Exposure Limits (PEL)
Acetic Acid	**Acetic acid**	Dilute solutions		PEL: 25µg/m^3 as a time weighted average concentration (TWA) for an 8hr work day
Acetonitrile	**Acetonitrile**	Liquid	GC-MS	PEL: 70µg/m^3 as a time weighted average concentration (TWA) for an 8hr work day
Asbestos	**Chrysotile**	Solid	ICP/SEM	
Arsenic & Arsenic Salts	**Arsenic trioxide, arsenic tribromide, and arsenic metal**	Solid	ICP-OES	PEL: 0.01µg/m^3 as a time weighted average concentration (TWA) for an 8hr work day
Ammonium Hydroxide	**Ammonium hydroxide**	Dilute solutions	Titration	
Amine Compounds	**Methyl amine**	Liquids	GC-MS	
Aniline	**Aniline**	Liquid	GC-MS, LC-MS	
Barium & Barium salts	**Barium elemental, barium sulfate, barium carbonate, barium chloride**	Solids	ICP-OES	PEL: 0.5µg/m^3 as a time weighted average concentration (TWA) for an 8hr work day
Benzene	**Benzene**	Liquid	GC-MS	PEL: 1µg/m^3 as a time weighted average concentration (TWA) for an 8hr work day
Benzoic acid	**Benzoic acid**	Solid	GC-MS	
Beryllium and Beryllium compounds	**Aluminum fine powder, aluminum chloride, aluminum carbide**	Fine powders	ICP-OES	PEL: 0.5µg/m^3 as a time weighted average concentration (TWA) for an 8hr work day. Applies to housekeeping limit of 3 ug/100 cm2 during non-operational periods (10 CFR 850.30). Applies to limits of 0.2 ug/100 cm2 for release to general public and 3 ug/100 cm2 for release to other beryllium facilities (10 CFR 850.31).
Carbon (activated)	**Carbon (activated)**	Fine powder	(visual)	PEL: 3.5µg/m^3 as a time weighted average concentration (TWA) for an 8hr work day
Cadmium & Cadmium compounds	**Cadmium oxide, cadmium metal, cadmium chloride**	Solids	ICP-OES	PEL: 0.2µg/m^3 as a time weighted average concentration (TWA) for an 8hr work day
Catechol	**Catechol**	Solid	GC-MS	PEL (NIOSH): 20µg/m^3 as a time weighted average concentration (TWA) for an 8hr work day
Chromium (VI valence state)	**Chromic acid (chromium trioxide), potassium dichromate**	Black/Red crystals	ICP-OES	PEL (NIOSH): 0.1µg/m^3 as a time weighted average concentration (TWA) for an 8hr work day

Chromium	**Chromium elemental**	Solid	ICP-OES	PEL (NIOSH): 1µg/m^3 as a time weighted average concentration (TWA) for an 8hr work day
Cyanide	**(Ferrocyanide (low toxicity))/Prussian Blue**	Powder	ICP-OES	
Chlorides	**Sodium chloride, calcium chloride, magnesium chloride**	Salts	ICP-OES/ Titration	
Copper	**Copper elemental, cuprous (I) oxide**	Metal flakes	ICP-OES	PEL: 1µg/m^3 as a time weighted average concentration (TWA) for an 8hr work day
Chlorinated HCs	**Trichloroethylene, tetrachloroethylene**	Liquids	GC-MS, LC-MS	
(DDT)	**Dichlorodiphenyltrichloroethane**	Solid	GC-MS, LC-MS	
2,4-Dichlorophenoxy-Acetic Acid	**2,4-Dichloro phenoxyacetic acid**	Solid	GC-MS	
Ethylene Glycol (Antifreeze)	**Ethylene glycol**	Viscous liquid	GC_MS	No OSHA or NIOSH limit
Formaldehyde	**Aqueous solution of formaldehyde**	Liquid	GC-MS, HPLC	PEL: 1µg/m^3 as a time weighted average concentration (TWA) for an 8hr work day
Hexanes	**Hexanes**	Liquid	GC-MS, HPLC	
Hydriodic Acid	**Hydriodic Acid (aqueous solution)**	Aqueous solution	ICP-OES	
Hydroquinone	**Hydroquinone**	Solid	GC-MS	PEL: 2µg/m^3 as a time weighted average concentration (TWA) for an 8hr work day
Iodine	**Iodine metal, potassium iodide**	Solids	ICP-OES	PEL: 1µg/m^3 as a time weighted average concentration (TWA) for an 8hr work day
Lead	**Lead elemental**	Solids	ICP-OES	PEL: 50µg/m^3 as a time weighted average concentration (TWA) for an 8hr work day
Leaded Dust	**Lead elemental, lead oxide**	Solids	ICP-OES	PEL: 50µg/m^3 as a time weighted average concentration (TWA) for an 8hr work day
Lead Based Paint Chips	**Lead oxide, (lead based paints), (lead paint chips)**	Solids	ICP-OES	PEL: 50µg/m^3 as a time weighted average concentration (TWA) for an 8hr work day
Lye (NaOH or KOH)	**Sodium hydroxide flakes/coarse**	Flakes	Titration	
Mercury Elemental	**Mercury elemental**	Liquid	ICP-OES	PEL: 0.1µg/m^3 as a time weighted average concentration (TWA) for an 8hr work day
Mercury Compounds	**Mercurous(I) chloride, mercury (II) oxide, mercury(II) sulfide**	Solids	ICP-OES	PEL: 0.01µg/m^3 as a time weighted average concentration (TWA) for an 8hr work day
Methyl-ethyl ketone	**Methyl-ethyl ketone**	Liquid	GC-MS, LC-MS	
Naphthalene	**Naphthalene**	Solid	GC-MS	

Nicotine	**Nicotine**	Solid	GC-MS	PEL: 0.5µg/m^3 as a time weighted average concentration (TWA) for an 8hr work day
Nickel & Nickel Salts	**Nickel elemental, nickel sulfate**	Solids	ICP-OES	PEL: 1µg/m^3 as a time weighted average concentration (TWA) for an 8hr work day
Nitric acid	**Nitric acid**	Liquid	Titration	PEL: 5µg/m^3 as a time weighted average concentration (TWA) for an 8hr work day
Paraquat	**Dichloride paraquat**	Liquid	GC-MS	PEL: 0.5µg/m^3 as a time weighted average concentration (TWA) for an 8hr work day
Petroleum Distillates	**Mineral spirits, kerosene, naphtha**	Liquids	GC-MS	PEL: 2000µg/m^3 as a time weighted average concentration (TWA) for an 8hr work day
PCB oil	**Mineral oil**	Viscous liquid	GC-MS, LC-MS	PEL: 0.5µg/m^3 as a time weighted average concentration (TWA) for an 8hr work day
Phosphorus Pentachloride	**Phosphorus pentachloride**	Solid	ICP-OES	
Selenium and Selenium compounds	**Selenium disulfide, selenium elemental**	Solid	ICP-OES	PEL: 0.2µg/m^3 as a time weighted average concentration (TWA) for an 8hr work day
Tributyl Tin Compounds	**TBT oxide, TBT chloride, TBT hydride**	Liquid	ICP-OES	PEL: 0.1mg/m^3 as a TWA for up to a 10 hour/day, 40 hour per week maximum.
Zinc	**Zinc elemental**	Solid	ICP-OES	PEL: 5mg/m^3 as a TWA for up to a 10 hour/day, 40 hour per week maximum.
Zinc Compounds	**Zinc oxide, zinc chloride, zinc acetate, zinc sulfide**	Solids	ICP-OES	PEL: 5mg/m^3 as a TWA for up to a 10 hour/day, 40 hours per week maximum.

1. Chemical compounds or class of compounds that are representative of toxic/hazardous chemicals or chemicals of concern.
2. Surrogates of toxic chemicals are used when chemical of interest is regulated or presents a hazard or in-house lab evaluations.
3. Air concentrations OSHA limits when available.

Decontamination Efficacy: DG products showed remarkable decontamination efficacies (% reduction in contaminant concentration) for the majority of the contaminant classes evaluated.

Highlights of decontamination efficacy (DE) values (% reduction in contaminant concentration) by utilizing this decontamination hydrogel technology for the wide range of TICs and chemicals classes of concern include 99.8%+ efficacy against arsenic compounds, 97.6%+ efficacy against cadmium compounds, 98.2%+ efficacy against cyanide compounds, 99.4%+ efficacy against industrial solvents (m-cresol), 99.8%+ efficacy against mercury compounds, 99.5%+ efficacy against aromatic liquids (toluene), 99.2%+ efficacy against aromatic solids (naphthalene), 98.5%+ efficacy against iodine compounds and 100% efficacy against amine compounds (aniline) on a variety of substrates (including stainless steel, aluminum and industrial grade concrete) as determined by residue swipe analysis. Swipe sampling methods are used in most industrial standard practices for the evaluation of the contamination (powder, dust or liquid) level on a substrate.

In particular, for non-porous surfaces (including stainless steel, carbon steel and aluminum), DG products showed DE of 99%+ for 21 out of 34 contaminants evaluated, DE of 97%+ for 8 out of 34 contaminants and DE of 95%+ for 3 out of 34 contaminants.

For porous surfaces (industrial grade concrete) DG products showed remarkable DE of 99%+ for 13 out of 34 contaminants evaluated, DE of 97%+ for 11 out of 34 contaminants, DE of 95%+ for 7 out of 34 contaminants, and DE of 81%+ for 3 out of 34 contaminants.

Table 2 below summarizes the DE values obtained for DG products on a range of inorganic and organic TICs and chemicals of concern that represent 25 different chemical classes and which were analyzed in-house on a variety of substrates, using various analytical testing methods and instrumentation. DE values determined via both the residue swipe analysis method and the direct analysis of contaminants encapsulated in the dried gels is presented. More details of the analytical method used and the decontamination efficacy values obtained for each of these chemical classes are given in the individual technical reports of each chemical class (provided in the Appendices).

Table 2. Decontamination Efficacy of DG on twenty-five (25) chemical classes, on a minimum of three (3) selected substrates that are representative of a broad range of different chemical classes used in shipyard activities and other industrial areas.

Toxic Industrial Chemical Classes/ Chemical Classes of Concern	Substrate Evaluated	Decontamination Efficacy (%) via Direct Gel Analysis	Decontamination Efficacy (%) via Swipe Method
Aldehydes	**Glutaraldehyde**		
	Stainless Steel	92.2 ± 0.23	100*
	Aluminum	92.4 ± 0.16	100*
	Concrete	77.5 ± 0.46	100*
Alkaloids	**Nicotine**		
	Stainless Steel	97.6 ± 0.15	99.8 ± 0.0
	Aluminum	97.7 ± 0.73	99.8 ± 0.0
	Concrete	88.0 ± 0.40	99.5 ± 0.0
Aluminum Compounds	**Aluminum powder**		
	Stainless Steel	43.3 ± 3.8**	97.9 ± 2.0**
	Carbon Steel	75.8 ± 9.6**	99.5 ± 4.6**
	Concrete	95.2 ± 11.2	94.0 ± 2.8
	Aluminum Oxide		
	Stainless Steel	101.1 ± 4.6	99.7 ± 7.7
	Carbon Steel	93.0 ± 15.2	99.9 ± 14.4
	Concrete	90.1 ± 1.9	99.7 ± 5.9
	Aluminum Chloride		
	Stainless Steel	90.8 ± 4.6[1]/ 95.9 ± 8.5[2]	98.9 ± 0.7
	Carbon Steel	105.9 ± 12.9	98.5 ± 5.9
	Concrete	73.6 ± 22.2[1]/ 87.7 ± 13.4[2]	93.0 ± 18.7
	Aluminum Potassium Sulfate		
	Stainless Steel	96.4 ± 4.9	95.1 ± 7.0
	Carbon Steel	95.4 ± 4.2	94.6 ± 4.0
	Concrete	95.0 ± 11.4	81.9 ± 8.9
Amine Compounds	**Aniline**		
	Stainless Steel	93.9 ± 0.48	100*
	Aluminum	93.8 ± 0.43	100*
	Concrete	78.2 ± 0.40	100*
Aromatic Liquids	**Toluene**		
	Stainless Steel	93.7 ± 0.41[3]/ 95.0 ± 0.18[5]	99.8 ± 0.0[3]/ 100*[4]
			100*[5]/100*[6]
	Floor Tile	94.2 ± 0.32[3]/ 95.3 ± 0.22[5]	99.8 ± 0.0[3]/ 100*[4]
			100*[5]/100*[6]
	Concrete	73.6 ± 0.54[3]/ 78.1 ± 0.19[5]	99.5 ± 0.0[3]/ 100*[4]
			100*[5]/100*[6]
Aromatic Solids	**Naphthalene**		
	Stainless Steel	96.3 ± 0.36	99.6 ± 0.0
	Aluminum	96.2 ± 0.16	99.6 ± 0.0
	Concrete	89.1 ± 0.75	99.2 ± 0.01
Arsenic Compounds	**Arsenic Trioxide**		
	Stainless Steel	86.9 ± 2.9**	99.6 ± 7.8
	Carbon Steel	78.3 ± 5.7**	99.3 ± 6.7
	Concrete	87.7 ± 6.0	99.8 ± 4.0
Barium Compounds	**Barium Chloride**		
	Stainless Steel	101.5 ± 4.1	99.3 ± 0.3
	Carbon Steel	97.7 ± 5.4	99.8 ± 5.5
	Concrete	85.0 ± 5.7	98.6 ± 1.9
	Barium Carbonate		
	Stainless Steel	99.2 ± 4.9	95.0 ± 2.8

	Carbon Steel	94.2 ± 2.9	96.5 ± 8.1
	Concrete	83.1 ± 3.4	90.1 ± 4.1
Beryllium Compounds	**Aluminum powder[7]**		
	Stainless Steel	43.3 ± 3.8**	97.9 ± 2.0**
	Carbon Steel	75.8 ± 9.6**	99.5 ± 4.6**
	Concrete	95.2 ± 11.2	94.0 ± 2.8
	Aluminum Oxide[7]		
	Stainless Steel	101.1 ± 4.6	99.7 ± 7.7
	Carbon Steel	93.0 ± 15.2	99.9 ± 14.4
	Concrete	90.1 ± 1.9	99.7 ± 5.9
Cadmium Compounds	**Cadmium Chloride**		
	Stainless Steel	94.2 ± 8.5	99.5 ± 1.7
	Aluminum	71.7 ± 0.9**	N/A[8]
	Carbon Steel	94.4 ± 5.2	98.5 ± 4.2
	Concrete	91.7 ± 4.7	97.6 ± 2.2
	Cadmium Oxide		
	Stainless Steel	100.1 ± 3.5	100.0 ± 0.2
	Carbon Steel	99.4 ± 5.6	100.0 ± 0.4
	Concrete	93.8 ± 4.3	98.4 ± 0.2
Cyanide Compounds	**Potassium Ferricyanide**		
	Stainless Steel	95.7 ± 2.4	99.4 ± 4.2
	Carbon Steel	94.4 ± 3.0	98.9 ± 3.4
	Concrete	92.0 ± 5.4	98.2 ± 3.2
Halogenated Solvents	**Tetrachloroethylene**		
	Stainless Steel	95.8 ± 0.22[3]/ 95.9 ± 0.32[5]	99.6 ± 0.01[3]/ 100*[4]
			99.5 ± 0.0[5]/ 100*[6]
	Floor Tile	95.9 ± 0.10[3]/ 95.9 ± 0.16[5]	100*[3]/ 100*[4]
			100*[5]/100*[6]
	Concrete	80.5 ± 0.01[3]/ 78.5 ± 0.27[5]	100*[3]/100*[4]
			100*[5]/100*[6]
Hydrocarbon Petroleum Distillates	**Kerosene**		
	Stainless Steel	95.8 ± 0.22[3]/ 95.9 ± 0.32[5]	95.8 ± 0.12[3]/ 99.8 ± 0.06[4]
			100*[5]/100*[6]
	Aluminum	95.9 ± 0.10[3]/ 95.9 ± 0.16[5]	96.0 ± 0.10[3]/ 99.8 ± 0.06[4]
			100*[5]/100*[6]
	Concrete	80.5 ± 0.01[3]/ 78.5 ± 0.27[5]	92.8 ± 0.16[3]/ 100*[4]
			100*[5]/100*[6]
Iodine Compounds	**Potassium Iodide**		
	Stainless Steel	99.5 ± 2.9	99.5 ± 2.9
	Carbon Steel	95.1 ± 5.1	98.8 ± 6.8
	Floor Tile	96.0 + 5.8	98.6 ± 1.1
	Concrete	87.3 ± 5.2	98.7 ± 3.2
Industrial Coolants	**Ethylene Glycol**		
	Stainless Steel	97.4 ± 0.41	95.5 ± 0.01
	Aluminum	97.5 ± 0.16	95.5 ± 0.01
	Concrete	79.5 ± 0.91	93.6 ± 0.01
Industrial Solvents	***m*-Cresol**		
	Stainless Steel	96.4 ± 0.42	99.8 ± 0.0
	Aluminum	96.8 ± 0.39	99.8 ± 0.0
	Concrete	83.8 ± 0.42	99.4 ± 0.0
Iron Compounds	**Iron Chloride**		
	Stainless Steel	98.3 ± 3.6	99.5 ± 5.1
	Aluminum	91.8 ± 2.7	N/A
	Carbon Steel	97.9 ± 4.1	99.5 ± 6.3
	Concrete	86.0 ± 3.3	98.8 ± 6.5
Mercury Compounds (Non elemental)	**Mercury (II) Chloride**		
	Stainless Steel	49.0 ± 10.7**	64.6 ± 13.8**
	Carbon Steel	57.7 ± 8.1**	57.7 ± 8.1**
	Concrete	76.7 ± 7.7	95.3 ± 4.8

	Mercury (II) Oxide		
	Stainless Steel	99.6 ± 5.6	99.9 ± 11.5
	Carbon Steel	100.4 ± 8.2	99.9 ± 7.4
	Concrete	98.0 ± 3.9	99.8 ± 5.4
Nickel Compounds	**Nickel Nitrate**		
	Stainless Steel	97.9 ± 2.3	99.9 ± 7.3
	Carbon Steel	94.1 ± 4.7	99.3 ± 8.5
	Concrete	87.6 ± 3.1	98.3 ± 4.6
Organic Acids	**Benzoic Acid**		
	Stainless Steel	98.6 ± 0.68	99.7 ± 0.01
	Aluminum	99.0 ± 0.53	99.8 ± 0.0
	Concrete	88.6 ± 0.41	98.4 ± 0.10
Organic Bases	**Triethylamine**		
	Stainless Steel	93.9 ± 0.21	99.9 ± 0.01
	Aluminum	93.8 ± 0.28	99.9 ± 0.01
	Concrete	75.7 ± 0.16	99.7 ± 0.07
Pesticides	**DDT**		
	Stainless Steel	97.6 ± 0.27	98.7 ± 0.0
	Carbon Steel	97.8 ± 0.42	98.7 ± 0.0
	Concrete	88.9 ± 0.57	96.1 ± 0.13
Phenolic Compounds	**Catechol**		
	Aluminum	96.7 ± 0.36	99.2 ± 0.01
	Carbon Steel	96.1 ± 0.01	99.2 ± 0.12
	Concrete	90.4 ± 0.58	97.3 ± 0.21
Selenium Compounds	**Selenium powder**		
	Stainless Steel	100.5 ± 4.7	99.2 ± 2.8
	Carbon Steel	94.4 ± 3.1	99.8 ± 7.1
	Concrete	88.2 ± 6.1	96.2 ± 7.4
Zinc Compounds	**Zinc powder**		
	Stainless Steel	86.9 ± 2.9**	77.3 ± 6.4**
	Carbon Steel	78.3 ± 5.7**	66.9 ± 2.9**
	Concrete	87.7 ± 6.0	86.5 ± 7.7
	Zinc Oxide		
	Stainless Steel	99.7 ± 6.1	100.0 ± 0.2
	Carbon Steel	95.7 ± 3.1	100.0 ± 0.4
	Concrete	86.3 ± 1.9	98.4 ± 0.2
	Zinc Acetate		
	Stainless Steel	99.8 ± 8.5	98.2 ± 6.0
	Carbon Steel	100.1 ± 3.5	98.4 ± 3.9
	Concrete	91.0 ± 7.1	97.6 ± 4.4

* Contaminant residue amount below limit of detection (LOD) (see individual TIC/Chemicals of Concern Reports in the Appendices for more details).
** Contaminant reacted with substrate, creating a fixed residue on the contaminated surface that could not be completely removed by DG, as evidenced by a lower than expected DE obtained via direct gel analysis.

1. 1st application of gel.
2. 2nd application of gel.
3. DG 1101 non-brushed (poured) onto contaminated surface.
4. DG 1101 brushed onto contaminated surface.
5. DG 1102 non-brushed (poured) onto contaminated surface.
6. DG 1102 brushed onto contaminated surface.
7. Suitable contaminant surrogate for extremely toxic contaminant class.
8. N/A: decontamination efficacy value has not been determined; more details are given in each technical report for a specific chemical class.

For complex surface decontamination scenarios, including volatile or reactive contaminants, DG decontamination efficacy was modest, primarily in part due to the experimental complexities that arose due to contaminant evaporation from coupon surfaces (loss of contaminant to atmosphere, resulting in artificially low decontamination efficacy values for some contaminants), or reaction of contaminants with either DG components, metal or polymeric coupon surfaces, or with environmental water (forming contaminant- DG component adducts bound into the film matrix, surface fixed coupon-metal oxides, irreversible surface sorption of contaminants, or "sticky"/viscous contaminant hydrates/gels intercalated into porous coupon surfaces or fixed strongly onto metal surfaces). Nevertheless, DG showed excellent decontamination efficacy on all classes of loose particle and non-reactive solid and liquid contaminants on all coupon surfaces investigated.

We noted that for some inorganic contaminant-substrate combinations a reaction took place between contaminant and substrate, resulting in a fixed residue. These contaminant-substrate combinations (first compound is the contaminant) are: 1) Aluminum-steel (carbon and stainless), 2) Arsenic-steel (carbon and stainless), 3) Cadmium-aluminum, 4) Mercury-steel (carbon and stainless), 5) Zinc-steel (carbon and stainless). This resulted in the inability of DG or other hydrogel formulations to completely remove the fixated contaminants from the contaminated surface, as evidenced by the lower than expected Decontamination Efficacy values determined via direct gel analysis. More details on the determination of the decontamination efficacy of DG, and other hydrogel formulations, via the direct analysis of encapsulated contaminant are given in the technical reports for each chemical class evaluated (provided in the Appendix).

For non-polar, lipophilic liquid "oil and grease-like" contaminants such as hydrocarbon petroleum distillates (kerosene), both DG 1101 and 1102 formulations were utilized, employing both poured (non-brushed) and brushed film applications.

4.2 Decontamination of PHNSY/NAVSEA Specific Contaminant/Substrate Combinations

The second major objective of this contract included the evaluation of DG products and other hydrogel formulations for 12 PHNSY/NAVSEA specific contaminant/substrate combinations. There is a significant number of toxic and hazardous chemicals and chemicals of concern/substrate combinations that are present in the naval setting. This objective comprised an in-depth evaluation of the decontamination efficacy of DG products on a relevant range of chemical classes that cover the spectrum of chemical contaminant classes of concern and specific contaminant/substrate combinations deemed of major concern commonly encountered in Navy shipyards, infrastructure and other areas.

PHNSYandIMF specifically identified some contaminants as being of immediate need of an efficient and cost effective decontamination solution. Examples included: the need for improved methods of decontaminating the copper and zinc containing particulates from the cracks, crevices and porosity of the dry-dock floor within the containment area after completion of the hydro-blasting operations. Improvements would be of value in reducing labor expenditures and improving Best Management Practices (BMP) for PHNSYandIMF's compliance with their National Pollutant Discharge Elimination System (NPDES) dry-dock permit. In another example, the need to clean up dye marker spills, which expends a significant amount of labor because of the high concentration of the dye, was identified.

The development of solutions to 12 Navy specific decontamination needs has been accomplished with great success as described below (Table 4). A programmatic request arose due to the PHNSY-IMF declining to provide cost models (cost comparison of the developed method vs. the current method) for these evaluations, and thus the evaluations were only based upon laboratory data.

Depending on their chemical properties and solvent solubility characteristics, some contaminants required significant and extensive experimental and analytical method development. All samples and controls were run in triplicates, which totaled a sum of 200+ different samples analyzed via ICP-OES, GC-MS, LC-MS, Mercury Vapor Analysis, and PL (Polarized Light) and TE (Transmission Electron) Microscopies.

This objective involved the decontamination efficacy determination of DG on twelve (12) priority contaminant/substrate combinations comprising four major chemical classes 1) Organics (PCBs, Motor Oil, Crude Oil, Jet Fuel), 2) Toxic Metals (Organotin, Mercury, Copper, Lead, Chromium), 3) Special-Concern Contaminants (Navy Dye Marker, Asbestos), and 4) Chemical Warfare Agent Simulants (Methyl Benzoate, 2-Chloroethyl Ethyl Sulfide (CEES), 2-Chloroethyl Phenyl Sulfide (CEPS)).

Contaminants evaluated during this objective included:

1. PCBs - once widely used as industrial dielectric additives in transformer and capacitor coolants and insulating fluids, due to undesirable carcinogenicity, PCB production has been banned since the 1970s. PCBs are considered chemically inert and resist environmental degradation, and as such remain environmentally persistent pollutants (evaluated on stainless steel, cementacious leveling compounds, painted metal deck plate, brass, aluminum, and concrete substrates);

2. Motor Oil - used throughout the world as a combustion engine lubricant, motor oil is derived from petroleum-based materials including crude oil, and is composed of a variety of saturated and unsaturated hydrocarbons including polyaromatic hydrocarbons (PAHs) (up to 6%). PAHs are widespread organic pollutants known for their carcinogenic, mutagenic, and tetratogenic properties (evaluated on stainless steel, aluminum, and concrete substrates);

3. Crude Oil - used throughout the world as an energy source and to provide a broad range of valuable and useful materials. Crude oil and its decomposition products are environmentally persistent toxins, and when released into the environment require extensive remediation typically resulting in the generation of large quantities of toxic contamination. Crude oil is comprised of 0.2-7% polyaromatic hydrocarbons (PAHs), widespread organic pollutants known for their carcinogenic, mutagenic, and tetratogenic properties (evaluated on stainless steel, aluminum, and concrete substrates);

4. Jet Fuel (Jet A) - is a C8-C16 kerosene-type flammable fuel. Jet Fuel vapors and liquid are harmful, ingestion of sufficient quantities can be fatal, therefore, Jet Fuel use requires adopting safe and proper handling, storage, and disposal procedures (evaluated on stainless steel, aluminum, and concrete substrates);

5. Organotin - compounds have been extensively used as biocides, wood preservatives, and as anti-biofouling agents, however, concerns over potent toxicity to marine life have led to a worldwide ban by the International Maritime Organization. Organotin compounds are considered environmentally persistent pollutants (evaluated on stainless steel, aluminum, and concrete substrates);

6. Mercury (elemental) - used in thermometers, electronics, lighting, gold and silver refinery, and combined with other metals to form useful amalgams. Mercury and most of its compounds are extremely toxic, causing neurological damage due to inhalation of vapors/dust or

ingestion (evaluated on stainless steel, concrete, porcelain, composite, and linoleum tiles substrates);

7. Copper compounds - are commonly used as components of semiconductors, pigments, fungicides, and as antifouling agents for marine paints. Due to its toxicity toward aquatic organisms, copper is considered an environmentally persistent pollutant. Elemental copper and copper (I) oxide were chosen as a representative copper compounds for evaluating DG's efficacy; DG is expected to have similar efficacy towards the wide range of copper compounds (evaluated on stainless, carbon steels, concrete, rubber, lexan substrates);

8. Lead compounds - used in batteries, bullets, as part of solders and alloys, and as a radiation shields. Specifically, lead (II) oxide is used in the production of ceramic glazes and leaded glass, and vulcanized rubber; lead (II) sulfate is commonly used as a component of battery electrodes. Lead and its compounds accumulate in soft tissue and bone, and are potent neurotoxins. The lead compounds PbO and $PbSO_4$ were chosen as representative lead compounds for evaluating DG's efficacy; DG is expected to have similar efficacy towards the wide range of lead compounds (evaluated on stainless steel, aluminum, and concrete substrates);

9. Potassium chromate - a strong oxidizing agent that is very toxic and may be fatal if swallowed, and can cause cancer on inhalation. Potassium chromate was chosen as a representative chromium compound for evaluating DG's efficacy; DG is expected to have similar efficacy towards the wide range of chromium compounds (evaluated on stainless, carbon steels, concrete substrates);

10. Asbestos - a set of six naturally occurring silicate minerals exploited commercially for their desirable physical properties, they all have in common their long and thin fibrous crystals. The inhalation of asbestos fibers can cause serious illnesses, including malignant lung cancer,

100% efficacy against PCBs, 99%+ efficacy against Organotin compound, 99%+ efficacy against Navy Dye Marker, 98.5-99%+ efficacy against Motor Oil, 98.9-99%+ efficacy against Jet Fuel, 98.9-99%+ efficacy against Crude Oil, up to 99%+ efficacy against elemental Mercury, up to 99%+ efficacy against Copper compounds, up to 100% efficacy against Lead (Lead (II) Sulfate), 100% efficacy against Asbestos, up to 99%+ efficacy against VX CWA (chemical warfare agent)-Simulant Methyl Benzoate, up to 97.2% efficacy against Sulfur Mustard CWA-Simulant CEPS (2-chloroethyl phenyl sulfide), and up to 98%+ efficacy against Potassium Chromate

mesothelioma, and asbestosis (evaluated on linoleum tile, painted drywall, and concrete substrates);

11. Navy Dye Marker - a water-soluble dye used as an ocean marker for a variety of applications and uses. Navy Dye Marker resists short-term environment-mediated degradation and possesses a strong chromophore, and as such even small amounts of dye are plainly visible on both porous and non-porous surfaces, complicating the complete and facile removal of Navy Dye Marker from commonly utilized surfaces (evaluated on stainless steel, aluminum, and concrete substrates);

12. Methyl Benzoate (VX-Simulant) - VX, an organo-phosphono-thioate, is a toxic nerve agent classified as a weapon of mass destruction by the United Nations. VX can be distributed as a liquid or aerosol, both pure and as a mixture, and due to its high viscosity and low volatility is considered an environmentally persistent biohazard. VX and its simulant methyl benzoate (MBz) have near identical Water:Octanol Coefficients (2.09 and 2.12, respectively), and as such behave similarly in both environmental settings and biological systems (evaluated on cadmium-plated, carbon steels, CARC, silicone and black rubbers substrates);

13. CEES (2-Chloroethyl Ethyl Sulfide) (Sulfur Mustard-Simulant) - Sulfur Mustard, bis-(2-chloroethyl) sulfide, is a powerful vesicant, cytotoxic agent regulated under the 1993 Chemical Weapons Convention (CWC). Sulfur mustard can be deployed by spraying, or more typically distributed via artillery shells and bombs. For experimental testing purposes, the half-mustard 2-chloroethyl ethyl sulfide (CEES) is used as an effective yet less toxic simulant for sulfur mustard chemical warfare agents (evaluated on cadmium-plated, carbon steels, CARC, silicone and black rubbers substrates); and

14. CEPS ((2-Chloroethyl Phenyl Sulfide) (Sulfur Mustard-Simulant) - Sulfur Mustard, bis-(2-chloroethyl) sulfide, is a powerful vesicant, cytotoxic agent regulated under the 1993 Chemical Weapons Convention (CWC). Sulfur mustard can be deployed by spraying, or more typically distributed via artillery shells and bombs. For experimental testing purposes, the half-mustard 2-chloroethyl phenyl sulfide (CEPS) is used as an effective yet less toxic simulant for sulfur mustard chemical warfare agents (evaluated on cadmium-plated, carbon steels, CARC, silicone and black rubbers substrates).

Highlights of decontamination efficacy (DE) values (% reduction in contaminant concentration) by utilizing DG formulations for the Navy priority contaminant/substrate combinations of concern include: 100% efficacy against PCBs, 99%+ efficacy against Organotin compound, 99%+ efficacy against

Navy Dye Marker, 98.5-99%+ efficacy against Motor Oil, 98.9-99%+ efficacy against Jet Fuel, 98.9-99%+ efficacy against Crude Oil, up to 99%+ efficacy against elemental Mercury, up to 99%+ efficacy against Copper compounds, up to 100% efficacy against Lead (Lead (II) Sulfate), 100% efficacy against Asbestos, up to 99%+ efficacy against VX CWA (chemical warfare agent)-Simulant Methyl Benzoate, up to 97.2% efficacy against Sulfur Mustard CWA-Simulant CEPS (2-chloroethyl phenyl sulfide), and up to 98%+ efficacy against Potassium Chromate, as determined by various analyses (including residue swipe analysis, solvent extraction, vapor analysis, tape adhesion, and microscopy – see individual contaminant technical reports for more details. Depending on the individual contaminant in question, sampling methods utilized in this Project were those typically utilized when conducting standardized industrial decontamination protocols (i.e. standardized EPA/JPEO-CBD methods).

In particular, for non-porous surfaces (including stainless, carbon, Cd-plated steels and aluminum), DG formulations showed DE of 99%+ for 10 out of 12 contaminants evaluated, and DE of 97%+ for the remaining 2 out of 12 contaminants.

For porous surfaces (including industrial grade concrete, composite, linoleum, porcelain tiles, painted drywall, silicone and black rubbers, CARC) DG formulations showed DE of 99%+ for 7 out of 12 contaminants evaluated, DE of 97%+ for 3 out of 12 contaminants, and DE of 93%+ for the remaining 2 out of 12 contaminants.

Success in CWA simulants decontamination work performed under this Task, and under the guidance of the Joint Program Executive Office for Chemical and Biological Defense (JPEO-CBD), resulted in the JPEO-CBD testing DG on classified CWAs at their own expense (refer to later sections of this report).

Table 3 below summarizes the DE values obtained for DG formulations on a range of inorganic and organic TICs and chemicals of concern that represent twelve priority contaminant/substrate combinations that were specifically identified by PHNSY/NAVSEA that covers to an extent a spectrum of chemical contaminant classes and chemical classes of concern that can be found in the Naval setting, using various analytical testing methods and instrumentation. DE values determined via both residue analysis and direct analysis of contaminants encapsulated in DG is presented. More details of the analytical methods used and the decontamination efficacy values obtained for each of these contaminant classes/substrate combinations are given in the technical reports provided in the Appendix section.

Table 3. Decontamination Efficacy of DG products on twelve (12) Navy priority contaminant and/or substrate combinations.

Navy Priority Contaminant/Substrate Combinations	Substrate Evaluated	Decontamination Efficacy (%) via Direct Gel Analysis	Decontamination Efficacy (%) via Swipe[1]/Extraction[2]/ Vapor Analysis[3] Method
PCBs	Stainless Steel[1]	93.3 ± 0.16[4]/ 95.6 ± 0.46[5]	92.4 ± 0.10[4]/ 94.1 ± 0.10[5]
			100*[6]/100*[7]
	Aluminum[1]	93.3 ± 0.16[4]/ 95.4 ± 0.38[5]	92.4 ± 0.10[4]/ 94.1 ± 0.10[5]
			100*[6]/100*[7]
	Concrete[1]	80.4 ± 0.31[4]/ 82.9 ± 0.25[5]	83.9 ± 0.18[4,8]/ 87.2 ± 0.11[5,8]
			100*[6,8]/100*[7,8]
			98.6 ± 0.10[4,9]/ 98.8 ± 0.15[5,9]
			100*[6,9]/100*[7,9]
			98.8 ± 0.10[4,10]/ 99.0 ± 0.12[5,10]
			100*[6,10]/100*[7,10]
Organotin	**Tributyltin chloride**		
	Stainless Steel[1]	95.2 ± 0.42[4]	96.2 ± 0.21[4]
			99.4 ± 0.10[6]
	Aluminum[1]	95.4 ± 0.48[4]	96.3 ± 0.21[4]
			99.3 ± 0.10[6]
	Concrete[1]	84.9 ± 0.88[4]	86.9 ± 0.16[4]
			99.0 ± 0.12[6]
Navy Dye Marker	**Yellow Navy Dye Marker**		
	Stainless Steel[1]	99.2 ± 0.10	99.4 ± 0.10
	Aluminum[1]	99.1 ± 0.10	99.3 ± 0.10
	Concrete[2]	97.8 ± 0.46	99.1 ± 0.10
Motor Oil	**Motor Oil Type SAE 5W-30**		
	Stainless Steel[1]	96.4 ± 0.24[4]/ 95.5 ± 0.22[5]	96.2 ± 0.16[4]/ 95.3 ± 0.16[5]
			99.4 ± 0.12[6]/ 99.5 ± 0.12[7]
	Aluminum[1]	96.8 ± 0.10[4]/ 96.4 ± 0.16[5]	96.2 ± 0.16[4]/ 95.3 ± 0.10[5]
			99.4 ± 0.16[6]/ 99.5 ± 0.10[7]
	Concrete[1]	87.8 ± 0.75[4]/ 86.0 ± 0.58[5]	87.2 ± 0.91[4]/ 85.6 ± 0.64[5]
			98.0 ± 0.15[6]/ 98.5 ± 0.15[7]
Jet Fuel	**Jet Fuel A**		
	Stainless Steel[1]	95.1 ± 0.11[4]/ 93.6 ± 0.22[5]	94.4 ± 0.22[4]/ 92.7 ± 0.43[5]
			99.2 ± 0.12[6]/ 99.4 ± 0.15[7]
	Aluminum[1]	95.6 ± 0.10[4]/ 93.6 ± 0.22[5]	94.5 ± 0.26[4]/ 92.9 ± 0.16[5]
			99.4 ± 0.15[6]/ 99.4 ± 0.15[7]
	Concrete[1]	91.0 ± 0.27[4]/ 89.2 ± 0.28[5]	90.3 ± 0.29[4]/ 89.2 ± 0.29[5]
			98.7 ± 0.10[6]/ 98.9 ± 0.17[7]
Crude Oil	**Light, Sweet Crude Oil**		
	Stainless Steel[1]	95.6 ± 0.42[4]/ 93.5 ± 0.33[5]	93.9 ± 0.10[4]/ 91.3 ± 0.16[5]
			99.0 ± 0.10[6]/ 99.5 ± 0.10[7]

	Aluminum[1]	95.7 ± 0.24[4]/ 93.5 ± 0.33[5]	93.9 ± 0.10[4]/ 94.1 ± 0.34[5]
			99.1 ± 0.10[6]/ 99.6 ± 0.10[7]
	Concrete[1]	84.5 ± 0.66[4]/ 81.7 ± 0.58[5]	85.9 ± 0.18[4]/ 86.6 ± 0.36[5]
			98.4 ± 0.10[6]/ 98.9 ± 0.10[7]
Mercury	**Elemental Mercury**		
	Stainless Steel[3]	ND1	98.4 ± 0.7[11]
	Concrete[3]	ND1	97.8 ± 0.7[11]
	Porcelain Tile[3,**]	ND1	99.2 ± 0.4[11]
			85.9 ± 11.7[12]
	Composite Tile[3,**]	ND1	91.7 ± 2.1[11]
			66.0 ± 11.7[12]
	Linoleum Tile[3,**]	ND1	95.4 ± 0.9[11]
			90.8 ± 5.8[12]
			99.0 ± 0.2[13]
Copper Compounds	**Elemental Copper**		
	Stainless Steel[1]	99.9 ± 6.2	99.9 ± 8.9
	Carbon Steel[1]	97.8 ± 4.8	99.5 ± 7.0
	Concrete[1]	98.2 ± 5.4	98.0 ± 2.9
	Rubber[1]	99.1 ± 9.7	99.4 ± 9.9
	Lexan[1]	98.3 ± 8.1	99.5 ± 4.9
	Copper (I) Oxide		
	Stainless Steel[1]	71.9 ± 4.2[14]	99.7 ± 8.7
		85.2 ± 4.6[15]	
		94.5 ± 4.1[16]	
	Carbon Steel[1]	64.1 ± 8.4[14]	98.9 ± 6.9
		100.5 ± 6.0[15]	
		ND2[16]	
	Concrete[1,**]	74.7 ± 18.1[14]	94.7 ± 3.9
		24.7 ± 5.2[15]	
		69.7 ± 26.0[16]	
Lead Compounds	**Lead (II) Oxide**		
	Stainless Steel[1,**]	96.8 ± 4.4	91.5 ± 5.0
	Aluminum[1,**]	93.4 ± 4.7	81.2 ± 7.4
	Concrete[1,**]	78.7 ± 8.2	91.2 ± 8.7
	Lead (II) Sulfate		
	Stainless Steel[1]	99.6 ± 5.0	99.4 ± 3.5
	Aluminum[1]	97.7 ± 2.0	99.8 ± 8.9
	Concrete[1]	87.5 ± 12.1	99.8 ± 7.6
Asbestos	**Chrysotile Fibers**		
	Linoleum Tile	ND2	100[*,17]
			99.97[18]
	Painted Drywall	ND2	100[*,17]
	Concrete	ND2	100[*,18]
Chemical Warfare Agents (Simulants)	**VX-Simulant (Methyl Benzoate)**		
	Cd-plated Steel[2]	ND2	96.7 ± 0.19
	Carbon Steel[2]	ND2	99.2 ± 0.11
	CARC[2,**]	ND2	67.3 ± 2.34
	Silicone Rubber[2,**]	ND2	62.3 ± 1.17
	Black Rubber[2,**]	ND2	26.5 ± 1.88
	Sulfur Mustard-Simulant (2-Chloroethyl Ethyl Sulfide (CEES))		
	Cd-plated Steel[2]	ND2	ND3
	Carbon Steel[2]	ND2	ND3
	CARC[2,**]	ND2	28.6 ± 0.0
	Silicone Rubber[2,**]	ND2	54.6 ± 0.20
	Black Rubber[2,**]	ND2	93.2 ± 0.12
	Sulfur Mustard-Simulant (2-Chloroethyl Phenyl Sulfide (CEPS))		
	Cd-plated Steel[2]	ND2	96.3 ± 0.1
	Carbon Steel[2]	ND2	97.2 ± 0.1
	CARC[2,**]	ND2	51.2 ± 0.51

	Silicone Rubber[2,**]	ND2	51.9 ± 0.32
	Black Rubber[2,**]	ND2	76.9 ± 0.11
Chromium Compounds	**Potassium Chromate**		
	Stainless Steel[1]	99.6 ± 5.9	97.7 ± 11.8
	Carbon Steel[1]	99.2 ± 9.6	98.9 ± 9.6
	Concrete[1,**]	91.9 ± 3.5	67.4 ± 13.7

* Contaminant residue amount below limit of detection (LOD) (see individual Chemical Reports in the Appendices for more detail).

** Contaminant reacted with substrate and/or with trace amounts of water, or absorbed into substrate, creating a fixed residue on/within the contaminated surface that could not be completely removed by DG; as evidenced by a lower than expected Decontamination Efficacy determined via direct analysis of the dried gel (if applicable).

ND1: not determined; elemental mercury contaminant was analyzed using a mercury vapor analyzer, and as such a Decontamination Efficacy via direct gel analysis was not determined.

ND2: not determined.

ND3: not determined; no contaminant detected for samples and/or controls.

1) Swipe method of analysis: use of swipe testing.
2) Extraction method of analysis: use of solvent extraction.
3) Vapor analysis method of analysis: use of mercury vapor analyzer.
4) DG 1101 non-brushed (poured) onto contaminated surface.
5) DG 1102 non-brushed (poured) onto contaminated surface.
6) DG 1101 brushed onto contaminated surface.
7) DG 1102 brushed onto contaminated surface.
8) DG was allowed to dry for 24 hrs.
9) 2nd DG application (3 days/72 h after 1st application).
10) 2nd DG application (7 days/168 h after 1st application).
11) Contaminant amount applied on a substrate was controlled by utilizing a pipet; removal of bulk mercury liquid to yield contaminant as very small liquid beads (<1 mm diameter); DG was poured onto contaminated surface.
12) Contaminant amount applied on a substrate was controlled by utilizing zinc dust; removal of bulk mercury liquid to yield contaminant as very small liquid beads (<1 mm diameter); DG was poured onto contaminated surface.
13) Contaminant amount applied on a substrate was controlled by utilizing zinc dust; removal of bulk mercury liquid to yield contaminant as very small liquid beads (<1 mm diameter); DG was brushed onto contaminated surface.
14) 100 mg of contaminant was applied on the substrate.
15) 50 mg of contaminant was applied on the substrate, 1st application of DG.
16) 50 mg of contaminant was applied on the substrate, 2nd application of DG.
17) Decontamination Efficacy determined by tape lift adhesion sampling, polarized light microscopy analysis method.
18) Decontamination eficacy determined by swipe sampling, transmission electron microscopy analysis method. Independent laboratory results.

DG was found to provide excellent Decontamination Efficacies (DE) for complex, difficult to clean contaminant/substrate combination scenarios as encountered in the Navy/naval industrial settings; some examples include: 1) for PCBs on concrete (a porous, sorptive ubiquitous naval working surface deemed difficult to effectively clean), brushing of both DG 1101 and 1102 on industrial concrete contaminated with PCB-laden mineral oil afforded 100% DE (as determined by residual swipe analysis), such that after 1, 3, and even 7 days after contamination, no PCBs were detected after surface decontamination (limit of detection (LOD) for PCBs approximated 250 ppb for the in-house evaluation); and 2) for Navy Dye Marker on concrete (a porous, sorptive ubiquitous naval working surface), simple pouring of DG 1101 on industrial concrete contaminated with a concentrated aqueous suspension of Navy Dye Marker resulted in 99.1% DE (as determined by solvent extraction) achieved one day after surface contamination; additionally, even when water droplets were added to decontaminated concrete substrates and allowed to stand for 5 and 24 hours, no resurfacing of Navy Dye Marker was evidenced (no color was incorporated into added water droplets or evaporated droplet residue (as determined by visual inspection)).

We noted that for some inorganic contaminant-substrate combinations a reaction took place between contaminant and substrate/trace amounts of water, resulting in a fixed residue. These contaminant-substrate combinations (first compound is the contaminant) are: 1) Copper (I) oxide-concrete, 2) Lead (II) oxide-stainless steel, aluminum, concrete, and 3) Potassium chromate-concrete. This resulted in the inability of DG or other improved formulations to completely remove the fixated contaminants from the contaminated surface, as evidenced by the lower than expected Decontamination Efficacy values determined via multiple analytical methods, including direct gel analysis (if applicable).

For non-polar, lipophilic liquid "oil and grease-like" contaminants such as PCB oil, both DG 1101 and 1102 formulations were utilized, employing both poured (non-brushed) and brushed film applications.

For complex surface decontamination scenarios, including volatile or reactive contaminants, DG decontamination efficacy was modest, primarily in part due to the experimental complexities that arose due to contaminant evaporation from coupon surfaces (loss of contaminant to atmosphere, resulting in artificially low decontamination efficacy values for some contaminants), or reaction of contaminants with either DG components, metal or polymeric coupon surfaces, or with environmental water (forming contaminant- DG component adducts bound into the film matrix, surface fixed coupon-metal oxides, irreversible surface sorption of contaminants, or "sticky"/viscous contaminant hydrates/gels intercalated into porous coupon surfaces or fixed strongly onto metal surfaces). Nevertheless, DG showed excellent decontamination efficacy on all classes of loose particle and non-reactive solid and liquid contaminants from all coupon surfaces investigated.

Decontamination effectiveness utilizing two or more DG applications: For contaminants exhibiting lower than expected DE, experiments were repeated using multiple DG applications in an attempt to improve surface decontamination efficacy. For those contaminant-substrate combinations that exhibited lower than expected decontamination efficacies, we noted that the contaminant reacted with the substrate, creating a visible, fixed residue on the contaminated surface that could not be completely removed by the hydrogel decontamination method. Multiple DG applications did not appreciably improve DE values, indicating that lower than expected decontamination efficacies initially achieved were the result of the reaction of contaminant with substrate, rather than a lack of ability or efficiency of DG to afford acceptable surface decontamination. Depending on the specific decontamination task and the decontamination efficacy that is required by the end-user though, multiple applications of DG products or other hydrogel formulations might prove to be of benefit and improve the overall decontamination efficacy.

Decontamination effectiveness utilizing variable DG film thickness: Variations in DG film thickness were evaluated in an effort to improve upon DG products or other hydrogel formulations surface decontamination effectiveness. Very thin gel film thickness is not recommended when the amount of contamination on a specific surface is appreciably high, because this results in the saturation of gel by the contaminant of interest. In addition, very thin films are difficult to peel-off from porous surfaces. In general, we noted that the most effective film thickness utilized was one that completely covered the contaminated area and that corresponded to an at least 1 to 5 contaminant to gel ratio by volume (visual estimate); sometimes a 1 to 10 contaminant to gel ratio is required. This ratio seemed to facilitate an optimized interaction between contaminant and DG chemically-active components.

A sufficiently thick film layer that allowed easy peel-off of the dried gel was achieved by applying that amount of DG necessary to completely cover the contaminated area. For substrates measuring 50-100 cm^2 surface area, approximately 6.0 grams of DG was found to be sufficient; additional amounts or multiple layers of film did not appreciably improve DG decontamination effectiveness. This corresponds to about 100-150 sq. ft/gallon for non-porous substrates and to 25-50 sq. ft/gallon for porous substrates. It is recommended that end-users test a small area to determine effective thickness for optimum decontamination efficacy and easy peel-off of the dried gel prior to applying DG products for the entire project.

Decontamination effectiveness utilizing DG brushing/mixing: Application of DG products onto contaminated surfaces by brush, especially for liquid contaminants such as hydrocarbon petroleum distillates and chlorinated solvents, had a profound positive effect on DG decontamination effectiveness, such that significantly improved decontamination efficacies were achieved when comparing brushed DG application methods (see Table 4) versus poured (non-brushed) application methods. For example, DG 1101 showed a 93.9% DE on stainless steel and an 85.9% DE on concrete against crude oil by pouring the formulation on top of the contaminated area. On the other hand, brushing of DG 1101 on top of the contaminated area yielded 99.0% and 98.4% DEs for crude oil on steel and concrete respectively. Brushing appears to aid in the emulsification and incorporation of the contaminants into the hydrogel matrix and is recommended especially for hydrophobic liquid contaminants and contaminants where there is a visible layer on top of the substrate.

Decontamination effectiveness utilizing variable DG film viscosity: Variations in DG film viscosity were evaluated to potentially improve upon DG surface decontamination effectiveness. Adjustments in the gel viscosity were made in an effort to enhance the decontamination and encapsulation of very fine powder contaminants in DG. What one would expect from a theoretical stand point is the improvement of decontamination efficacy of the hydrogel formulations on concrete with decreasing viscosity of the formulations since that would result in increased penetration of the hydrogel solution in the concrete pores. In-house tests though showed that lower viscosity formulations didn't seem to improve the overall decontamination efficacy. Upon drying, low viscosity films provided very thin films that were difficult to peel off from the surfaces evaluated (especially concrete).

Product efficacy data and application instructions for TICs/Chemicals of Concern contaminant-substrate combinations: Individual detailed technical reports for all chemical classes evaluated have been created and are provided in the Appendix sections of this report. An end-user format has been also provided using a reporting style that effectively details important decontamination efficacy results, testing details, and experimental design in a relevant, easy-to-understand format developed for the end-users. Along with the product efficacy data for all combinations of TICs and substrates evaluated, application instructions for end-users are provided. These include directions on how to apply the gel products as well as tips for optimum decontamination efficacies (Appendix B).

5. Field Testing/Validation of Decontamination Performance

The final objective of this contract included field testing and further validation of decontamination methods developed in house throughout the duration of this contract towards the performance goal of ≥25% cost reduction for three (3) of the twelve (12) PHNSY/NAVSEA priority chemical contaminants/substrate combinations.

Three field tests have been performed on three Navy priority chemical contaminants/substrate combinations. These include PCB oil decontamination aboard USS Missouri, decontamination of lead dust from building 6 in PHNSY and evaluation of the decontamination efficacy of DG against chemical warfare agents by JPEO-CBD. Details about the decontamination procedure, the sampling methods and the evaluation of the results for each field test are given below.

5.1 First Field Test: Aboard USS Missouri (BB-63) (PCB Oil)

Polychlorinated Biphenyls (PCBs) were widely used as industrial dielectric additives in transformer and capacitor coolants and insulating fluids. Due to undesirable carcinogenicity, PCB production has been banned since the 1970s. PCBs are considered chemically inert and resist environmental degradation, and as such remain environmentally persistent pollutants.

The USS Missouri maritime museum vessel contains several areas with limited access to the public due to PCB oil contamination. Field testing aboard the USS Missouri maritime museum vessel showed that excellent surface decontamination was achieved by applying DG 1102 onto decades-old contaminated surfaces, resulting in encapsulation of PCB contaminant by DG's active components. For all contaminated surfaces tested, EPA PCB limit standards (Regulations 761.79, 761.123) of $\leq 10\ \mu g/100\ cm^2$ were achieved upon decontamination with a single application DG 1102 (partially developed under a previous CEROS contract for the removal of hydrophobic oils and other contaminants from surfaces ahead of paint recoat).

Figure 8 below shows photos taken aboard the USS Missouri (BB-63) during PCB decontamination field testing.

Figure 8. Field testing aboard USS Missouri maritime museum vessel. A variety of substrates were decontaminated.

Missouri personnel informed us that these areas had previously been extensively decontaminated by repeatedly scrubbing with solvents (hexanes) and wiping. Even after several attempts, PCBs levels did not meet EPA regulatory limits for open public access. DG achieved that objective after only one application.

Table 4 below summarizes the decontamination efficacies of DG 1102 on multiple contaminated sites aboard the USS Missouri maritime museum. Notably, all areas were decontaminated to PCB levels below EPA (Regulations 761.79, 761.123) acceptable limits of 10µg/100cm^2 after a single application.

Table 4. PCB contamination before and after application of DG 1102 on variety of substrates including stainless steel, cementacious leveling compound surface (after removal of floor tile), painted deck plate, and brass surfaces as determined by residual swipe testing.*

Description	CONTAMINATED SURFACE MATERIAL	Before Decon (µg/100cm^2)	After Decon 1st application DG 1102 (µg/100cm^2)	After Decon 2nd application DG 1102 (µg/100cm^2)
2-111-2-Q Gen Wksp Oil on #8 Radial Press	**Stainless steel**	89.1µg/ 100cm^2 Aroclor 1254	<1.00µg/ 100 cm^2 (wire brush utilized)	<1.00µg/ 100cm^2 (wire brush utilized)
2-68-0-L Crew Living Space oil on deck from winch (underlayment)	**Cementacious Leveling Compounds**	14.7µg/ 100cm^2 Aroclor 1254	1.82 µg/ 100 cm^2 Aroclor 1260 (Stand up wire brush utilized)	6.14µg/ 100cm^2 Aroclor 1260 (Stand up wire brush utilized)
4-79-2-C Fwd Battery Plot oil on deck from overhead metal panel	**Painted Metal Deck Plate**	661µg/ 100cm^2 Aroclor 1254	<1.00µg/ 100 cm^2 (brushing with a paint brush)	<1.00µg/ 100cm^2 (brushing with a paint brush)
			3.32 µg/ 100 cm^2 Aroclor 1260 (scrubbing)	<1.00µg/ 100cm^2 (scrubbing)
4-74-2-M 16" FWD Magazine cosmolene film on brass	**Brass**	132µg/ 100cm^2 Aroclor 1254, 213µg/ 100cm^2 Aroclor 1260 Total PCBs 345µg/100cm^2	<1.00µg/ 100cm^2 (scrubbing)	<1.00µg/ 100cm^2 (scrubbing)

* PCB surface limit standards (following EPA Regulations 761.79, 761.123) ≤ 10 µg/100 cm^2.

The analysis of the samples above was performed by SGS North America Inc.; Environmental Services (Anchorage, AK). USS Missouri personnel utilized gauze pads pre-wetted with hexanes to sample 100cm^2 of coverage area before and after decon at the sites of USS Missouri that were decontaminated by DG. Sample wipes were put into tightly sealed pre-labeled amber glass containers and sent to SGS North America Inc. for test analysis of PCB oil.

The results above are in agreement with "in house" evaluation results (presented in previous sections) where decontamination efficacies were >99.99% for DG 1101 and 1102 brushed on concrete, aluminum and stainless steel surfaces as determined by residual swipe analysis.

5.2 Cost Analysis

Cost analysis was performed by a PHNSYandIMF subcontractor that performs similar decontamination jobs but on a larger scale aboard USS Missouri (Gregory Perry; President of EnviroServices and Training Center, LLC.) and was submitted to CBIP after completion of the final field tests. The total cost of decontaminating a 240 sq. ft catwalk area by current standard cleaning methods utilizing organic solvents, wipes and sorbents was compared to the total cost of decontaminating the same area by DG.

In this cost model, comparison to an actual job, previously costed and performed by EnviroServices and Training Center, LLC, was utilized. The same job was costed out by EnviroServices utilizing DG coverage, time and efficacy data determined during the USS Missouri decontamination field test. Greg Perry, President of EnviroServices, was present and observed the field testing on the USS Missouri.

The scope of work was to perform decontamination over two separate catwalks contaminated with PCBs. Each catwalk was 40 feet long and 3 feet wide. Catwalks were made of diamond steel plates that had been painted. It took two rounds of cleaning with pentanone, scrubbing and wiping to reach cleanup goal. Decontamination was not completed until the surfaces passed evaluation.

Estimates below include man hours for cleaning, scrubbing, application, removal and waste disposal for the actual job that had been previously performed at PHNSYandIMF and separately the job estimate utilizing DG. The total cost for each method has been divided into labor cost, cost of the products used, the hazmat suits and equipment, and the supplies.

Table 5. Cost Estimate for Cleaning Two 120 sq. ft Catwalks by Standard Cleaning Methods.

Labor	Qty		Unit	Unit Rate		Extension
HMT I	16		hrs	$65.00		$1,040.00
HMT I	16		hrs	$65.00		$1,040.00
HMT II	1		hrs	$75.00		$75.00
AA	0.5		hrs	$55.00		$27.50
PM	2		hrs	$125.00		$250.00
Subtotal						$2,432.50

Materials/ Equipment	Qty		Unit	Unit Rate		Extension
Truck	2		day	$110.00		$220.00
Tyvek	8		each	$12.00		$96.00
Gloves (outer)	16		pair	$5.00		$80.00
Clay Sorbent	10		bag	$15.00		$150.00
Triwal	1		each	$115.00		$115.00
Penatone/diesel	1		lot	$100.00		$100.00
Labels	2		each	$1.00		$2.00
Subtotal						$763.00

Vend/Trav/ Subs/Misc.	Qty		Unit	Unit Rate	Markup	Extension
					1.15	$0.00
					1.15	$0.00
Subtotal						$0.00

Transportation/ Disposal	Qty		Unit	Unit Rate		Extension
Transportation	1		TW	$275.00		$275.00
Disposal	1		TW	$1,170.00		$1,170.00
						$0.00
Subtotal						$1,445.00

Billing Summary			L/E/M/V Subtotals	$4,640.50
			Liability Ins. (3%)	$139.22
			Tax (4.712%)	$225.22
			BID TOTAL	$5,004.94

Table 6 below shows the same cost estimate for the same job if decontamination was performed with DG. These cost estimates for DG have been provided to us by the same subcontractor that performed the actual decontamination work via traditional cleaning with solvents and scrubbing.

Table 6. Cost Estimate for Decontaminating Two 120-Square Foot Catwalks with DG.

Labor	Qty		Unit	Unit Rate		Extension
HMT I	4		hrs	$65.00		$260.00
HMT I	4		hrs	$65.00		$260.00
HMT II	1		hrs	$75.00		$75.00
AA	0.5		hrs	$55.00		$27.50
PM	0.5		hrs	$125.00		$62.50
Subtotal						$685.00

Materials/ Equipment	Qty		Unit	Unit Rate		Extension
Truck	1		day	$110.00		$110.00
Tyvek	2		each	$12.00		$24.00
Gloves (outer)	2		pair	$5.00		$10.00
Clay Sorbent	0		bag	$15.00		$0.00
Triwal	1		each	$115.00		$115.00
Penatone/diesel	0		lot	$100.00		$0.00
Labels	2		each	$1.00		$2.00
Subtotal						$261.00

Vend/Trav/ Subs/Misc.	Qty		Unit	Unit Rate	Markup	Extension
					1.15	$0.00
					1.15	$0.00
Subtotal						$0.00

Transportation/ Disposal	Qty		Unit	Unit Rate		Extension
Transportation	1		TW	$60.00		$60.00
Disposal	1		TW	$235.00		$235.00
						$0.00
Subtotal						$295.00

Billing Summary			L/E/M/V Subtotals	$1,241.00
			Liability Ins. (3%)	$37.23
			Tax (4.712%)	$60.23
			BID TOTAL	$1,338.46

Results and cost analysis from an independent cleaning firm show that standard decontamination methods such as cleaning and scrubbing with a solvent correspond to $20.83/sq. ft. When DeconGel is used instead the cost drops to $5.58/sq. ft. This is a reduction in cost of more than 73% when DG is utilized to decontaminate machinery and bilge areas contaminated with PCBs.

This reduction in cost out exceeds the contract deliverable of reducing the cost by ≥25% over current standard methods of decontamination.

Based on the above estimates, standard decontamination methods such as cleaning and scrubbing with a solvent correspond to $20.83/sq. ft. When DG is used instead the cost drops to $5.58/sq. ft. This is a reduction in cost of more than 73% when DG is utilized to decontaminate machinery and bilge areas contaminated with PCBs. This reduction in cost out exceeds the contract deliverable of reducing the cost by ≥25% over current standard methods of decontamination.

5.3 Second Field Test: Building 6 at PHNSY (Lead Dust)

Lead compounds are used in batteries, bullets, as part of solders and alloys, and used to vulcanize rubber and as radiation shields. However, lead is a toxic heavy metal: upon exposure and ingestion/inhalation, lead and its compounds accumulate in soft tissue and bone, and act as potent neurotoxins.

Building 6 Foundry Complex at PHNSY is a 52,000-square-foot structure build in 1915 as a foundry facility for casting metal ship replacement parts. Foundry operations were discontinued in 1997. Currently the primary objective is remedial action to reduce or eliminate risk to human health under continued industrial or future commercial land scenarios. Amongst other contaminants Building 6 foundry complex contains significant amounts of dust waste comprised of heavy metals,

mostly lead. DG was evaluated as a decontamination method on a big open area of the smelter section composed of a variety of complex equipment and substrates such as a smelter, a metal circuit board, bare and painted concrete, bare and painted metal, and glass window surfaces.

While there are standards set by OSHA (Occupational Safety and Health Administration) for the amount of lead dust workers are allowed to be exposed to in the air, there are no legal standards for the amount of lead in surfaces in the workplace. However, OSHA, a government agency that protects workers, requires that wipe samples collected on surfaces in the eating areas in workplaces not exceed 200 micrograms/square foot (µg/ft^2). PHNSY&IMF agreed that this limit is more stringent than that required for remediation of Building 6 and that this limit could be used as a conservative estimated limit for the purpose of this field test intended to determine the suitability of using DG in the decontamination of lead dust in Building 6.

DG formulations (both sprayable and non-sprayable versions were evaluated) achieved excellent surface decontamination efficacy against lead dust. Decontamination Efficacies (DEs) were ≥85% for most of the areas tested. With the exception of the smelting equipment that is planned to be removed from the building, all other areas decontaminated had average post decontamination lead levels below the 200 µg/ft^2 specification. PHNSY&IMF supported both the use of the very conservative lead limits for work place eating areas as well as the apparent feasibility of the estimated costs. Based upon these estimates the contract deliverable has been exceeded.

During this field test, DG was also evaluated as a fixative/"leave on" product with great success for equipment that is to be removed before the decontamination project starts. Figures 9 through 13 below show photos taken during the field testing at Building 6.

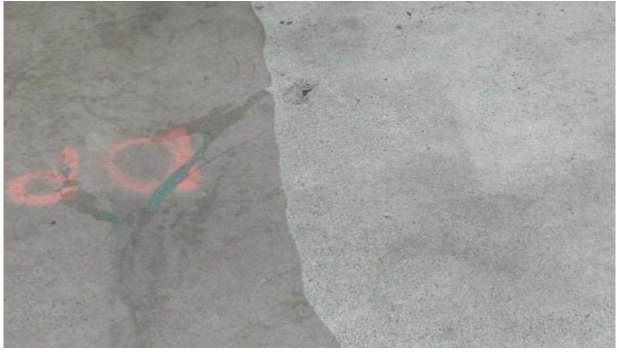
Figure 9. Overview of the large open floor area in Building 6 that DG was evaluated as a method of decontamination. The section consisted of a bare concrete area, a smelter with complex architecture, a control panel, painted concrete walls and glass windows.

Figure 10. Sampling before decontamination and application of DG (1121) by spraying on vertical and three-dimensional surfaces.

Figure 11. Application of DG (1101) by trowel (left photo) and brush (right photo).

Figure 12. Overview of the area that was decontaminated. DG was allowed to dry for 48 hrs before a second application.

Figure 13. Dried DG can be easily peeled off from a variety of surfaces including bare concrete, three dimensional control panels and equipment, windows and walls.



Excellent surface decontamination was achieved by applying DG 1101 and 1121 onto contaminated surfaces, resulting in encapsulation of lead-containing dust by DG's active components. Decontamination efficacy highlights, specifically the DEs obtained after the second application (avg. % values) of DG 1101 formulation against lead dust contaminant ranged from 95.7%+ and 95.9%+ (on bare metal surfaces with DG 1101 applied by brush and trowel respectively) to 81.6%+ and 87.8%+ (on bare concrete surfaces with DG 1101 applied by brush and trowel respectively) as determined by residual swipe analysis. Decontamination efficacy highlights, specifically the DEs (avg. DE% values) obtained after the second application of DG 1121 (sprayed version) against lead dust contaminant ranged from 95.6%+ (on painted metal), to 94.6%+ (on painted concrete with DG 1121 applied by spraying followed by brush) and 90.2%+ (on painted concrete with DG 1121 applied by spraying), to 92.1%+ (on glass windows), to 55.3%+ (on rusted bare metal) as determined by residual swipe analysis.

More than half of the areas evaluated showed remaining lead contamination below this limit after a single application of DG. As explained above, areas that remaining lead contamination was above this limit included surfaces (such as the smelting equipment) that proved to be very problematic to decontaminate due to the magnitude and type (heavy lead buildups cured over the years) of contamination on those areas. Even on such problematic substrates though decontamination efficacies achieved were higher than 85% after one application and higher than 95% after two applications (e.g., DG 1121 on horizontal and vertical surfaces on the smelter wheel, Table 12 below).

Tables 8 through 15 below summarize the average lead surface contamination (µg/ft^2) before and after decontamination with DG (both sprayable 1121 and non-sprayable 1101 versions) on a variety of surfaces. Decontamination efficacies for one and two applications of DG (average values) for each set of decontamination methods/surfaces that were evaluated are also provided. Application methods included spraying, brushing with a wire brush and troweling with a stand-up trowel.

Table 7. Surface lead contamination before and after decontamination with DG 1101 on bare metal surfaces (horizontal cap) and decontamination efficacies (average % values) for one and two applications of DG 1101 on such surfaces. Application of DG 1101 was done by brush.

Bare Metal Horizontal surfaces*	Lead Contamination before Decon (µg/ft^2)	Lead remaining After Decon 1st Application (µg/ft^2)	Lead remaining After Decon 2nd Application (µg/ft^2)	Decon Efficacy (%) 1st Application (Avg)	Decon Efficacy (%) 2nd Application (Avg)
DG 1101	1950.0 ± 450	460 ± 80	79.5 ± 0.5	**74.08 ± 10.08**	**95.7 ± 0.97**

* Two locations sampled.

Table 8. Surface lead contamination before and after decontamination with DG 1101 on bare metal surfaces (horizontal cap) and decontamination efficacies (average % values) for one and two applications of DG 1101 on such surfaces. Application of DG 1101 was done by trowel.

Bare Metal Horizontal surfaces*	Lead Contamination before Decon (µg/ft^2)	Lead remaining After Decon 1st Application (µg/ft^2)	Lead remaining After Decon 2nd Application (µg/ft^2)	Decon Efficacy (%) 1st Application (Avg)	Decon Efficacy (%) 2nd Application (Avg)
DG 1101	1600.0 ± 0	405 ± 15	65 ± 10	**74.69 ± 0.94**	**95.94 ± 0.63**

* Two locations sampled.

Table 9. Surface lead contamination before and after decontamination with DG 1101 on bare concrete surface and decontamination efficacies (average % values) for one and two applications of DG 1101 on such surfaces. Application of DG 1101 was done by brush.

Bare Concrete Horizontal surfaces*	Lead Contamination before Decon (µg/ft^2)	Lead remaining After Decon 1st Application (µg/ft^2)	Lead remaining After Decon 2nd Application (µg/ft^2)	Decon Efficacy (%) 1st Application (Avg)	Decon Efficacy (%) 2nd Application (Avg)
DG 1101	1150.0±792.8	337.7±158.6	129.5 ± 71.8	**52.80 ± 29.38**	**81.60 ± 14.99**

* Nine locations sampled.

Table 10. Surface lead contamination before and after decontamination with DG 1101 on bare concrete surface and decontamination efficacies (average % values) for one and two applications of DG 1101 on such surfaces. Application of DG 1101 was done by trowel.

Bare Concrete Horizontal surfaces*	Lead Contamination before Decon (µg/ft^2)	Lead remaining After Decon 1st Application (µg/ft^2)	Lead remaining After Decon 2nd Application (µg/ft^2)	Decon Efficacy (%) 1st Application (Avg)	Decon Efficacy (%) 2nd Application (Avg)
DG 1101	857 ± 471.7	233.4 ± 98.8	≤ 84.2 ± 42.5	**69.80 ± 11.06**	**≥ 87.84 ± 8.02**

* Ten locations sampled.

Table 11. Surface lead contamination before and after decontamination with DG 1121 on painted metal surfaces (smelter wheel compartment) and decontamination efficacies (average % values) for one and two applications of DG 1121 on such surfaces. Application of DG 1121 was done by spraying; brushing afterwards was performed on some areas.

Smelting equipment[1];Painted Metal Horizontal and Vertical surfaces*	Lead Contamination before Decon (µg/ft^2)	Lead remaining After Decon 1st Application (µg/ft^2)	Lead remaining After Decon 2nd Application (µg/ft^2)	Decon Efficacy (%) 1st Application (Avg)	Decon Efficacy (%) 2nd Application (Avg)
DG 1121	1140.0±7315.7	1170 ± 732.7	558 ± 537.7	**85.06 ± 7.90**	**95.63 ± 2.11**

* Three locations sampled.

1. PHNSY personnel has informed us that this type of equipment will be removed before the decontamination of Building 6 begins.

Table 12. Surface lead contamination before and after decontamination with DG 1121 on glass surfaces (windows) and decontamination efficacies (average % values) for two applications of DG 1121 on such surfaces. Application both by spraying and by brushing of DG 1121.

Windows; Glass Surfaces Vertical*	Lead Contamination before Decon (µg/ft^2)	Lead remaining After Decon 1st Application (µg/ft^2)	Lead remaining After Decon 2nd Application (µg/ft^2)	Decon Efficacy (%) 1st Application (Avg)	Decon Efficacy (%) 2nd Application (Avg)
DG 1121	296.6 ± 36.8	≤ 23.3 ± 0.5	≤ 23 ± 0	**≥ 92.0 ± 1.1**	**≥ 92.1 ± 1.0**

* Three areas sampled. Two of the areas were scrubbed with a wire brush after spraying DG 1121 on the windows. Scrubbing of the formulation showed no favorable effect on the decontamination efficacy of DG.

Table 13. Surface lead contamination before and after decontamination with DG 1121 on bare metal surfaces (rusty metal door) and decontamination efficacies (average % values) for two applications of DG 1121 on such surfaces.

Metal Door; Bare Metal Vertical surfaces*	Lead Contamination before Decon (µg/ft^2)	Lead remaining After Decon 1st Application (µg/ft^2)	Lead remaining After Decon 2nd Application (µg/ft^2)	Decon Efficacy (%) 1st Application (Avg)	Decon Efficacy (%) 2nd Application (Avg)
DG 1121	290.0 ± 40.0	260.0 ± 50	130 ± 20	**11.03 ± 4.97**	**55.27 ± 0.73**

* Two locations sampled.

The rusty condition of the metal door is believed to be the reason for the relatively low DE values (Table 14 above) shown on this location. DG has difficulty in efficiently decontaminating and peeling off from such surfaces. Nevertheless DG decontaminated the metal door to levels below the OSHA guidance as seen above.

Table 14. Surface lead contamination before and after decontamination with DG 1121 of painted concrete surfaces (vertical orientation) and decontamination efficacies (average % values) for two applications of DG 1121 on such surfaces.

Walls; Concrete Vertical surfaces*	Lead Contamination before Decon (µg/ft^2)	Lead remaining After Decon 1st Application (µg/ft^2)	Lead remaining After Decon 2nd Application (µg/ft^2)	Decon Efficacy (%) 1st Application (Avg)	Decon Efficacy (%) 2nd Application (Avg)
DG 1121	470.00 ± 120.00	67.00 ± 14.00	32.5 ± 1.50	**83.93 ± 7.08**	**92.51 ± 2.23**

* Two locations sampled. One of the areas was scrubbed with a wire brush after DG 1121 was sprayed on the wall. Scrubbing of the formulation showed no favorable effect on the decontamination efficacy.

Table 15 below summarizes decontamination efficacy data for all the different types of substrates/methods evaluated. All data acquired during this field test along with an area map of the locations of sampling are provided in the Appendix.

Table 15. Average Decontamination Efficacy after two applications (%) of DG 1101 (application by trowel) and DG 1121 (spray application) on a variety of substrates at Building 6.

Lead Contaminated Substrates	**Decontamination Efficacy (%) After 2 applications**	
	DG 1101 (trowel)	**DG 1121 (spray)**
Glass	**Not Conducted**	**> 92.12 ± 1.00[1,*]**
Bare Metal	**95.9 ± 0.63[2] / 95.7 ± 0.7[3]**	**55.3 ± 0.7[4]**
Painted Metal [4]	**Not Conducted**	**95.63 ± 2.11[5]**
Concrete (non-painted)[5]	**> 87.84 ± 8.02[6,*] / 81.60 ± 14.99[7]**	**Not Conducted**
Concrete (painted)[6]	**Not Conducted**	**90.20 ± 0[8] / 94.74 ± 0[9]**

: Analysis: Lead in Dust by Flame AAS (EPA Regulation SW 846 3050B/7000B), limit of detection (LOD) = 23 ug/ft^2, Pb-levels below LOD translate into DE values that are greater than (>) calculated values

1. Replicate samples; two samples were omitted because initial lead challenge levels were below the OSHA guidance limit of 200µg/ft^2, all vertical surfaces, non-scrubbed spraying DG 1121 hydrogel application
2. Replicate samples; Application by trowel of DG 1101. Horizontal surfaces.
3. Replicate samples; Application by brush of DG 1101. Horizontal surfaces.
4. Two replicate samples; Application by spraying of DG 1121. Vertical surfaces.
5. Replicate samples; Application by spraying of DG 1121. Two samples were omitted because initial lead challenge levels were below the OSHA guidance limit of 200µg/ft^2. Vertical surfaces.
6. 10 replicate samples; Application by trowel of DG 1101. Horizontal surfaces.
7. Replicate samples; Application by brush of DG 1101. Horizontal surfaces.
8. Replicate samples; two samples were omitted because initial lead challenge levels were below the OSHA guidance limit of 200µg/ft^2, all vertical surfaces, non-scrubbed spraying DG 1121 hydrogel application.
9. One sample; DG 1121 was scrubbed with a wire brush after been sprayed on the wall. Vertical surface.

Notes: Prior to and after decontamination with DG, test areas were swipe tested according to ASTM method E1728-03, "Standard Practice for Collection of Settled Dust Samples Using Wipe Sampling Methods for Subsequent Lead Determination". A GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC) was used as supplied (wetted with deionized water) for sampling of a square-foot of surface area.

All swipe samples were immediately contained in a capped 100 mL digestion polypropylene vessel and sent to an independent lab (LA Testing, Inc.; Garden Grove, CA) for Pb-level determination (EPA SW-846 Methods 3050B/7000B (Flame AAS Analysis)). Atomic absorption spectroscopy (AAS) is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of heavy metals and related compounds.

A sufficient amount of DG was either 1) poured and then troweled (1101) (and either let to stand until dry (non-scrubbed) or scrubbed into the surface with a small, handheld wirebrush), or 2) sprayed via paint sprayer (1121) (and either let to stand until dry (non-scrubbed) or scrubbed into the surface with a small, handheld wirebrush) to uniformly coat contaminated surfaces and ensure the formation of an easily peelable film upon DG drying. Applied DG was allowed to dry for 24-48 h, dried DG was then peeled off the contaminated surface, and the surface was immediately swipe tested.

Two applications of DG 1101 and 1121 were conducted in an effort to provide superior Pb-dust decontamination over what is achieved when utilizing a single DG film application on porous and non-porous surfaces. After the first decontamination round, a second application of DG was applied for both troweled and sprayed DG applications.

For some contaminated surfaces, DG was applied via scrubbing to facilitate physical/mechanical removal of lead deposits. No significant statistical difference in decontamination efficacy was noted between methods employing DG scrubbing and methods omitting the scrubbing technique (troweling or spraying).

The decontamination efficacy was calculated according to the following equation:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

5.4 Cost Analysis

A cost analysis for the decontamination project performed at Building 6 has been completed and can be seen in Table 17. Cost estimates for DG include man hours for cleaning, scrubbing, application, removal and waste disposal.

Table 16. Cost estimate for decontaminating 240 sq. ft of diverse surfaces (120 sq. ft of bare concrete floor, 60 sq. ft of painted wall and 60 sq. ft of glass painted metal) contaminated with lead by DG.

Labor	Qty		Unit	Unit Rate		Extension
HMT I	8		hrs	$65.00		$520.00
HMT I	8		hrs	$65.00		$520.00
HMT II	1		hrs	$75.00		$75.00
AA	0.5		hrs	$55.00		$27.50
PM	0.5		hrs	$125.00		$62.50
Subtotal						$1,205.00

Materials/ Equipment	Qty		Unit	Unit Rate		Extension
Truck	1		day	$110.00		$110.00
Tyvek	4		each	$12.00		$48.00
Gloves (outer)	4		pair	$5.00		$20.00
Triwal	2		each	$115.00		$230.00
DG	9.7		gal	$106.67		$1,034.70
Labels	4		each	$1.00		$4.00
Subtotal						$1,446.70

Transportation/ Disposal	Qty		Unit	Unit Rate		Extension
Transportation	1		TW	$60.00		$60.00
Disposal	1		TW	$235.00		$235.00
						$0.00
Subtotal						$295.00

Billing Summary			L/E/M/V Subtotals	$2,946.70
			Liability Ins. (3%)	$88.40
			Tax (4.712%)	$143.01
			BID TOTAL	$3,178.11

Table 17 below shows the amount (in gallons) and coverage for DG for this type of work; coverage numbers are based on the field test performed at PHNSY-IMF Building 6 Foundry complex.

Table 17. Amounts and coverage of DG for 240 sq. ft of surfaces of diverse nature contaminated with lead.

Square footage (sq. ft)	Substrate Type	Coverage (sq. ft/Gallons)	DG (Gallons)
120	bare concrete floor	35	3.43
60	painted walls	75	0.8
60	glass/bare metal/painted metal	100	0.6
		Total gallons per application	4.83
		Total gallons for two applications	9.66

This estimate is based upon the listed assumptions that the job consists of the exact area definitions and the coverage required to coat those areas. An estimated job consisting of 50% porous bare concrete, 25% glass/painted metal/bare metal, and 25% painted walls was estimated to cost $13.24.

DG formulations (both sprayable and non-sprayable versions were evaluated) achieved excellent surface decontamination efficacy against lead dust. Decontamination Efficacies (DEs) were ≥85% for most of the areas tested. With the exception of the smelting equipment that is planned to be removed from the building, all other areas decontaminated had average post decontamination lead levels below the 200 $\mu g/ft^2$ specification. An estimated job consisting of 50% porous bare concrete, 25% glass/painted metal/bare metal, and 25% painted walls was estimated to cost $13.24. Note that PHNSY&IMF previously determined the decontamination of Building 6 to be cost infeasible. A comparison that can be made is the $20.83/sq. ft for decontaminating PCB oil from nonporous steel which would result in a >36% in estimated cost savings. Vicky N. Ah Quin, NAVSEA L6SS Black Belt, PHNSY Engineering & Planning Department, C200L was our assigned technical liaison at PHNSY&IMF and approved the use of the 200 $\mu g/ft^2$ target limit for lead dust surface contamination and use of the cost models from EnviroServices, a PHNSY&IMF subcontractor.

PHNSY&IMF approved both the use of the very conservative lead limits for work place eating areas as well as the feasibility of the estimated costs. Based upon these estimates the contract deliverable has been exceeded.

5.5 Third Field Test: JPEO-CBD Testing on Live Chemical Warfare Agents

Significant results were produced in “in house” Chemical Warfare Agent (CWA) simulant decontamination tests. This work performed under the direction of the Joint Program Executive Office for Chemical and Biological Defense (JPEO-CBD), resulted in the JPEO-CBD performing testing of DG on classified live CWAs at their own expense. This work constitutes field testing as this is the appropriate venue for CWA field testing. The official data could not be released because the testing was performed on classified emerging threat agents. JPEO-CBD informed us that DG performed exceptionally well as a physical decontamination method for classified emerging threat agents on sorbent substrates such as rubber and Chemical Agent Resistant Coating (CARC). Although the efficacy of the current DG formulation was not sufficient to be a total decontamination solution, they recommended that we combine the significant ability of DG to draw CWAs out of sorbent substrates and emulsify them (both of which increase the access of chemical neutralizers to the CWA) with direct CWA neutralization technologies that do not have the ability to pull CWAs out of the substrates in which they have absorbed.

In house evaluations of DG on Chemical Warfare Agent Simulant contaminants (with associated evaluated substrates) included:

1) Methyl Benzoate (VX-Simulant)- VX, an organo-phosphono-thioate, is a toxic nerve agent classified as a weapon of mass destruction by the United Nations. VX can be distributed as a liquid or aerosol, both pure and as a mixture, and due to its high viscosity and low volatility is considered an environmentally persistent biohazard. VX and its simulant methyl benzoate (MBz) have near identical Water:Octanol Coefficients (2.09 and 2.12, respectively), and as such behave similarly in both environmental settings and biological systems (evaluated on cadmium-plated, carbon steels, CARC, silicone and black rubbers substrates);

2) CEES (2-Chloroethyl Ethyl Sulfide) (Sulfur Mustard-Simulant) - Sulfur Mustard, bis-(2-chloroethyl) sulfide, is a powerful vesicant, cytotoxic agent regulated under the 1993 Chemical Weapons Convention (CWC). Sulfur mustard can be deployed by spraying, or more typically distributed via artillery shells and bombs. For experimental testing purposes, the half-mustard 2-chloroethyl ethyl sulfide (CEES) is used as an effective yet less toxic simulant for sulfur mustard chemical warfare agents (evaluated on cadmium-plated, carbon steels, CARC, silicone and black rubbers substrates); and

3) CEPS ((2-Chloroethyl Phenyl Sulfide) (Sulfur Mustard-Simulant) - Sulfur Mustard, bis-(2-chloroethyl) sulfide, is a powerful vesicant, cytotoxic agent regulated under the 1993 Chemical Weapons Convention (CWC). Sulfur mustard can be deployed by spraying, or more typically distributed via artillery shells and bombs. For experimental testing purposes, the half-mustard 2-chloroethyl phenyl sulfide (CEPS) is used as an effective yet less toxic simulant for sulfur mustard chemical warfare agents (evaluated on cadmium-plated, carbon steels, CARC, silicone and black rubbers substrates).

Table 18 below summarizes decontamination efficacy evaluations performed in house of DG against CWA simulants (VX and mustard gas simulants).

Table 18. Decontamination Efficacy evaluations of DG against CWA simulants on multiple surfaces as determined via Direct Gel Analysis and residue analysis after solvent extraction.

Substrate Evaluated	Decontamination Efficacy (%) via Extraction[1] Method
VX-Simulant (Methyl Benzoate)	
Cd-plated Steel	96.7 ± 0.19
Carbon Steel	99.2 ± 0.11
CARC**	67.3 ± 2.34
Silicone Rubber**	62.3 ± 1.17
Black Rubber**	26.5 ± 1.88
Sulfur Mustard-Simulant (2-Chloroethyl Ethyl Sulfide (CEES))	
Cd-plated Steel	ND
Carbon Steel	ND
CARC**	28.6 ± 0.0
Silicone Rubber**	54.6 ± 0.20
Black Rubber**	93.2 ± 0.12
Sulfur Mustard-Simulant (2-Chloroethyl Phenyl Sulfide (CEPS))	
Cd-plated Steel	96.3 ± 0.1
Carbon Steel	97.2 ± 0.1
CARC**	51.2 ± 0.51
Silicone Rubber**	51.9 ± 0.32
Black Rubber**	76.9 ± 0.11

*Contaminant residue amount below limit of detection (LOD)
ND: no contaminant detected for samples and/or controls

1. Extraction method of analysis: use of solvent extraction
2. DG 1101 was poured onto the contaminated surface

5.6 Cost Analysis

This work, performed under this contract and under the direction of the JPEO-CBD, positioned our successful acquisition of a contract with the Telemedicine and Advanced Technology Research Center *(*TATRC) to incorporate chemical and biological neutralization capability into our hydrogel (DG) technology with the ultimate objective being the development a single chemical biological radiological and nuclear (CBRN) decontamination technology. No cost model is provided for the CWA decontamination work performed under this contract or for the work performed by JPEO-CBD because the decontamination performance was not sufficient to be considered a final solution. However, as there is no current product that can perform this decontamination on sorbent substrates, the cost of decontamination of CWAs is a secondary consideration with the development of an effective decontamination solution being the primary consideration. As there is no current method for decontamination of CWAs in sorbent substrates, no cost comparison can be calculated. Based upon the positive results and verification of value to the military by the JPEO-CBD and TATRC the contract deliverable for this field test has been exceeded.

APPENDICES

Appendix A

6. Individual Technical and End-User Reports of a Range of Toxic Industrial Chemicals and Chemicals of Concern

6.1. Technical Report for Aldehydes (Glutaraldehyde)

TITLE: Surface Decontamination of Aldehydes (Glutaraldehyde) by DeconGel 1101

ANALYST: Geoff Nadolski PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel formulation 1101 on stainless steel, aluminum, and concrete surfaces contaminated with Aldehydes (glutaraldehyde) using GC/MS (Gas Chromatography/Mass Spectrometry) following Environmental Protection Agency (EPA) SW-846 Methods 3500C (Sampling) and 8270C (Analysis).

HAZARDOUS MATERIALS RELEVANCE

Glutaraldehyde is a volatile liquid aldehyde used as a disinfectant, fixative, and plastics crosslinker. Glutaraldehyde is a strong and toxic disinfectant and can cause severe mucosal membrane irritation. Glutaraldehyde was chosen as a representative aldehyde; DeconGel is expected to have similar efficacy towards the wide range of aldehydes

SUMMARY RESULTS:

- Excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with aldehydes (Glutaraldehyde) resulting in encapsulation of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 100% (on concrete) to 100% (on stainless steel) to 100% (on aluminum) for Glutaraldehyde determined by residual swipe analysis, and 77.5% (on concrete) to 92.2% (on stainless steel) to 92.4% (on aluminum) as determined by direct DeconGel analysis.
- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of organic compounds dissolved in a suitable solvent able to completely solvate aniline as well as DeconGel components. When necessary, experimental methods were customized to afford complete dissolution of organic contaminants. Additionally, analytical methods and associated equipment (GC column, GC temperature gradient program, MS sample ionization parameters) were appropriately developed to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 and 2 show the decontamination efficacies of DeconGel 1101 on stainless steel, aluminum, and concrete surfaces contaminated with Aldehydes (glutaraldehyde) as determined by residual swipe testing and direct DeconGel analysis.

Table 1. Decontamination efficacy of DeconGel 1101 on Glutaraldehyde Aldehyde contaminated stainless steel, aluminum, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	805.32 ± 0.91
	Residual	ND*
	Decon. Efficacy (%)	**100****
Aluminum	Control	805.03 ± 2.28
	Residual	ND
	Decon. Efficacy (%)	**100****
Concrete	Control	654.15 ± 3.76
	Residual	ND
	Decon. Efficacy (%)	**100****

1222x dilution factor for samples and controls

* ND: not detected, limit of detection (LOD) for glutaraldehyde approximates 250 ppb

** An ND value for residual experiments results in a Decontamination Efficacy of 100%

Table 2. Decontamination efficacy of DeconGel 1101 on Glutaraldehyde Aldehyde contaminated stainless steel, aluminum, and concrete surfaces as determined by direct DeconGel analysis.

Direct DeconGel Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	858.02 ± 1.15
	Encapsulated in Gel	791.53 ± 0.91
	Decon. Efficacy (%)	**92.2 ± 0.23**
Aluminum	Control	856.89 ± 0.44
	Encapsulated in Gel	791.39 ± 1.76
	Decon. Efficacy (%)	**92.4 ± 0.16**
Concrete	Control	856.33 ± 2.0
	Encapsulated in Gel	663.87 ± 1.75
	Decon. Efficacy (%)	**77.5 ± 0.46**

1222x dilution factor for samples and controls

METHOD DEVELOPMENT OPTIMIZATIONS/OBSERVATIONS:

- Contaminant liquid greater than 100 uL were difficult to spread evenly throughout coupon surfaces, forming zones of liquid saturation which tended to overload poured DeconGel, significantly limiting the ability of DeconGel to encapsulate/emulsify surface contamination. This issue was circumvented by applying a modest amount (90 uL, 50% glutaraldehyde in water) of contaminant liquid throughout the surface of interest using a small brush. Application of a homogenous, thin layer of contaminant on the respective substrate facilitated an optimized interaction between contaminant and DeconGel, and provided an accurate measure of DeconGel's decontamination efficacy in a scaled-down yet real-world setting. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel. Glutaraldehyde readily dissolves in acetonitrile (50 mL) used to prepare all analytical samples and controls.

- Acceptable direct DeconGel decontamination efficacies ranging from 77.5 to 92.4% for DeconGel 1101 were achieved, however, because glutaraldehyde is prone to both evaporate and react/decompose over the required 24 h drying time needed by DeconGel once applied to a contaminated surface, this could contribute to lower than expected DeconGel decontamination efficacies determined by the direct DeconGel testing method.

- ASTM method E1728-03, a standardized swipe testing method used for sampling of contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. Air-dried GhostWipe™

(Environmental Express; Mt. Pleasant, SC) swipes were utilized in this swipe testing method.

- Direct chemical analysis of the dried peeled DeconGel samples was also utilized to provide an improved understanding of DeconGel's decontamination efficacy.
- Standardized EPA SW-846 Sampling Method 3500C "Organic Extraction and Sample Preparation" was followed as a guideline to prepare all samples and controls. All samples, controls, and standards were prepared using the same solvent and appropriate experimental conditions to ensure accurate and GC/MS instrumental analysis.
- GC/MS instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of organic compounds. Standardized EPA SW-846 Analytical Method 8270C "SEMIVOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)" was followed as a guideline to prepare all samples and controls.
- To ensure accurate determination of DeconGel decontamination efficacy, a standard curve of the analyte of interest was prepared using sufficiently pure analyte; the respective standards were diluted to a known concentration (ppm) using the same solvent as used for samples and controls.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

Decontamination Efficacy (DeconGel Testing) =

(Contaminant (ppm) of DeconGel Direct/Contaminant (ppm) of DeconGel Control) x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 90 uL (50% glutaraldehyde in water) of contaminant was evenly applied via brushing on 1) aluminum (surface area: 56.3 cm^2), 2) stainless steel (surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using an air-dried GhostWipe™ swipe

(Environmental Express; Mt. Pleasant, SC). Swipe and gel samples were suspended in 50 mL acetonitrile for 24 h. Samples were then analyzed via GC/MS (see below).

Controls Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied via brushing on 1) aluminum (surface area: 56.3 cm^2), 2) stainless steel (surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 50 mL acetonitrile for 24 h and analyzed via GC/MS (see below).

For DeconGel Control samples, a respectable amount of contaminant and approximately 6.0 g of dry DeconGel 1101 (pre-poured gel on the respective uncontaminated substrate and let dry for 24 h) was suspended in 50 mL acetonitrile for 24 h and analyzed via GC/MS (see below).

Reagents and Standards

Glutaraldehyde (pentane-1,5-dial) (CAS# 111-30-8, Fisher Scientific; Fair Lawn, NJ) was used as received.

Analytical Instrumentation

A Thermo DSQII GC/MS with autosampler in positive mode was used to determine glutaraldehyde concentration (ppm, wt/wt) of all samples and controls, using a Restek (Bellefonte, PA) Rtx-5 capillary column (30 m x 0.25 mm, 0.5 um).

A 7-point standard curve derived from three independently prepared stock solutions was prepared using DMSO as the working solvent. The calibration curve exhibits a curve fitting as approximated by the coefficient of determination of linear regression R^2, where $R^2 = 1.0$ (see Figure 1).

GC method: start at 45 ^oC, hold for 1 min, ramp at 30 ^oC/min to 320 ^oC, hold 10 min.

Glutaraldehyde GC/MS data: 4.3 min; $M^+ = 100$.

6.2. Technical Report for Alkaloids (Nicotine)

TITLE: Surface Decontamination of Alkaloids (Nicotine) by DeconGel 1101

ANALYST: Geoff Nadolski PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel formulation 1101 on stainless steel, aluminum, and concrete surfaces contaminated with Alkaloids (nicotine) using LC/MS (Liquid Chromatography/Mass Spectrometry) following Environmental Protection Agency (EPA) SW-846 Methods 3500C (Sampling) and 8321B (Analysis).

HAZARDOUS MATERIALS RELEVANCE: Nicotine is an aromatic (pyridine derivative) alkaloid produced by the nightshade family of plants such as tobacco. Nicotine stimulates production of cytochrome P450 liver enzymes during metabolism and possesses high oral and topical toxicities in comparison to other alkaloids. Nicotine was chosen as a representative alkaloid; DeconGel is expected to have similar efficacy towards the wide range of alkaloids.

SUMMARY RESULTS:

- Excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with alkaloids (nicotine) resulting in encapsulation of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 99.5% (on concrete) to 99.8% (on aluminum) to 99.8% (on stainless steel) for Nicotine determined by residual swipe analysis, and 88.0% (on concrete) to 97.6% (on stainless steel) to 97.7% (on stainless steel) for Nicotine as determined by direct DeconGel analysis.
- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of organic compounds dissolved in a suitable solvent able to completely solvate nicotine as well as DeconGel components. When deemed necessary, experimental methods were customized to afford complete dissolution of organic contaminants. Additionally, analytical methods and associated equipment (LC column, LC gradient program, MS sample ionization parameters) were appropriately developed to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 and 2 show the decontamination efficacies of DeconGel 1101 on stainless steel, aluminum, and concrete surfaces contaminated with Alkaloids (nicotine) as determined by residual swipe testing and direct DeconGel analysis.

Table 1. Decontamination efficacy of DeconGel 1101 on Nicotine Alkaloid contaminated stainless steel, aluminum, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	209.74 ± 1.49
	Residual	0.36 ± 0.005
	Decon. Efficacy (%)	**99.8 ± 0.0**
Aluminum	Control	209.85 ± 1.75
	Residual	0.30 ± 0.007
	Decon. Efficacy (%)	**99.8 ± 0.0**
Concrete	Control	152.94 ± 1.51
	Residual	0.77 ± 0.001
	Decon. Efficacy (%)	**99.5 ± 0.0**

2200x dilution factor for samples and controls

Table 2. Decontamination efficacy of DeconGel 1101 on Nicotine Alkaloid contaminated stainless steel, aluminum, and concrete surfaces as determined by direct DeconGel analysis.

Direct DeconGel Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	210.67 ± 1.05
	Encapsulated in Gel	205.55 ± 0.93
	Decon. Efficacy (%)	**97.6 ± 0.15**
Aluminum	Control	210.17 ± 1.23
	Encapsulated in Gel	205.82 ± 1.20
	Decon. Efficacy (%)	**97.7 ± 0.73**
Concrete	Control	209.44 ± 0.98
	Encapsulated in Gel	184.08 ± 1.09
	Decon. Efficacy (%)	**88.0 ± 0.40**

2200x dilution factor for samples and controls

METHOD DEVELOPMENT OPTIMIZATIONS/OBSERVATIONS:

- We noted that amounts of contaminant liquid greater than 100 uL were difficult to spread evenly throughout coupon surfaces, forming zones of liquid saturation which tended to overload poured DeconGel, significantly limiting the ability of DeconGel to encapsulate/emulsify surface contamination. This issue was circumvented by applying a modest amount (25 uL) of contaminant liquid throughout the surface of interest using a small brush. Application of a homogenous, thin layer of contaminant on the respective substrate facilitated an optimized interaction between contaminant and DeconGel, and provided an accurate measure of DeconGel's decontamination efficacy in a scaled-down yet real-world setting. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel. Nicotine readily dissolves in DMSO (50 mL) used to prepare all analytical samples and controls.

- ASTM method E1728-03, a standardized swipe testing method used for sampling of contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. Air-dried GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes (pre-wetted with DMSO immediately prior to performing swipe analysis) were utilized in this swipe testing method.

- Direct chemical analysis of the dried peeled DeconGel samples was also utilized to provide an improved understanding of DeconGel's decontamination efficacy.

- Standardized EPA SW-846 Sampling Method 3500C "Organic Extraction and Sample Preparation" was followed as a guideline to prepare all samples and controls. All samples, controls, and standards were prepared using the same solvent and appropriate experimental conditions to ensure accurate and LC/MS instrumental analysis.

- LC/MS instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of organic compounds. Standardized EPA SW-846 Analytical Method 8321B "Solvent-Extractable Nonvolatile Compounds by High-Performance Liquid Chromatography/Thermospray/Mass Spectrometry (HPLC/TS/MS) or Ultraviolet (UV) Detection" was followed as a guideline to prepare all samples and controls.

- To ensure accurate determination of DeconGel decontamination efficacy, a standard curve of the analyte of interest was prepared using sufficiently pure analyte; the respective standards were diluted to a known concentration (ppm) using the same solvent as used for samples and controls.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

Decontamination Efficacy (DeconGel Testing) =

(Contaminant (ppm) of DeconGel Direct/Contaminant (ppm) of DeconGel Control) x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 25 uL of contaminant was evenly applied via brushing on 1) stainless steel (surface area: 56.3 cm^2), 2) aluminum (surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe and gel samples were suspended in 50 mL DMSO for 24 h. Samples were then analyzed via LC/MS (see below).

Controls Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied via brushing on 1) stainless steel (surface area: 56.3 cm^2), 2) aluminum (surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 50 mL DMSO for 24 h and analyzed via LC/MS (see below).

For DeconGel Control samples, a respectable amount of contaminant and approximately 6.0 g of dry DeconGel 1101 (pre-poured gel on the respective uncontaminated substrate and let to dry for 24 h) was suspended in 50 mL DMSO for 24 h and analyzed via LC/MS (see below).

Reagents and Standards

Nicotine (3-[(2S)-1-methylpyrrolidin-2-yl]pyridine) (CAS# 54-11-5, Fisher Scientific; Fair Lawn, NJ) was used as received.

Analytical Instrumentation

A Thermo LCQ LC/MS with autosampler in positive mode was used to determine nicotine concentration (ppm, wt/wt) of all samples and controls, using a Grace Davison (Deerfield, IL) C18-Select analytical column (150 x 4.6 mm, 5 um).

A 6-point standard curve derived from three independently prepared stock solutions was prepared using DMSO as the working solvent. .

LC method using A= water (0.1% formic acid), B=acetonitrile (0.1% formic acid); start at 90%A to 2 min at 70%A, to 3 min at 50%A, to 5 min at 30%A, to 6 min at 100%B, hold until 16 min at 90% A hold until 17 min.

Nicotine LC/MS data: 1.4 min; lambda max = 250, 273 nm; M^+ = 162.

6.3. Technical Report for Aluminum Compounds (Aluminum powder, Aluminum Oxide, Aluminum Chloride, and Aluminum Potassium Sulfate)

TITLE: Surface Decontamination of Aluminum Compounds (Elemental Aluminum Powder (Al), Aluminum Oxide (Al_2O_3), Aluminum Chloride ($AlCl_3$) and Aluminum Potassium Sulfate ($AlK(SO_4)_2$)) by DeconGel 1101

AUTHOR: Andreas Mylonakis PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with Aluminum Compounds (elemental aluminum powder (Al), aluminum oxide (Al_2O_3), aluminum chloride ($AlCl_3$), and aluminum potassium sulfate ($AlK(SO_4)_2$)) using ICP-OES (Inductively Coupled Plasma-Optical Emission Spectroscopy) following Environmental Protection Agency (EPA) SW-846 Methods 3005A (sampling) and 6010C (analysis).

HAZARDOUS MATERIALS RELEVANCE: Aluminum and its compounds are widely used in a number of industries including consumer goods such as cosmetics and aluminum cans. Aluminum compounds can become easily airborne and as such are considered inhalation hazards. Aluminum powder, aluminum oxide, aluminum chloride, and aluminum potassium sulfate were chosen as representative aluminum compounds for evaluating DeconGel's efficacy; DeconGel is expected to have similar efficacy towards the wide range of aluminum compounds.

SUMMARY RESULTS:

Excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with aluminum compounds resulting in encapsulation of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 for aluminum compounds (except aluminum powder elemental which is corrosive and reacts with the metal surfaces evaluated) ranged from greater than 93.0% (on concrete) to greater than 95.4% (on carbon steel) to greater than 96.4% (on stainless steel) as determined by residual swipe analysis, and from greater than 81.1% (on concrete) to greater than 93.0% (on carbon steel) to greater than 95.1% (on stainless steel) as determined by direct DeconGel analysis.

Aluminum powder is reactive to some metal surfaces including carbon steel and stainless steel. DeconGel could not completely remove aluminum elemental powder that reacted with and created a fixed deposit on steel surfaces. Nevertheless, DeconGel showed excellent decontamination efficacy on loose aluminum powder contaminant from such surfaces.

Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of inorganics in aqueous samples. When necessary, the digestion methods were customized to result in the complete dissolution of the inorganic contaminants and to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 through 8 show the decontamination efficacies of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with elemental aluminum powder (Al), aluminum oxide (Al_2O_3), aluminum chloride ($AlCl_3$) and aluminum potassium sulfate ($AlK(SO_4)_2$) as determined by residual swipe testing and direct DeconGel analysis.

Table 1. Decontamination efficacies of DeconGel 1101 on aluminum powder (Al) contaminated stainless steel, carbon steel, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation		
		DeconGel 1101[a]	**DeconGel 1101[b]**	**DeconGel 1101[c]**
Stainless Steel*	Control	1113.0 ± 17.0	NA	NA
	Residual	23.3 ± 5.3	NA	NA
	Decon. Efficacy (%)	**97.9 ± 2.0**	**NA**	**NA**
Carbon Steel*	Control	944.7 ± 0.2	NA	NA
	Residual	4.7 ± 4.3	NA	NA
	Decon. Efficacy (%)	**99.5 ± 4.6**	**NA**	**NA**
Concrete	Control	1014.0 ± 4.0	423.3 ± 34.8	511.7 ± 12.7
	Residual	60.4 ± 17.0	4.2 ± 0.8	1.8 ± 0.2
	Decon. Efficacy (%)	**94.0 ± 2.8**	**99.0 ± 8.5**	**99.6 ± 3.8**

2280x dilution factor for samples and controls

*Aluminum powder reacted with these surfaces creating a fixed residue on the contaminated surface that could not be completely removed by DeconGel

NA: not applicable

a. 100 mg of contaminant was applied on the substrate.
b. 50 mg of contaminant was applied on the substrate.
c. 50 mg of contaminant was applied on the substrate; a fresh standard was made.

Table 2. Decontamination efficacies of DeconGel 1101 on aluminum powder (Al) contaminated stainless steel, carbon steel and concrete surfaces as determined by direct DeconGel analysis.

Direct DeconGel Analysis (ppm)		Formulation
		DeconGel 1101
Stainless Steel*	Control	514.1 ± 24.4
	Encapsulation in Gel	291.3 ± 16.2
	Decon. Efficacy (%)	**43.3 ± 3.8**
Carbon Steel*	Control	528.0 ± 16.1
	Encapsulation in Gel	400.2 ± 49.0
	Decon. Efficacy (%)	**75.8 ± 9.6**
Concrete	Control	400.5 ± 37.5
	Encapsulation in Gel	381.5 ± 27.8
	Decon. Efficacy (%)	**95.2 ± 11.2**

2280x dilution factor for samples and controls
*Aluminum powder reacted with these surfaces creating a fixed residue on the contaminated surface that could not be completely removed by DeconGel.

Table 3. Decontamination efficacies of DeconGel against aluminum oxide (Al_2O_3) contaminated stainless steel, carbon steel, and concrete surfaces as determined by residual swipe testing method.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	420.8 ± 7.0
	Residual	1.4 ± 0.6
	Decon. Efficacy (%)	**99.7 ± 7.7**
Carbon Steel	Control	505.5 ± 22.4
	Residual	0.3 ± 0.3
	Decon. Efficacy (%)	**99.9 ± 14.4**
Concrete	Control	420.2 ± 6.1
	Residual	1.3 ± 0.7

	Decon. Efficacy (%)	**99.7 ± 5.9**

2280x dilution factor for samples and controls

Table 4. Decontamination efficacies of DeconGel against aluminum oxide (Al_2O_3) contaminated stainless steel, carbon steel and concrete surfaces as determined by direct DeconGel analysis.

Direct DeconGel Analysis (ppm)		**Formulation**	
		DeconGel 1101[a]	**DeconGel 1101[b]**
Stainless Steel	Control	501.3 ± 11.6	NA
	Encapsulated in Gel	506.7 ± 20.0	NA
	Decon. Efficacy (%)	**101.1 ± 4.6**	**NA**
Carbon Steel	Control	477.2 ± 56.5	NA
	Encapsulated in Gel	443.8 ± 50.0	NA
	Decon. Efficacy (%)	**93.0 ± 15.2**	**NA**
Concrete	Control	494.5 ± 26.7	9685.9 ± 125.7*
	Encapsulated in Gel	254.8 ± 23.3	8700.9 ± 49.8*
	Decon. Efficacy (%)	**51.5 ± 5.5**	**90.1 ± 1.9**

2280x dilution factor for samples and controls

* ICP-OES readings have been normalized with regards to the amount (g) of contaminant applied on the surface and are reported in "ppm/g of contaminant applied". Refer to "Materials and Method" section for more details.

a. 50mg of contaminant was applied onto the surface before DeconGel 1101 application.

b. Contaminant amounts ranging from 0.0088g to 0.0136g were evenly brushed onto concrete before DeconGel 1101 application.

Table 5. Decontamination efficacies of DeconGel 1101 against aluminum chloride ($AlCl_3$) contaminated stainless steel, carbon steel, and concrete surfaces as determined by residual swipe testing method.

Swipe Testing (ppm)		**Formulation**
		DeconGel 1101
Stainless Steel	Control	325.0 ± 7.9
	Residual	3.6 ± 2.3
	Decon. Efficacy (%)	**98.9 ± 0.7**
Carbon Steel	Control	319.2 ± 18.4
	Residual	4.9 ± 1.4
	Decon. Efficacy (%)	**98.5 ± 5.9**
Concrete	Control	369.4 ± 73.7
	Residual	25.8 ± 4.4
	Decon. Efficacy (%)	**93.0 ± 18.7**

2280x dilution factor for samples and controls

Table 6. Decontamination efficacies of DeconGel 1101 against aluminum chloride ($AlCl_3$) contaminated stainless steel, carbon steel and concrete surfaces as determined by direct DeconGel analysis.

Direct DeconGel Analysis (ppm)		**Formulation**		
		DeconGel 1101 (1st Application)	**DeconGel 1101[a] (2nd Application)**	**DeconGel 1101[b]**
Stainless Steel*	Control	411.3 ± 2.3	355.4 ± 4.8	388.9 ± 22.5
	Encapsulation in Gel	373.6 ± 17.2	340.9 ± 6.3	419.6 ± 8.8
	Decon. Efficacy (%)	**90.8 ± 4.6**	**95.9 ± 8.5**	**107.8 ± 6.6**
Carbon Steel*	Control	485.9 ± 8.7	NA	362.8 ± 29.9
	Encapsulation in Gel	409.4 ± 14.8	NA	384.4 ± 34.5
	Decon. Efficacy (%)	**84.3 ± 17.6**	**NA**	**105.9 ± 12.9**
Concrete	Control	545.6 ± 3.4	485.9 ± 3.2	521.7 ± 10.4
	Encapsulation in Gel	401.8 ± 7.0	426.1 ± 15.4	256.3 ± 60.7
	Decon. Efficacy (%)	**73.6 ± 22.2**	**87.7 ± 13.4**	**50.8 ± 11.8**

2280x dilution factor for samples and controls

a. A second coating of DeconGel 1101 was applied. Decon Efficacy values correspond to the total of decontamination efficacy after two DeconGel applications. The carbon steel control sample spilled resulting in no decontamination efficacy determination.

b. Samples and associated controls were completed in triplicate. DeconGel was applied roughly 30 minutes after applying $AlCl_3$.

Table 7. Decontamination efficacies of DeconGel 1101 on aluminum potassium sulfate ($AlK(SO_4)_2$) contaminated stainless steel, carbon steel, and concrete surfaces as determined by residual swipe testing method.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	441.8 ± 20.7
	Residual	15.9 ± 3.9
	Decon. Efficacy (%)	**96.4 ± 4.9**
Carbon Steel	Control	442.0 ± 13.6
	Residual	20.4 ± 7.4
	Decon. Efficacy (%)	**95.4 ± 4.2**
Concrete	Control	399.8 ± 47.5
	Residual	20.1 ± 3.3
	Decon. Efficacy (%)	**95.0 ± 11.4**

2280x dilution factor for samples and controls

Table 8. Decontamination efficacies of DeconGel 1101 on aluminum potassium sulfate ($AlK(SO_4)_2$) contaminated stainless steel, carbon steel, and concrete surfaces as determined by direct DeconGel analysis.

Direct Gel Analysis (ppm)		Formulation	
		DeconGel 1101	DeconGel 1101*
Stainless Steel	Control	553.9 ± 7.9	480.9 ± 12.4
	Encapsulated in Gel	509.9 ± 6.9	457.4 ± 31.4
	Decon. Efficacy (%)	**92.0 ± 1.2**	**95.1 ± 7.0**
Carbon Steel	Control	NA	493.1 ± 10.6
	Encapsulated in Gel	NA	466.3 ± 17.0
	Decon. Efficacy (%)	**NA**	**94.6 ± 4.0**
Concrete	Control	473.9 ± 1.9	485.2 ± 19.6
	Encapsulated in Gel	388.1 ± 42.1	345.4 ± 32.4
	Decon. Efficacy (%)	**81.9 ± 8.9**	**71.2 ± 7.3**

2280x dilution factor for samples and controls

* DeconGel was applied roughly 30 minutes after applying $AlCl_3$.

METHOD DEVELOPMENT OPTIMIZATIONS/OBSERVATIONS:

Amounts of contaminant solid greater than 0.05 g were difficult to spread evenly throughout coupon surfaces, forming clumps and piles which tended to overload poured DeconGel, significantly limiting the ability of DeconGel to encapsulate/emulsify surface contamination. This issue was circumvented by applying a modest amount (0.05 g) of contaminant solid throughout the surface of interest. Application of a homogenous, thin layer of the aluminum contaminants on the respective substrate facilitated an optimized interaction between contaminant and DeconGel; and it provided an accurate measure of DeconGel's decontamination efficacy in a scaled-down yet real-world setting. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel. Both Al_2O_3 and Al powder readily dissolve in aqueous acidic solutions used to prepare all samples and controls (20% HCl, 15% HNO_3, 65% DI H_2O).

Aluminum chloride ($AlCl_3$) and aluminum potassium sulfate ($AlK(SO_4)_2$) are highly hygroscopic and as such are difficult to precisely weigh out. Application of a homogenous, thin layer of a modest amount (0.05g) of contaminant on the respective substrate facilitated an optimized interaction between contaminant and DeconGel, and provided an accurate measure of DeconGel's decontamination efficacy in a scaled-down yet real-world setting. When allowed to sit (30 min) on the concrete prior to gel application, these contaminants absorbed moisture from the air and soaked into the dry concrete. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel. Both aluminum chloride and aluminum potassium sulfate readily dissolve in aqueous acidic solutions used to prepare all samples and controls (20% HCl, 15% HNO_3, 65% DI H_2O).

Aluminum powder underwent a chemical reaction with stainless steel and carbon steel surfaces (Figure 1) which prevented some contamination from being sampled (swipe testing) and intercalated into dry DeconGel (direct gel testing). Nevertheless DeconGel showed acceptable decontamination efficacy against loose aluminum powder contamination.

Figure 1. Aluminum powder reacted

with the stainless steel surface creating a fixed Al surface deposit.

ASTM method E1728-03, a standardized swipe testing method used for the sampling of inorganic contaminants was the integral method used to accurately

evaluate DeconGel's decontamination efficacy. GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes (pre-wetted with DI H_2O) were utilized in this swipe testing method. Direct chemical analysis of the dried peeled DeconGel samples was also utilized to provide an improved understanding of DeconGel's decontamination efficacy. Standardized EPA SW-846 Sampling Method 3005A "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy" was followed as a guideline to prepare all samples and controls. When deemed necessary, digestion methods were customized by increasing hydrochloric and nitric acid concentrations from 25% to 35% wt, and/or by heating samples to higher temperatures using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC) to facilitate the complete digestion/dissolution of the inorganic contaminants. All samples, controls, and standards were prepared using the same dissolution solution and experimental conditions to ensure both correct instrument calibration and accurate analytical results.
ICP-OES instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of inorganic compounds. Standardized EPA SW-846 Analytical Method 6010C "Inductively Coupled Plasma-Atomic Emission Spectrometry" was followed as a guideline to prepare all samples and controls.

To ensure accurate determination of DeconGel decontamination efficacy, calibration standards of the analyte of interest were prepared using either a sufficiently pure analyte or an appropriate ICP-MS Standard (Ricca Chemical Company; Arlington, TX); the respective standards were diluted to a known concentration (ppm) using the same digestion method as the one used for samples and controls. Instrument blank controls were DI H_2O ($\geq$17 M-Ohm).

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

Decontamination Efficacy (DeconGel Testing) =

(Contaminant (ppm) of DeconGel Direct/Contaminant (ppm) of DeconGel Control) x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.05 to 0.10 g of contaminant was evenly applied on 1) stainless steel (commercial grade, surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24-48 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe and gel samples were suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% deionized (DI) H_2O) for 24 h. When deemed necessary, samples were heated to 94 ^oC for 4-14 h to effectively complete dissolution of inorganics using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC). Samples were allowed to cool to room temperature and were then analyzed via ICP-OES (see below).

Because aluminum oxide is a very fine powder, it easily clumped and formed hard deposits upon gel application. To circumvent this and evaluate a more "real world" scenario in which the surface would be swept prior to gel application, a smaller amount of aluminum oxide powder (ranging from 0.0088g to 0.0136g) was applied on the concrete panel. A paint brush was used to evenly spread the contamination to avoid any mounds of powder. Approximately 6.0 g of DeconGel 1101 was applied on top of the contamination without agitation to cover the entire contaminated area. The gel was allowed to dry for 24 hours prior to peeling. The process was repeated three times. The peeled gels were then placed in 125 ml metal-free digestion cups and filled to 100 mL using the acidic aqueous solution (20% HCl, 15% HNO_3). Digestion and analysis of the samples was performed following the same procedure described above. ICP-OES readings for both sample and control concentrations were normalized with respect to the amount of contaminant applied on the substrate.

Controls Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied on 1) stainless steel (commercial grade, surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O) for 24 h and analyzed via ICP-OES (see below).

For DeconGel Control samples, the respective amount of contaminant and approximately 6.0 g of dry DeconGel1101 (pre-poured gel on the respective un-contaminated substrate and let to dry for 24-48 h) was suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O) for 24 h and analyzed via ICP-OES (see below).

Reagents and Standards

Aluminium metal, Al, finest powder grade, (CAS# 7429-90-5, Fisher Scientific; Fair Lawn, NJ), was used as received.

Aluminium oxide, Al_2O_3, 60-325 Mesh, (CAS# 1344-28-1, Fisher Scientific; Fair Lawn, NJ), was used as received.

Aluminum Chloride, $AlCl_3$, fine crystalline solid, (CAS# 7446-70-7, Fisher Scientific; Fair Lawn, NJ), was used as received.

Aluminum Potassium Sulfate, $AlK(SO_4)_2$, fine crystalline solid, (CAS# 7784-24-9, Fisher Scientific; Fair Lawn, NJ), was used as received.

A 1000 ppm calibration standard of each contaminant was prepared using the aluminum and aluminum compounds in freshly prepared aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O). DI H_2O (> 17 M-Ohm) was used as the blank sample.

Analytical Instrumentation

A Thermo ICP-OES instrument model radial iCap 6300 was used to determine aluminum concentration (ppm) of all samples and controls using a freshly prepared 1000 ppm calibration standard. Analyte (aluminum) analyzed at 308.2 nm.

Pump Speed: 0.5 mL/min

6.4. Technical Report for Amine Compounds (Aniline)

TITLE: Surface Decontamination of Amine Compounds (Aniline) by DeconGel 1101

ANALYST: Geoff Nadolski PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel formulation 1101 on stainless steel, aluminum, and concrete surfaces contaminated with Plastics Feedstock (Aniline) using GC/MS (Gas Chromatography/Mass Spectrometry) following Environmental Protection Agency (EPA) SW-846 Methods 3500C (Sampling) and 8270C (Analysis).

HAZARDOUS MATERIALS RELEVANCE: Aniline is a volatile aromatic amine used mainly as a chemical feedstock to produce plastics including polyurethane, and used as a rubber additive. Aniline is acutely toxic, and prolonged exposure can result in hemolytic anemia and tumor formation in the spleen. Aniline was chosen as a representative industrial plastics feedstock; DeconGel is expected to have similar efficacy towards the wide range of plastics feedstock.

SUMMARY RESULTS:

- Excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with plastic feedstock (Aniline) resulting in encapsulation of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 100% (on concrete) to 100% (on stainless steel) to 100% (on aluminum) for Aniline determined by residual swipe analysis, and 78.2% (on concrete) to 93.8% (on aluminum) to 93.9% (on stainless steel) for Aniline as determined by direct DeconGel analysis.

- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of organic compounds dissolved in a suitable solvent able to completely solvate aniline as well as DeconGel components. When deemed necessary, experimental methods were customized to afford complete dissolution of organic contaminants. Additionally, analytical methods and associated equipment (GC column, GC temperature gradient program, MS sample ionization parameters) were appropriately developed to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 and 2 show the decontamination efficacies of DeconGel 1101 on stainless steel, aluminum, and concrete surfaces contaminated with Plastics Feedstock (aniline) as determined by residual swipe testing and direct DeconGel analysis.

Table 1. Decontamination efficacy of DeconGel 1101 on Aniline Plastics Feedstock contaminated stainless steel, aluminum, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	449.79 ± 2.14
	Residual	ND*
	Decon. Efficacy (%)	**100****
Aluminum	Control	449.88 ± 1.56
	Residual	ND
	Decon. Efficacy (%)	**100****
Concrete	Control	394.92 ± 0.43
	Residual	ND
	Decon. Efficacy (%)	**100****

1142x dilution factor for samples and controls

* ND: not detected, limit of detection (LOD) for aniline approximates 100 ppb

** An ND value for residual experiments results in a Decontamination Efficacy of 100%

Table 2. Decontamination efficacy of DeconGel 1101 on Aniline Plastics Feedstock contaminated stainless steel, aluminum, and concrete surfaces as determined by direct DeconGel analysis.

Direct DeconGel Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	476.97 ± 1.87
	Encapsulated in Gel	448.08 ± 0.85
	Decon. Efficacy (%)	**93.9 ± 0.48**
Aluminum	Control	476.85 ± 1.83
	Encapsulated in Gel	447.25 ± 0.56
	Decon. Efficacy (%)	**93.8 ± 0.43**
Concrete	Control	476.90 ± 1.34
	Encapsulated in Gel	373.17 ± 1.11
	Decon. Efficacy (%)	**78.2 ± 0.40**

1142x dilution factor for samples and controls

METHOD DEVELOPMENT OPTIMIZATIONS/OBSERVATIONS:

- We noted that amounts of contaminant liquid greater than 100 uL were difficult to spread evenly throughout coupon surfaces, forming zones of liquid saturation which tended to overload poured DeconGel, significantly limiting the ability of DeconGel to encapsulate/emulsify surface contamination. This issue was circumvented by applying a modest amount (35 uL) of contaminant liquid throughout the surface of interest using a small brush. Application of a homogenous, thin layer of contaminant on the respective substrate facilitated an optimized interaction between contaminant and DeconGel, and provided an accurate measure of DeconGel's decontamination efficacy in a scaled-down yet real-world setting. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel. Aniline readily dissolves in 60% methanol in water (50 mL) used to prepare all analytical samples and controls.

- Acceptable direct DeconGel decontamination efficacies ranging from 78.2 to 93.9% were achieved, however, aniline is prone to oxidize and evaporate at ambient temperature over the required 24 h drying time needed by DeconGel once applied to a contaminated surface, this could contribute to lower than expected DeconGel decontamination efficacies determined by the direct DeconGel testing method.

- ASTM method E1728-03, a standardized swipe testing method used for sampling of contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. Air-dried GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes were utilized in this swipe testing method.

- Direct chemical analysis of the dried peeled DeconGel samples was also utilized to provide an improved understanding of DeconGel's decontamination efficacy.

- Standardized EPA SW-846 Sampling Method 3500C "Organic Extraction and Sample Preparation" was followed as a guideline to prepare all samples and controls. All samples, controls, and standards were prepared using the same solvent and appropriate experimental conditions to ensure accurate and GC/MS instrumental analysis.

- GC/MS instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of organic compounds. Standardized EPA SW-846 Analytical Method 8270C "SEMIVOLATILE ORGANIC COMPOUNDS BY GAS

CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)" was followed as a guideline to prepare all samples and controls.

- To ensure accurate determination of DeconGel decontamination efficacy, a standard curve of the analyte of interest was prepared using sufficiently pure analyte; the respective standards were diluted to a known concentration (ppm) using the same solvent as used for samples and controls.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =
[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

Decontamination Efficacy (DeconGel Testing) =
(Contaminant (ppm) of DeconGel Direct/Contaminant (ppm) of DeconGel Control) x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 35 uL of contaminant was evenly applied via brushing on 1) aluminum (surface area: 56.3 cm^2), 2) stainless steel (surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe and gel samples were suspended in 50 mL 60% methanol in water for 24 h. Samples were then analyzed via GC/MS (see below).

Controls Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied via brushing on 1) aluminum (surface area: 56.3 cm^2), 2) stainless steel (surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 50 mL 60% methanol in water for 24 h and analyzed via GC/MS (see below).

For DeconGel Control samples, a respective amount of contaminant and approximately 6.0 g of dry DeconGel 1101 (pre-poured gel on the respective uncontaminated substrate and let to dry for 24 h) was suspended in 50 mL 60% methanol in water for 24 h and analyzed via GC/MS (see below).

Reagents and Standards

Aniline (phenylamine) (CAS# 62-53-3, Fisher Scientific; Fair Lawn, NJ) was used as received.

Analytical Instrumentation

A Thermo DSQII GC/MS with autosampler in positive mode was used to determine aniline concentration (ppm, wt/wt) of all samples and controls, using a Restek (Bellefonte, PA) Rtx-5 capillary column (30 m x 0.25 mm, 0.5 um).

A 7-point standard curve derived from three independently prepared stock solutions was prepared using DMSO as the working solvent.

GC method: start at 45 ºC, hold for 1 min, ramp at 30 ºC/min to 320 ºC, hold 10 min.

Aniline GC/MS data: 4.0 min; $M^+ = 93$.

6.5. Technical Report for Aromatic Liquids (Toluene)

TITLE: Surface Decontamination of Aromatic Liquids (Toluene) by DeconGel 1101 and 1102

ANALYST: Geoff Nadolski PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel formulation 1101 and 1102 on stainless steel, floor tile, and concrete surfaces contaminated with Aromatic Liquids (toluene) using LC/MS (Liquid Chromatography/Mass Spectrometry) following Environmental Protection Agency (EPA) SW-846 Methods 3500C (Sampling) and 8321B (Analysis).

HAZARDOUS MATERIALS RELEVANCE: Toluene is a volatile aromatic hydrocarbon widely used as a solvent, industrial feedstock, and as an octane booster in gasoline fuels. If inhaled, ingested, or internalized in large quantities, toluene can cause neurological, liver, and kidney damage, unconsciousness, and death. Toluene was chosen as a representative aromatic liquid; DeconGel is expected to have similar efficacy towards the wide range of aromatic liquids.

SUMMARY RESULTS:

- Excellent surface decontamination was achieved by applying both DeconGel 1101 and 1102, both via brushing or pouring (non-brushed) onto contaminated surfaces, resulting in encapsulation of toluene contaminant by DeconGel's active components. Decontamination efficacies of poured DeconGel 1101 ranged from 99.5% (on concrete) to 99.8% (on floor tile) to 99.8% (on stainless steel), brushed DeconGel 1101 ranged from 100% (on concrete) to 100% (on floor tile) to 100% (on stainless steel), as determined by residual swipe analysis. Decontamination efficacies of poured DeconGel 1102 ranged from 100% (on concrete) to 100% (on floor tile) to 100% (on stainless steel), brushed DeconGel 1102 ranged from 100% (on concrete) to 100% (on floor tile) to 100% (on stainless steel), as determined by residual swipe analysis. Decontamination efficacies of DeconGel 1101 ranged from 73.6% (on concrete) to 93.7% (on stainless steel) to 94.2% (on floor tile), DeconGel 1102 ranged from 78.1% (on concrete) to 95.0% (on stainless steel) to 95.3% (on floor tile), as determined by direct DeconGel analysis.

- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of organic compounds dissolved in a suitable solvent able to completely solvate toluene as well as DeconGel components. When deemed necessary, experimental methods were customized to afford complete dissolution of organic contaminants. Additionally, analytical methods and associated equipment (LC column, LC gradient program, MS sample ionization parameters) were appropriately developed to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 and 2 show the decontamination efficacies of DeconGel 1101 and 1102 on stainless steel, floor tile, and concrete surfaces contaminated with Aromatic Liquids (toluene) as determined by residual swipe testing and direct DeconGel analysis.

Table 1. Decontamination efficacies of DeconGel 1101 and 1102 on Toluene Aromatic Liquid contaminated stainless steel, floor tile, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		**Formulation**	
		DeconGel 1101	**DeconGel 1102**
Stainless Steel	Control	658.42 ± 2.86	658.42 ± 2.86
	Residual (non-brushed)	1.32 ± 0.10	ND*
	Residual (brushed)	ND	ND
	Decon. Efficacy (non-brushed) (%)	**99.8 ± 0.0**	**100****
	Decon. Efficacy (brushed) (%)	**100****	**100****
Floor Tile	Control	631.67 ± 1.07	631.67 ± 1.07
	Residual (non-brushed)	1.33 ± 0.06	ND
	Residual (brushed)	ND	ND
	Decon. Efficacy (non-brushed) (%)	**99.8 ± 0.0**	**100****
	Decon. Efficacy (brushed) (%)	**100****	**100****
Concrete	Control	482.95 ± 0.62	482.95 ± 0.62
	Residual (non-brushed)	2.48 ± 0.07	ND
	Residual (brushed)	ND	ND
	Decon. Efficacy (non-brushed) (%)	**99.5 ± 0.0**	**100****
	Decon. Efficacy (brushed) (%)	**100****	**100****

1571x dilution factor for samples and controls

* ND: not detected, limit of detection (LOD) for toluene approximates 250 ppb

** An ND value for residual experiments results in a Decontamination Efficacy of 100%

Table 2. Decontamination efficacies of DeconGel 1101 and 1102 on Toluene Aromatic Liquid contaminated stainless steel, floor tile, and concrete surfaces as determined by direct DeconGel analysis.

Direct DeconGel Testing (ppm)		**Formulation**	
		DeconGel 1101	**DeconGel 1102**
Stainless Steel	Control	703.45 ± 0.66	703.45 ± 0.66
	Encapsulated in Gel (non-brushed)	658.95 ± 2.31	668.48 ± 1.24
	Decon. Efficacy (non-brushed) (%)*	**93.7 ± 0.41**	**95.0 ± 0.18**
Floor Tile	Control	702.97 ± 1.42	702.97 ± 1.42
	Encapsulated in Gel (non-brushed)	662.21 ± 0.93	670.03 ± 0.75
	Decon. Efficacy (non-brushed) (%)	**94.2 ± 0.32**	**95.3 ± 0.22**
Concrete	Control	702.37 ± 1.03	702.37 ± 1.03
	Encapsulated in Gel (non-brushed)	516.97 ± 2.99	548.48 ± 2.08
	Decon. Efficacy (non-brushed) (%)	**73.6 ± 0.54**	**78.1 ± 0.19**

1571x dilution factor for samples and controls

* Decontamination Efficacy by direct DeconGel testing was determined only for non-brushed films, such that brushing DeconGel onto contaminated surfaces results in a loss of some portion of contaminant on brushing tool.

METHOD DEVELOPMENT OPTIMIZATIONS/OBSERVATIONS:

- We noted that amounts of contaminant liquid greater than 100 uL were difficult to spread evenly throughout coupon surfaces, forming zones of liquid saturation which tended to overload poured DeconGel, significantly limiting the ability of DeconGel to encapsulate/emulsify surface contamination. This issue was circumvented by applying a modest amount (40 uL) of contaminant liquid throughout the surface of interest using a small brush. Application of a homogenous, thin layer of contaminant on the respective substrate facilitated an optimized interaction between contaminant and DeconGel, and provided an accurate measure of DeconGel's decontamination efficacy in a scaled-down yet real-world setting. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel. Toluene readily dissolves in DMSO (50 mL) used to prepare all analytical samples and controls.

- Acceptable direct DeconGel decontamination efficacies ranging from 73.6 to 94.2% for DeconGel 1101, and decontamination efficacies ranging from 78.1 to 95.3% for DeconGel 1102 were achieved, however, because toluene is prone to evaporate over the required 24 h drying time needed by DeconGel once applied to a contaminated surface, this could contribute to lower than expected DeconGel decontamination efficacies determined by the direct DeconGel testing method.

- ASTM method E1728-03, a standardized swipe testing method used for sampling of contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. Air-dried GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes were utilized in this swipe testing method.

- Direct chemical analysis of the dried peeled DeconGel samples was also utilized to provide an improved understanding of DeconGel's decontamination efficacy. Decontamination efficacy was determined only for non-brushed (poured) DeconGel films, such that brushing DeconGel onto contaminated surfaces resulted in a loss of some portion of contaminant on the brushing tool used, and as such accurate decontamination efficacies for brushed DeconGel 1101 and 1102 could not be determined. For brushed films, accurate decontamination efficacies were best determined utilizing swipe testing.

- Standardized EPA SW-846 Sampling Method 3500C "Organic Extraction and Sample Preparation" was followed as a guideline to prepare all samples and controls. All samples, controls, and standards were prepared using the same solvent and appropriate experimental conditions to ensure accurate and LC/MS instrumental analysis.

- LC/MS instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of organic compounds. Standardized EPA SW-846 Analytical Method 8321B "Solvent-Extractable Nonvolatile Compounds by High-Performance Liquid Chromatography/Thermospray/Mass Spectrometry (HPLC/TS/MS) or Ultraviolet (UV) Detection" was followed as a guideline to prepare all samples and controls.

- To ensure accurate determination of DeconGel decontamination efficacy, a standard curve of the analyte of interest was prepared using sufficiently pure analyte; the respective standards were diluted to a known concentration (ppm) using the same solvent as used for samples and controls.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =
[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

Decontamination Efficacy (DeconGel Testing) =
(Contaminant (ppm) of DeconGel Direct/Contaminant (ppm) of DeconGel Control) x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 40 uL of contaminant was evenly applied via brushing on 1) stainless steel (surface area: 56.3 cm^2), 2) floor tile (surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe and gel samples were suspended in 50 mL DMSO for 24 h. Samples were then analyzed via LC/MS (see below).

Controls Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied via brushing on 1) stainless steel (surface area: 56.3 cm^2), 2) floor tile (surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 50 mL DMSO for 24 h and analyzed via LC/MS (see below).

For DeconGel Control samples, a respectable amount of contaminant and approximately 6.0 g of dry DeconGel 1101 (pre-poured gel on the respective uncontaminated substrate and let to dry for 24 h) was suspended in 50 mL DMSO for 24 h and analyzed via LC/MS (see below).

Reagents and Standards

Toluene (methylbenzene) (CAS# 108-88-3, Fisher Scientific; Fair Lawn, NJ) was used as received.

Analytical Instrumentation

A Thermo LCQ LC/MS with autosampler in positive mode was used to determine toluene concentration (ppm, wt/wt) of all samples and controls, using a Grace Davison (Deerfield, IL) C18-Select analytical column (150 x 4.6 mm, 5 um).

A 5-point standard curve derived from three independently prepared stock solutions was prepared using DMSO as the working solvent. LC method using A= water (0.1% formic acid), B=acetonitrile (0.1% formic acid); start at 95%A to 3 min at 70%A, to 6 min at 50%A, to 12 min at 30%A, to 15 min at 90%B, to 16 min at 100%B, hold until 22 min at 100% B, to 23 min at 95%A, hold until 24 min.

Toluene LC/MS data: 13.3 min; lambda max = 262, 268 nm; $M^+ = 92$.

6.6. Technical Report for Aromatic Solids (Naphthalene)

TITLE: Surface Decontamination of Aromatic Solids (Naphthalene) by DeconGel 1101

ANALYST: Geoff Nadolski PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel formulation 1101 on stainless steel, aluminum, and concrete surfaces contaminated with Aromatic Solids (naphthalene) using LC/MS (Liquid Chromatography/Mass Spectrometry) following Environmental Protection Agency (EPA) SW-846 Methods 3500C (Sampling) and 8321B (Analysis).

HAZARDOUS MATERIALS RELEVANCE: Naphthalene is a polycyclic aromatic hydrocarbon (PAH) that is volatile, readily subliming at room temperature. Naphthalene is the most abundant single component of coal tar, and is used as a chemical precursor to other chemicals, as a wetting agent/surfactant, and is used as a fumigant. Exposure to naphthalene may damage red blood cells, and the International Agency for Research on Cancer (IARC) classifies naphthalene as a potential carcinogen to animals and humans. Napthalene was chosen as a representative solid aromatic compound; DeconGel is expected to have similar efficacy towards the wide range of aromatic solids.

SUMMARY RESULTS:

- Excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with aromatic solids (naphthalene) resulting in encapsulation of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 99.2% (on concrete) to 99.6% (on stainless steel) to 99.2% (on aluminum) for Naphthalene as determined by residual swipe analysis, and 89.1% (on concrete) to 96.2% (on aluminum) to 96.3% (on stainless steel) for Naphthalene as determined by direct DeconGel analysis.

- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of organic compounds dissolved in a suitable solvent able to completely solvate naphthalene as well as DeconGel components. When necessary, experimental methods were customized to afford complete dissolution of organic contaminants. Additionally, analytical methods and associated equipment (LC column, LC gradient program, MS sample ionization parameters) were appropriately utilized to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 and 2 show the decontamination efficacies of DeconGel 1101 on stainless steel, aluminum, and concrete surfaces contaminated with Aromatic Solids (Naphthalene) as determined by residual swipe testing and direct DeconGel analysis respectively.

Table 1. Decontamination efficacy of DeconGel 1101 on Naphthalene Aromatic Solid contaminated aluminum, stainless steel, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	223.46 ± 0.38
	Residual	0.94 ± 0.01
	Decon. Efficacy (%)	**99.6 ± 0.0**
Aluminum	Control	223.47 ± 0.30
	Residual	0.95 ± 0.01
	Decon. Efficacy (%)	**99.6 ± 0.0**
Concrete	Control	169.0 ± 0.77
	Residual	1.27 ± 0.01
	Decon. Efficacy (%)	**99.2 ± 0.01**

2200x dilution factor for samples and controls

Table 2. Decontamination efficacy of DeconGel 1101 on Naphthalene Aromatic Solid on aluminum, stainless steel, and concrete surfaces as determined by direct DeconGel analysis.

Direct DeconGel Testing (ppm)		**Formulation**
		DeconGel 1101
Stainless Steel	Control	223.48 ± 0.89
	Encapsulated in Gel	215.26 ± 0.48
	Decon. Efficacy (%)	**96.3 ± 0.36**
Aluminum	Control	223.94 ± 0.78
	Encapsulated in Gel	215.55 ± 0.70
	Decon. Efficacy (%)	**96.2 ± 0.16**
Concrete	Control	223.71 ± 0.57
	Encapsulated in Gel	198.90 ± 1.62
	Decon. Efficacy (%)	**89.1 ± 0.75**

2200x dilution factor for samples and controls

METHOD DEVELOPMENT OPTIMIZATIONS/OBSERVATIONS:

- We noted that amounts of contaminant solid greater than 0.05 g were difficult to spread evenly throughout coupon surfaces, forming clumps and piles which tended to overload poured DeconGel, significantly limiting the ability of DeconGel to encapsulate/emulsify surface contamination. This issue was circumvented by applying a modest amount (0.025 g) of contaminant solid throughout the surface of interest. Application of a homogenous, thin layer of contaminant on the respective substrate facilitated an optimized interaction between contaminant and DeconGel, and provided an accurate measure of DeconGel's decontamination efficacy in a scaled-down yet real-world setting. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel. Naphthalene readily dissolves in DMSO (50 mL) used to prepare all analytical samples and controls.

- ASTM method E1728-03, a standardized swipe testing method used for sampling of contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. Air-dried GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes (pre-wetted with DMSO immediately prior to performing swipe analysis) were utilized in this swipe testing method.

- Direct chemical analysis of the dried peeled DeconGel samples was also utilized to provide an improved understanding of DeconGel's decontamination efficacy.

- Acceptable direct DeconGel decontamination efficacies ranging from 89.1 to 96.3% were achieved, however, naphthalene is prone to sublime at ambient temperature over the required 24 h drying time needed by DeconGel once applied to a contaminated surface.

- Standardized EPA SW-846 Sampling Method 3500C "Organic Extraction and Sample Preparation" was followed as a guideline to prepare all samples and controls. All samples, controls, and standards were prepared using the same solvent and appropriate experimental conditions to ensure accurate and LC/MS instrumental analysis.

- LC/MS instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of organic compounds. Standardized EPA SW-846 Analytical Method 8321B "Solvent-Extractable Nonvolatile Compounds by High-Performance Liquid Chromatography/Thermospray/Mass Spectrometry (HPLC/TS/MS) or Ultraviolet (UV) Detection" was followed as a guideline to prepare all samples and controls.

- To ensure accurate determination of DeconGel decontamination efficacy, a standard curve of the analyte of interest was prepared using sufficiently pure analyte; the respective standards were diluted to a known concentration (ppm) using the same solvent as used for samples and controls.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

Decontamination Efficacy (DeconGel Testing) =

(Contaminant (ppm) of DeconGel Direct/Contaminant (ppm) of DeconGel Control) x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.025 g of contaminant was evenly applied on 1) aluminum (surface area: 56.3 cm^2), 2) stainless steel (surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC) wetted with DMSO (2 mL) immediately prior to swipe analysis. Swipe and gel samples were suspended in 50 mL DMSO for 24 h. Samples were then analyzed via LC/MS (see below).

Controls Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied on 1) aluminum (surface area: 56.3 cm^2), 2) stainless steel (surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC) wetted with DMSO (2 mL) immediately prior to swipe analysis. Swipe samples were suspended in 50 mL DMSO for 24 h and analyzed via LC/MS (see below).

For DeconGel Control samples, a respectable amount of contaminant and approximately 6.0 g of dry DeconGel 1101 (pre-poured gel on the respective uncontaminated substrate and let to dry for 24 h) was suspended in 50 mL DMSO for 24 h and analyzed via LC/MS (see below).

Reagents and Standards

Naphthalene (bicyclo[4.4.0]deca-1,3,5,7,9-pentene) (CAS# 91-20-3, Fisher Scientific; Fair Lawn, NJ) was used as received.

Analytical Instrumentation

A Thermo LCQ LC/MS with autosampler in positive mode was used to determine naphthalene concentration (ppm, wt/wt) of all samples and controls, using a Grace Davison (Deerfield, IL) C18-Select analytical column (150 x 4.6 mm, 5 um).

A 5-point standard curve derived from three independently prepared stock solutions was prepared using DMSO as the working solvent.

LC method using A= water (0.1% formic acid), B=acetonitrile (0.1% formic acid); start at 90%A to 2 min at 70%A, to 3 min at 50%A, to 5 min at 30%A, to 6 min at 100%B, hold until 16 min at 90% A hold until 17 min.

Naphthalene LC/MS data: 8.8 min; lambda max = 250, 313 nm; M^+ = 128.

Table 1. Decontamination efficacy of DeconGel 1101 on KI contaminated stainless steel, carbon steel, floor tile, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	531.3 ± 2.5
	Residual	8.9 ± 4.1
	Decon. Efficacy (%)	**99.5 ± 2.9**
Carbon Steel	Control	521.8 ± 11.6
	Residual	6.2 ± 4.8
	Decon. Efficacy (%)	**98.8 ± 6.8**
Floor Tile	Control	293.3 ± 19.8
	Residual	4.14 ± 3.30
	Decon. Efficacy (%)	**98.6 ± 1.1**
Concrete	Control	514.4 ± 11.5
	Residual	6.7 ± 2.8
	Decon. Efficacy (%)	**98.7 ± 3.2**

21880x dilution factor for samples and controls

NOTES:

- Evaluation of the decontamination efficacy of DeconGel on KI on waxed floor tile was conducted to simulate an iodine/iodide spill as evidenced in a medical/clinical setting, such that the formed KI deposit is intended to simulate a radioactive and/or pharmaceutical form of iodine/iodide. DeconGel effectively decontaminated several forms of iodide contamination (from loose residual solids to dense deposits) on multiple types of surfaces (from non-porous, inert metals to waxy, chemically active plastics, to porous substrates).

- ASTM method E1728-03, a standardized swipe testing method used for sampling of inorganic contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes (pre-wetted with DI H_2O) were utilized in this swipe testing method.

- Standardized EPA SW-846 Sampling Method 3005A "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy" was followed as a guideline to prepare all samples and controls.

- To ensure accurate determination of DeconGel decontamination efficacy, calibration standards of the analyte of interest were prepared using either a sufficiently pure analyte or an appropriate ICP-MS Standard (Ricca

Chemical Company; Arlington, TX); the respective standards were diluted to a known concentration (ppm) using the same digestion method as the one used for samples and controls. Instrument blank controls were DI H_2O ($\geq$17 M-Ohm).

- ICP-OES instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of inorganic compounds. Standardized EPA SW-846 Analytical Method 6010C "Inductively Coupled Plasma-Atomic Emission Spectrometry" was followed as a guideline to prepare all samples and controls.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(KI (ppm) of Swipe Control) – (KI (ppm) of Residual Swipe)/KI (ppm) of Swipe Control] x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.05 g KI was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons; for waxed floor tile (surface area: 221.9 cm^2), 1.0 mL aqueous KI (50 mg/mL) was homogeneously pipetted onto the coupon surface and let to stand for one week until all water had evaporated and a KI deposit had formed. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24-48 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 100 mL aqueous acidic solution (15% HCl, 15% HNO_3, 70% deionized (DI) H_2O) for 24 h. When deemed necessary, samples were heated to 90 ^{o}C for 8-24 h to afford complete dissolution of inorganics using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC). Samples were allowed to cool to room temperature and were then analyzed via ICP-OES (see below).

Controls Methods

For Swipe Control samples, 0.05 g KI was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons: for waxed floor tile (surface area: 221.9 cm^2), 1.0 mL aqueous KI (50 mg/mL) was homogeneously pipetted onto the coupon surface and let to stand for one week until all water had evaporated and a KI deposit had formed. Coupon surfaces were swipe tested (ASTM method 1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 100 mL aqueous acidic solution (15% HCl, 15% HNO_3, 70% DI H_2O) for 24 h and analyzed via ICP-OES (see below).

Reagents and Standards

Reagent grade potassium iodide (KI) (CAS# 7681-11-0, Fisher Scientific; Fair Lawn, NJ) was used as received.

A 10.0 ppm calibration standard was prepared using reagent grade granular KI and freshly prepared aqueous acidic solution (15% HCl, 15% HNO_3, 70% DI H_2O). DI H_2O was used as the blank sample.

Analytical Instrumentation

A Thermo ICP-OES instrument model Radial iCap 6300 was used to determine KI concentration (ppm) of all samples and controls using a freshly prepared 10.0 ppm calibration standard.

Analyte (iodine) analyzed at 183.0 nm

Pump Speed: 0.5 mL/min

6.7. Technical Report for Arsenic (Arsenic Trioxide)

TITLE: Surface Decontamination of Arsenic Compounds (Arsenic Trioxide (As_2O_3)) by DeconGel 1101

AUTHOR: Andreas Mylonakis PhD, Senior Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with Arsenic (Arsenic Trioxide (As_2O_3)) using ICP-OES (Inductively Coupled Plasma-Optical Emission Spectroscopy) following Environmental Protection Agency (EPA) SW-846 Methods 3005A (sampling) and 6010C (analysis).

HAZARDOUS MATERIALS RELEVANCE: Arsenic compounds are used as insecticides and are used to make semiconductors. Arsenic compounds are inhalation hazards and are toxic if ingested. Arsenic Trioxide was chosen as a representative arsenic compound for evaluating DeconGel's efficacy; DeconGel is expected to have similar efficacy towards the wide range of arsenic compounds.

SUMMARY RESULTS:

- Excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with arsenic (arsenic trioxide) resulting in encapsulation of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 99.8% (on concrete) to 99.3% (on carbon steel) to 99.6% (on stainless steel) as determined by swipe analysis, and from 84.7% (on concrete) to 95.1% (on carbon steel) to 96.5% (on stainless steel) as determined by direct DeconGel analysis.

- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of inorganic compounds in aqueous samples. When deemed necessary, experimental methods were customized to afford complete dissolution of inorganic contaminants, and to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 and 2 show the decontamination efficacies of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with arsenic trioxide as determined by residual swipe testing and direct DeconGel analysis.

Table 1. Decontamination efficacy of DeconGel 1101 on As_2O_3 contaminated stainless steel, carbon steel, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	513.0 ± 37.4
	Residual	1.8 ± 1.3
	Decon. Efficacy (%)	**99.6 ± 7.8**
Carbon Steel	Control	539.4 ± 20.8
	Residual	3.8 ± 3.0
	Decon. Efficacy (%)	**99.3 ± 6.7**
Concrete	Control	487.7 ± 18.0
	Residual	1.1 ± 0.6
	Decon. Efficacy (%)	**99.8 ± 4.0**

2280x dilution factor for samples and controls

Table 2. Decontamination efficacy of DeconGel 1101 on As_2O_3 contaminated stainless steel, carbon steel and concrete surfaces as determined by direct DeconGel analysis

Direct DeconGel Analysis (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	330.1 ± 9.2
	Encapsulated in Gel	286.8 ± 5.3
	Decon. Efficacy (%)	**86.9 ± 2.9**
Carbon Steel	Control	317.0 ± 13.1
	Encapsulated in Gel	248.1 ± 14.8
	Decon. Efficacy (%)	**78.3 ± 5.7**
Concrete	Control	310.1 ± 16.1
	Encapsulated in Gel	271.9 ± 12.1
	Decon. Efficacy (%)	**87.7 ± 6.0**

21880x dilution factor for samples and controls

METHOD DEVELOPMENT OPTIMIZATIONS/OBSERVATIONS:

- We noted that amounts of contaminant solid greater than 0.05 g were difficult to spread evenly throughout coupon surfaces, forming clumps and piles which tended to overload poured DeconGel, significantly limiting the ability of DeconGel to encapsulate/emulsify surface contamination. This issue was circumvented by applying a modest amount (0.05 g) of contaminant solid throughout the surface of interest. Application of a homogenous, thin layer of contaminant on the respective substrate facilitated an optimized interaction between contaminant and DeconGel, and provided an accurate measure of DeconGel's decontamination efficacy in a scaled-down yet real-world setting. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel. As_2O_3 readily dissolves in aqueous acidic solutions used to prepare all samples and controls (20% HCl, 15% HNO_3, 65% DI H_2O).

- ASTM method E1728-03, a standardized swipe testing method used for the sampling of inorganic contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes (pre-wetted with DI H_2O) were utilized in this swipe testing method.

- Direct chemical analysis of the dried peeled DeconGel samples was also utilized to provide an improved understanding of DeconGel's decontamination efficacy.

- Standardized EPA SW-846 Sampling Method 3005A "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy" was followed as a guideline to prepare all samples and controls. When deemed necessary, digestion methods were customized by increasing hydrochloric and nitric acid concentrations from 25% to 35% wt, and/or by heating samples to higher temperatures using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC) to afford the complete digestion/dissolution of the inorganic contaminants. All samples, controls, and standards were prepared using the same dissolution solution and experimental conditions to ensure both correct instrument calibration and accurate analytical results.

- ICP-OES instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of inorganic compounds. Standardized EPA SW-846 Analytical Method 6010C "Inductively Coupled Plasma-Atomic Emission Spectrometry" was followed as a guideline to prepare all samples and controls.

- To ensure accurate determination of DeconGel decontamination efficacy, calibration standards of the analyte of interest were prepared using either a sufficiently pure analyte or an appropriate ICP-MS Standard (Ricca Chemical Company; Arlington, TX); the respective standards were diluted to a known concentration (ppm) using the same digestion method as the one used for samples and controls. Instrument blank controls were DI H_2O ($\geq$17 M-Ohm).

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

Decontamination Efficacy (DeconGel Testing) =

(Contaminant (ppm) of DeconGel Direct/Contaminant (ppm) of DeconGel Control) x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.05 g As_2O_3 was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24-48 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe and gel samples were suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% deionized (DI) H_2O) for 24 h. When deemed necessary, samples were heated to 94°C for 4-14 h to effectively complete dissolution of inorganics using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC). Samples were allowed to cool to room temperature and were then analyzed via ICP-OES (see below).

Controls Methods

For Swipe Controls, 0.05 g As_2O_3 was evenly applied on 1) stainless steel (commercial grade, surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 100 mL aqueous acidic solution (15% HCl, 15% HNO_3, 70% DI H_2O) for 24 h and analyzed via ICP-OES (see below).

For DeconGel Controls, 0.05 g As_2O_3 and approximately 6.0 g of dry DeconGel 1101 (pre-poured gel on the respective un-contaminated substrate and let to dry for 24-48 h) was suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O) for 24 h and analyzed via ICP-OES (see below).

Reagents and Standards

Reagent grade Arsenic Trioxide, As_2O_3, (CAS# 1327-53-3, Fisher Scientific; Fair Lawn, NJ), was used as received.

A 1000 ppm calibration standard was prepared using reagent grade powdered As_2O_3 and freshly prepared aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O). DI H_2O was used as the blank sample.

Analytical Instrumentation

A Thermo ICP-OES instrument model radial iCap 6300 was used to determine As_2O_3 concentration (ppm) of all samples and controls using a freshly prepared 1000 ppm calibration standard.

Analyte (arsenic) analyzed at 228.8 nm

Pump Speed: 0.5 mL/min

6.8. Technical Report for Barium Compounds (Barium Chloride, Barium Carbonate)

TITLE: Surface Decontamination of Barium Compounds (Barium Chloride ($BaCl_2$) and Barium Carbonate ($BaCO_3$)) by DeconGel 1101

AUTHOR: Andreas Mylonakis PhD, Senior Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel 1101 on stainless steel, aluminum, carbon steel, and concrete surfaces contaminated with Barium Compounds (barium chloride ($BaCl_2$) and barium carbonate ($BaCO_3$)) using ICP-OES (Inductively Coupled Plasma-Optical Emission Spectroscopy) following Environmental Protection Agency (EPA) SW-846 Methods 3005A (sampling) and 6010C (analysis).

HAZARDOUS MATERIALS RELEVANCE: Barium and its compounds are utilized in the electronics industry as well as in the production of steel, fireworks, and pigments. Barium chloride is a water soluble salt of barium and is considered a severe health hazard and a poison. Barium carbonate is classified as an A4 carcinogen by ACGIH[2]. Barium and its compounds are considered hazardous and can produce adverse health effects in the case of skin contact, inhalation, or ingestion. Barium chloride and barium carbonate were chosen as representative barium compounds for evaluating DeconGel's efficacy; DeconGel is expected to have similar efficacy towards the wide range of barium compounds.

SUMMARY RESULTS:

- Excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with barium compounds (barium chloride, barium carbonate) resulting in encapsulation of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 98.6% and 90.1% (on concrete) to 99.8% and 96.5% (on carbon steel) to 99.3% and 95.0% (on stainless steel) as determined by swipe analysis and 85.0% and 83.1% (on concrete) to 97.7% and 94.2% (on carbon steel) to 101.5% and 99.2% (on stainless steel) as determined by direct gel analysis for barium chloride and barium carbonate, respectively.

- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of inorganic compounds in aqueous samples. When deemed

---

[2] American Conference of Industrial Hygienists (ACGIH); http://www.acgih.org/home.htm (2010)

necessary, experimental methods were customized to afford complete dissolution of inorganic contaminants, and to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 through 4 show the decontamination efficacies of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with barium chloride and barium carbonate as determined by residual swipe testing and direct DeconGel analysis.

Table 1. Decontamination efficacies of DeconGel 1101 against Barium Chloride ($BaCl_2$) contaminated stainless steel, carbon steel, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	5.5 ± 0.2
	Residual	0.035 ± 0.02
	Decon. Efficacy (%)	**99.3 ± 0.3**
Carbon Steel	Control	5.63 ± 0.27
	Residual	0.014 ± 0.01
	Decon. Efficacy (%)	**99.8 ± 5.5**
Concrete	Control	8.69 ± 0.04
	Residual	0.12 ± 0.02
	Decon. Efficacy (%)	**98.6 ± 1.9**

228000x dilution factor for samples and controls

Table 2. Decontamination efficacies of DeconGel 1101 against Barium Chloride ($BaCl_2$) contaminated stainless steel, carbon steel and concrete surfaces as determined by direct DeconGel analysis.

Direct DeconGel Analysis (ppm)		Formulation		
		DeconGel 1101[a]	**DeconGel 1101[b]**	**DeconGel 1101[c]**
Stainless Steel	Control	11.01 ± 0.09	5.65 ± 0.58	5.15 ± 0.13
	Encapsulated in Gel	10.2 ± 1.08	5.35 ± 0.49	5.23 ± 0.17
	Decon. Efficacy (%)	**92.6 ± 9.8**	**105.7 ± 14.6**	**101.5 ± 4.1**
Carbon Steel	Control	10.61 ± 0.02	5.07 ± 1.69	5.11 ± 0.17
	Encapsulated in Gel	9.96 ± 0.38	5.09 ± 0.56	4.99 ± 0.22
	Decon. Efficacy (%)	**93.8 ± 3.4**	**100.3 ± 35.2**	**97.7 ± 5.4**
Concrete	Control	9.71 ± 0.09	NA	5.06 ± 0.13
	Encapsulated in Gel	8.48 ± 7.7	NA	4.28 ± 0.26
	Decon. Efficacy (%)	**87.4 ± 8.3**	NA	**85.0 ± 5.7**

228000x dilution factor for samples and controls

a. 100mg of contaminant was applied on the surface representing a heavy deposit of contamination.

b. 50mg of contaminant was applied. The gel on concrete did not fully dry after 72 hrs and was not analyzed.

c. 50mg of contaminant was applied. BaCl2 remained on the panel for 24 hours prior to gel application. Gel was allowed to dry for 72 hours.

Table 3. Decontamination efficacies of DeconGel 1101 against barium carbonate ($BaCO_3$) contaminated stainless steel, carbon steel, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	5.31 ± 0.13
	Residual	0.26 ± 0.06
	Decon. Efficacy (%)	**95.0 ± 2.8**
Carbon Steel	Control	5.15 ± 0.42
	Residual	0.18 ± 0.06
	Decon. Efficacy (%)	**96.5 ± 8.1**
Concrete	Control	4.95 ± 0.21
	Residual	0.49 ± 0.07
	Decon. Efficacy (%)	**90.1 ± 4.1**

228000x dilution factor for samples and controls

Table 4. Decontamination efficacies of DeconGel 1101 against barium carbonate ($BaCO_3$) contaminated stainless steel, carbon steel, and concrete surfaces as determined by Direct DeconGel analysis.

Direct DeconGel Analysis (ppm)		**Formulation**
		DeconGel 1101
Stainless Steel	Control	5.31 ± 0.13
	Encapsulated in Gel	5.26 ± 0.2
	Decon. Efficacy (%)	**99.2 ± 4.9**
Carbon Steel	Control	5.47 ± 0.05
	Encapsulated in Gel	5.16 ± 0.15
	Decon. Efficacy (%)	**94.2 ± 2.9**
Concrete	Control	5.41 ± 0.12
	Encapsulated in Gel	4.50 ± 0.16
	Decon. Efficacy (%)	**83.1 ± 3.4**

228000x dilution factor for samples and controls

METHOD DEVELOPMENT OPTIMIZATIONS/OBSERVATIONS:

- Barium chloride ($BaCl_2$) is highly hygroscopic and as such is difficult to precisely weigh out. Application of a homogenous, thin layer of a modest amount (0.05g) of contaminant on the respective substrate facilitated an optimized interaction between contaminant and DeconGel, and provided an accurate measure of DeconGel's decontamination efficacy in a scaled-down yet real-world setting. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel. $BaCl_2$ and $BaCO_3$ readily dissolve in aqueous acidic solutions used to prepare all samples and controls (20% HCl, 15% HNO_3, 65% DI H_2O).

- ASTM method E1728-03, a standardized swipe testing method used for sampling of inorganic contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes (pre-wetted with DI H_2O) were utilized in this swipe testing method.

- Direct chemical analysis of the dried peeled DeconGel samples was also utilized to provide an improved understanding of DeconGel's decontamination efficacy.

- Standardized EPA SW-846 Sampling Method 3005A "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy" was followed as a guideline to prepare all samples and controls. When deemed necessary, the digestion methods were customized by increasing hydrochloric and nitric acid concentrations from 25% to 35% wt, and/or by heating samples to higher temperatures using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC) for the complete digestion/dissolution of the inorganic contaminants. All samples, controls, and standards were prepared using the same dissolution solution and experimental conditions to ensure both correct instrument calibration and accurate analytical results.

- ICP-OES instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of inorganic compounds. Standardized EPA SW-846 Analytical Method 6010C "Inductively Coupled Plasma-Atomic Emission Spectrometry" was followed as a guideline to prepare all samples and controls.
- To ensure accurate determination of DeconGel decontamination efficacy, calibration standards of the analyte of interest were prepared using either a sufficiently pure analyte or an appropriate ICP-MS Standard (Ricca Chemical Company; Arlington, TX); the respective standards were diluted to a known concentration (ppm) using the same digestion method as the one used for samples and controls. Instrument blank controls were DI H_2O ($\geq$17 M-Ohm).
- Barium sulfate ($BaSO_4$) is a white powder which after repeated attempts could not be fully digested/dissolved in various acidic aqueous solutions containing hydrochloric acid, nitric acid, sulfuric acid and combinations thereof, in varying concentrations and temperatures; barium sulfate was thus disregarded from further evaluation tests.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

Decontamination Efficacy (DeconGel Testing) =

(Contaminant (ppm) of DeconGel Direct/Contaminant (ppm) of DeconGel Control) x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.05 to 0.10 g of contaminant was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24-48 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe and gel samples were suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% deionized (DI) H_2O) for 24 h. When deemed necessary, samples were heated to 94 °C for 4-14 h to effectively complete dissolution of inorganics using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC). Samples were allowed to cool to room temperature, diluted 1:100 with DI H_2O and were then analyzed via ICP-OES (see below).

Controls Methods

For Swipe Controls samples, 0.05 g of contaminant was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O) for 24 h, diluted 1:100 with DI H_2O and analyzed via ICP-OES (see below).

For DeconGel Controls samples, a respectable amount of contaminant (0.10 or 0.05g) and approximately 6.0 g of dry DeconGel (pre-poured gel on the respective un-contaminated substrate and let to dry for 24-48 h) was suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O) for 24 h, diluted 1:100 with DI H_2O, and analyzed via ICP-OES.

Reagents and Standards

Barium Chloride Dihydrate, $BaCl_2{\cdot}2H_2O$, (CAS# 10326-27-9, Fisher Scientific; Fair Lawn, NJ), was used as received.

Barium Carbonate, $BaCO_3$, (CAS# 513-77-9 Fisher Scientific; Fair Lawn, NJ), was used as received.

A 10.0 ppm calibration standard of each contaminant was prepared using the reagent grade barium compounds and freshly prepared aqueous acidic solution

(20% HCl, 15% HNO_3, 65% DI H_2O). DI H_2O ($\geq$17 M-Ohm) was used as the blank sample.

Analytical Instrumentation

A Thermo ICP-OES instrument model radial iCap 6300 was used to determine barium concentration (ppm) of all samples and controls using a freshly prepared 10.0 ppm calibration standard.

Analyte (barium) analyzed at 455.4 nm

Pump Speed: 0.5 mL/min.

6.9. Technical Report for Beryllium Compounds (Aluminum Powder and Aluminum Oxide; surrogates of Beryllium Dust and Beryllium Oxide Respectively)

TITLE: Surface Decontamination of Beryllium Compounds Utilizing Beryllium Surrogates (Elemental Aluminum Powder (Al) and Aluminum Oxide (Al_2O_3)) by DeconGel 1101

AUTHOR: Andreas Mylonakis PhD, Senior Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with Beryllium Surrogate Compounds (aluminum powder (Al) and aluminum oxide (Al_2O_3)) using ICP-OES (Inductively Coupled Plasma-Optical Emission Spectroscopy) following Environmental Protection Agency (EPA) SW-846 Methods 3005A (sampling) and 6010C (analysis).

HAZARDOUS MATERIALS RELEVANCE: Beryllium compounds are used in the aerospace industry to construct lightweight and resilient alloys. Beryllium is directly corrosive to living tissue; beryllium dust is toxic by inhalation and can cause berylliosis, an incurable chronic lung disease. Due to beryllium toxicity, the commonly used beryllium surrogate aluminum was used in this study. Aluminum powder and aluminum oxide were chosen as representative beryllium surrogate compounds for evaluating DeconGel's efficacy; DeconGel is expected to have similar efficacy towards the wide range of beryllium surrogate compounds.

SUMMARY RESULTS:

- Excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with beryllium surrogates (aluminum powder and aluminum oxide) resulting in encapsulation of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 99.7% (on concrete) to 99.9% (on carbon steel) to 99.7% (on stainless steel) as determined by residual swipe analysis and from 101.1% (on stainless steel) to 93.0% (on carbon steel) to 90.1% (on concrete) as determined by direct DeconGel analysis.

- DeconGel showed excellent decontamination efficacy against aluminum metal powder and aluminum oxide (surrogates for beryllium metal dust and beryllium oxide respectively) from concrete surfaces. Aluminum powder is reactive to some metal surfaces including carbon steel and stainless steel.

DeconGel could not completely remove aluminum elemental powder that reacted with and created a fixed deposit on steel surfaces. Nevertheless, DeconGel showed excellent decontamination efficacy on loose aluminum powder contaminant from such surfaces.

- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guid^{e}lines for determination of inorganic compounds in aqueous samples. When deemed necessary, experimental methods were customized to afford complete dissolution of inorganic contaminants, and to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 through 4 show the decontamination efficacies of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with aluminum powder (Al) and aluminum oxide (Al_2O_3) as determined by residual swipe testing and direct DeconGel analysis.

Table 1. Decontamination efficacies of DeconGel 1101 on aluminum powder (Al) contaminated stainless steel, carbon steel, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation		
		DeconGel 1101[a]	**DeconGel 1101[b]**	**DeconGel 1101[c]**
Stainless Steel*	Control	1113.0 ± 17.0	NA	NA
	Residual	23.3 ± 5.3	NA	NA
	Decon. Efficacy (%)	**97.9 ± 2.0**	**NA**	**NA**
Carbon Steel*	Control	944.7 ± 0.2	NA	NA
	Residual	4.7 ± 4.3	NA	NA
	Decon. Efficacy (%)	**99.5 ± 4.6**	**NA**	**NA**
Concrete	Control	1014.0 ± 4.0	423.3 ± 34.8	511.7 ± 12.7
	Residual	60.4 ± 17.0	4.2 ± 0.8	1.8 ± 0.2
	Decon. Efficacy (%)	**94.0 ± 2.8**	**99.0 ± 8.5**	**99.6 ± 3.8**

2280x dilution factor for samples and controls

*Aluminum powder reacted with these surfaces creating a fixed residue on the contaminated surface that could not be completely removed by DeconGel

NA: not applicable

d. 100 mg of contaminant was applied on the substrate.
e. 50 mg of contaminant was applied on the substrate.
f. 50 mg of contaminant was applied on the substrate; a fresh standard was made.

Table 2. Decontamination efficacies of DeconGel 1101 on aluminum powder (Al) contaminated stainless steel, carbon steel and concrete surfaces as determined by direct DeconGel analysis.

Direct DeconGel Analysis (ppm)		Formulation
		DeconGel 1101
Stainless Steel*	Control	514.1 ± 24.4
	Encapsulated in Gel	291.3 ± 16.2
	Decon. Efficacy (%)	**43.3 ± 3.8**
Carbon Steel*	Control	528.0 ± 16.1
	Encapsulated in Gel	400.2 ± 49.0
	Decon. Efficacy (%)	**75.8 ± 9.6**
Concrete	Control	400.5 ± 37.5
	Encapsulated in Gel	381.5 ± 27.8
	Decon. Efficacy (%)	**95.2 ± 11.2**

2280x dilution factor for samples and controls
*Aluminum powder reacted with these surfaces creating a fixed residue on the contaminated surface that could not be completely removed by DeconGel.

Table 3. Decontamination efficacies of DeconGel against aluminum oxide (Al_2O_3) contaminated stainless steel, carbon steel, and concrete surfaces as determined by residual swipe testing method.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	420.8 ± 7.0
	Residual	1.4 ± 0.6
	Decon. Efficacy (%)	**99.7 ± 7.7**
Carbon Steel	Control	505.5 ± 22.4
	Residual	0.3 ± 0.3
	Decon. Efficacy (%)	**99.9 ± 4.4**
Concrete	Control	420.2 ± 6.1
	Residual	1.3 ± 0.7
	Decon. Efficacy (%)	**99.7 ± 5.9**

2280x dilution factor for samples and controls

Table 4. Decontamination efficacies of DeconGel against aluminum oxide (Al_2O_3) on stainless steel, carbon steel and concrete surfaces as determined by direct DeconGel analysis.

Direct Gel Testing (ppm)		Formulation	
		DeconGel 1101[a]	DeconGel 1101[b]
Stainless Steel	Control	501.3 ± 11.6	NA
	Encapsulated in Gel	506.7 ± 20.0	NA
	Decon. Efficacy (%)	**101.1 ± 4.6**	**NA**
Carbon Steel	Control	477.2 ± 56.5	NA
	Encapsulated in Gel	443.8 ± 50.0	NA
	Decon. Efficacy (%)	**93.0 ± 15.2**	**NA**
Concrete	Control	494.5 ± 26.7	9685.9 ± 125.7*
	Encapsulated in Gel	254.8 ± 23.3	8700.9 ± 49.8*
	Decon. Efficacy (%)	**51.6 ± 5.5**	**90.1 ± 1.9**

2280x dilution factor for samples and controls

* ICP-OES readings have been normalized with regards to the amount (g) of contaminant applied on the surface and are reported in "ppm/g of contaminant applied". Refer to "Materials and Method" section for more details.

a. 50mg of contaminant was applied onto the surface before DeconGel 1101 application.

b. Contaminant amounts ranging from 0.0088g to 0.0136g were evenly brushed onto concrete before DeconGel 1101 application.

METHOD DEVELOPMENT OPTIMIZATIONS/OBSERVATIONS:

- We noted that amounts of contaminant solid greater than 0.05 g were difficult to spread evenly throughout coupon surfaces, forming clumps and piles which tended to overload poured DeconGel, significantly limiting the ability of DeconGel to encapsulate/emulsify surface contamination. This issue was circumvented by applying a modest amount (0.05 g) of contaminant solid throughout the surface of interest. Application of a homogenous, thin layer of the aluminum contaminants on the respective substrate facilitated an optimized interaction between contaminant and DeconGel, and provided an accurate measure of DeconGel's decontamination efficacy in a scaled-down yet real-world setting. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel. Both Al_2O_3 and Al powder readily dissolve in aqueous acidic solutions used to prepare all samples and controls (20% HCl, 15% HNO_3, 65% DI H_2O).

- Aluminum powder underwent a chemical reaction with stainless steel and carbon steel surfaces (Figure 1) which prevented some contamination from being sampled during the swipe sampling step. Nevertheless DeconGel showed acceptable decontamination efficacy of loose aluminum powder contamination.

Figure 1. Aluminum powder reacted with the stainless steel surface creating a fixed Al surface deposit.

- ASTM method E1728-03, a standardized swipe testing method used for the sampling of inorganic contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. GhostWipe™

(Environmental Express; Mt. Pleasant, SC) swipes (pre-wetted with DI H_2O) were utilized in this swipe testing method.

- Direct chemical analysis of the dried peeled DeconGel samples was also utilized to provide an improved understanding of DeconGel's decontamination efficacy.

- Standardized EPA SW-846 Sampling Method 3005A "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy" was followed as a guideline to prepare all samples and controls. When deemed necessary, digestion methods were customized by increasing hydrochloric and nitric acid concentrations from 25% to 35% wt, and/or by heating samples to higher temperatures using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC) to facilitate the complete digestion/dissolution of the inorganic contaminants. All samples, controls, and standards were prepared using the same dissolution solution and experimental conditions to ensure both correct instrument calibration and accurate analytical results.

- ICP-OES instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of inorganic compounds. Standardized EPA SW-846 Analytical Method 6010C "Inductively Coupled Plasma-Atomic Emission Spectrometry" was followed as a guideline to prepare all samples and controls.

- To ensure accurate determination of DeconGel decontamination efficacy, calibration standards of the analyte of interest were prepared using either a sufficiently pure analyte or an appropriate ICP-MS Standard (Ricca Chemical Company; Arlington, TX); the respective standards were diluted to a known concentration (ppm) using the same digestion method as the one used for samples and controls. Instrument blank controls were DI H_2O ($\geq$17 M-Ohm).

- Beryllium surrogates (aluminium metal powder (Al) and aluminium oxide (Al_2O_3) for beryllium metal powder (Be) and beryllium oxide (BeO) respectively) have been utilized in this evaluation study due to the high toxicity and carcinogenicity of beryllium metal and beryllium compounds.

- Aluminum carbide was initially considered to be used as a beryllium surrogate (beryllium carbide surrogate) for DeconGel decontamination efficacy evaluations. Due to safety concerns it was disregarded from further evaluations.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

Decontamination Efficacy (DeconGel Testing) =

(Contaminant (ppm) of DeconGel Direct/Contaminant (ppm) of DeconGel Control) x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.05 to 0.10 g of contaminant was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24-48 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe and gel samples were suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% deionized (DI) H_2O) for 24 h. When deemed necessary, samples were heated to 94°C for 4-14 h to effectively complete dissolution of inorganics using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC). Samples were allowed to cool to room temperature and were then analyzed via ICP-OES (see below).

Because aluminum oxide is a very fine powder, it clumped and formed hard deposits upon gel application. To circumvent this and represent a “real world” scenario in which the surface would be swept prior to gel application, a smaller amount of aluminum oxide powder (ranging from 0.0088g to 0.0136g) was applied on the concrete panel. A paint brush was then used to evenly spread the contamination to avoid any mounds of powder. Approximately 6.0 g of DeconGel 1101 was applied on top of the contamination without agitation to cover the entire contaminated area. The gel was allowed to dry for 24 hours prior to peeling. The process was repeated three times. The peeled gels were placed in 125 ml metal-free digestion cups and filled to 100 mL using the acidic aqueous solution (20% HCl, 15% HNO_3).

Digestion and analysis of the samples was performed following the same procedure described above. ICP-OES readings for both samples and controls were normalized with respect to the amount of contaminant applied on the substrate.

Controls Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O) for 24 h and analyzed via ICP-OES (see below).

For DeconGel Control samples, the respective amount of contaminant and approximately 6.0 g dry DeconGel (pre-poured gel on the respective un-contaminated substrate and let to dry for 24-48 h) was suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O) for 24 h and analyzed via ICP-OES (see below).

Reagents and Standards

Aluminium metal, Al, finest powder grade, (CAS# 7429-90-5, Fisher Scientific; Fair Lawn, NJ), was used as received.

Aluminium oxide, Al_2O_3, 60-325 Mesh, (CAS# 1344-28-1, Fisher Scientific; Fair Lawn, NJ), was used as received.

A 1000 ppm calibration standard of each contaminant was prepared using the beryllium surrogates and freshly prepared aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O). DI H_2O was used as the blank sample.

Analytical Instrumentation

A Thermo ICP-OES instrument model radial iCap 6300 was used to determine aluminum concentration (ppm) of all samples and controls using a freshly prepared 1000 ppm calibration standard.

Analyte (aluminum) analyzed at 308.2 nm; Pump Speed: 0.5 mL/min

6.10. Technical Report for Cadmium Compounds (Cadmium Chloride, Cadmium Oxide)

TITLE: Surface Decontamination of Cadmium Compounds (Cadmium Chloride ($CdCl_2$) and Cadmium Oxide (CdO)) by DeconGel 1101

AUTHOR: Andreas Mylonakis PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel 1101 on stainless steel, aluminum, carbon steel, and concrete surfaces contaminated with Cadmium Compounds (cadmium chloride ($CdCl_2$) and cadmium oxide (CdO)) using ICP-OES (Inductively Coupled Plasma-Optical Emission Spectroscopy) following Environmental Protection Agency (EPA) SW-846 Methods 3005A (sampling) and 6010C (analysis).

HAZARDOUS MATERIALS RELEVANCE: Cadmium metal and cadmium compounds are highly toxic. Cadmium chloride is often used in the process of making cadmium sulfide, a common yellow pigment while cadmium oxide is a conductive material used in the making of photovoltaic cells and photodiodes. Cadmium compounds are known carcinogens (classified A2 by ACGIH[3]) and fatal inhalation hazards. It is important to contain and remove cadmium particles which can become airborne. Cadmium chloride and cadmium oxide were chosen as representative cadmium compounds for evaluating DeconGel's efficacy; DeconGel is expected to have similar efficacy towards the wide range of cadmium compounds.

SUMMARY RESULTS:

- Excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with cadmium (cadmium chloride and cadmium oxide) resulting in encapsulation of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 99.5% and 100% (on stainless steel), to 98.5% and 100% (carbon steel) to 97.6% and 98.4% (on concrete) as determined by residue swipe analysis and from 88.6% and 100.0% (stainless steel), to 94.4% and 99.4% (carbon steel) to 64.3% and 93.8 (concrete) as determined by direct DeconGel analysis for cadmium chloride and cadmium oxide respectively.

- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of inorganic compounds in aqueous samples. When deemed

---

[3] A2: suspected human carcinogen; American Conference of Industrial Hygienists (ACGIH); http://www.acgih.org/home.htm (2010)

necessary, experimental methods were customized to afford complete dissolution of inorganic contaminants, and to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 through 4 show the decontamination efficacies of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with cadmium chloride and cadmium oxide as determined by residual swipe testing and direct DeconGel analysis.

Table 1. Decontamination efficacies of DeconGel 1101 against Cadmium Chloride ($CdCl_2$) contaminated stainless steel, carbon steel, and concrete surfaces as determined by residual swipe testing method.

Swipe Testing (ppm)		**Formulation**
		DeconGel 1101
Stainless Steel	Control	464.7 ± 7.0
	Residual	2.25 ± 1.81
	Decon. Efficacy (%)	**99.5 ± 1.7**
Carbon Steel	Control	521.1 ± 21.8
	Residual	7.81 ± 1.17
	Decon. Efficacy (%)	**98.5 ± 4.2**
Concrete	Control	483.0 ± 2.5
	Residual	11.63 ± 7.80
	Decon. Efficacy (%)	**97.6 ± 2.2**

2280x dilution factor for samples and controls

Table 2. Decontamination efficacies of DeconGel against Cadmium Chloride ($CdCl_2$) contaminated stainless steel, carbon steel and concrete surfaces as determined by direct DeconGel analysis.

Direct DeconGel Testing (ppm)		Formulation	
		DeconGel 1101[a]	**DeconGel 1101[b]**
Stainless Steel	Control	863.5 ± 2.2	501.2 ± 24.1
	Encapsulated in Gel	764.9 ± 6.4	472.2 ± 35.8
	Decon Efficacy (%)	**88.6 ± 7.5**	**94.2 ± 8.5**
Aluminum*	Control	863.5 ± 2.2	NA
	Encapsulated in Gel	619.1 ± 7.5	NA
	Decon. Efficacy (%)	**71.7 ± 0.9**	NA
Carbon Steel	Control	NA	548.4 ± 11.7
	Encapsulated in Gel	NA	517.8 ± 26.3
	Decon. Efficacy (%)	NA	**94.4 ± 5.2**
Concrete	Control	863.2 ± 2.2	501.7 ± 5.4
	Encapsulated in Gel	554.8 ± 57.5	460.0 ± 21.6
	Decon. Efficacy (%)	**64.3 ± 6.7**	**91.7 ± 4.7**

2280x dilution factor for samples and controls

* $CdCl_2$ reacted with aluminum surface forming a precipitate that caused clogging of the ICP-OES. Only one sample was analyzed; further testing was disregarded. .

a. 100mg of $CdCl_2$ was applied to the surface prior to application of DeconGel

b. 50mg of $CdCl_2$ was applied to the surface prior to application of DeconGel

Table 3. Decontamination efficacies of DeconGel against Cadmium Oxide (CdO) contaminated stainless steel, carbon steel, and concrete surfaces as determined by residual swipe testing method.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	553.4 ± 33.2
	Residual	0.086 ± 0.075
	Decon. Efficacy (%)	**100.0 ± 0.2**
Carbon Steel	Control	578.8 ± 14.3
	Residual	0.204 ± 0.104
	Decon. Efficacy (%)	**100.0 ± 0.4**
Concrete	Control	543.2 ± 22.4
	Residual	8.52 ± 7.23
	Decon. Efficacy (%)	**98.4 ± 0.2**

2280x dilution factor for samples and controls

Table 4. Decontamination efficacies of DeconGel against Cadmium Oxide (CdO) on stainless steel, carbon steel and concrete surfaces as determined by direct DeconGel testing method.

Direct DeconGel Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	305.2 ± 2.3
	Encapsulated in Gel	305.4 ± 10.6
	Decon. Efficacy (%)	**100.1 ± 3.5**
Carbon Steel	Control	315.5 ± 10.9
	Encapsulated in Gel	313.6 ± 13.8
	Decon. Efficacy (%)	**99.4 ± 5.6**
Concrete	Control	307.0 ± 3.8
	Encapsulated in Gel	288.1 ± 12.6
	Decon. Efficacy (%)	**93.8 ± 4.3**

2280x dilution factor for samples and controls

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

Decontamination Efficacy (DeconGel Testing) =

(Contaminant (ppm) of DeconGel Direct/Contaminant (ppm) of DeconGel Control) x 100%

METHOD DEVELOPMENT OPTIMIZATIONS/OBSERVATIONS:

- We noted that amounts of contaminant solid greater than 0.05 g were difficult to spread evenly throughout coupon surfaces, forming clumps and piles which tended to overload poured DeconGel, significantly limiting the ability of DeconGel to encapsulate/emulsify surface contamination. This issue was circumvented by applying a modest amount (0.05 g) of contaminant solid throughout the surface of interest. Cadmium chloride ($CdCl_2$) is highly hygroscopic and as such is difficult to precisely weigh out. Application of a homogenous, thin layer of contaminant on the respective substrate facilitated an optimized interaction between contaminant and DeconGel, and provided an accurate measure of DeconGel's decontamination efficacy in a scaled-down yet real-world setting. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel. $CdCl_2$ and CdO readily dissolve in aqueous acidic solutions used to prepare all samples and controls (20% HCl, 15% HNO_3, 65% DI H_2O).

- ASTM method E1728-03, a standardized swipe testing method used for sampling inorganic contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes (pre-wetted with DI H_2O) were utilized in this swipe testing method.
- Direct chemical analysis of the dried peeled DeconGel samples was also utilized to provide an improved understanding of DeconGel's decontamination efficacy.
- Standardized EPA SW-846 Sampling Method 3005A "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy" was followed as a guideline to prepare all samples and controls. When deemed necessary, digestion methods were customized by increasing hydrochloric and nitric acid concentrations from 25% to 35% wt, and/or by heating samples to higher temperatures using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC) to afford the complete digestion/dissolution of the inorganic contaminants. All samples, controls, and standards were prepared using the same dissolution solution and experimental conditions to ensure both correct instrument calibration and accurate analytical results.
- ICP-OES instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of inorganic compounds. Standardized EPA SW-846 Analytical Method 6010C "Inductively Coupled Plasma-Atomic Emission Spectrometry" was followed as a guideline to prepare all samples and controls.
- To ensure accurate determination of DeconGel decontamination efficacy, calibration standards of the analyte of interest were prepared using either a sufficiently pure analyte or an appropriate ICP-MS Standard (Ricca Chemical Company; Arlington, TX); the respective standards were diluted to a known concentration (ppm) using the same digestion method as the one used for samples and controls. Instrument blank controls were DI H_2O ($\geq$17 M-Ohm).
- Elemental cadmium was received as a large-mesh metal, and was the smallest mesh commercially available. Even so, the large mesh size of cadmium metal contaminant was deemed unusable in an experimental setting, such that metal particles were unable to be spread into a homogenous layer. As such, the contaminant was eliminated from the experimental design in preference of other more suitable forms of cadmium.

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.05 to 0.10 g of contaminant was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), 3) aluminum (commercial grade, surface area: 56.3 cm^2) or 4) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24-48 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe and gel samples were suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% deionized (DI) H_2O) for 24 h. When deemed necessary, samples were heated to 94 °C for 4-14 h to effectively complete dissolution of inorganics using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC). Samples were allowed to cool to room temperature and were then analyzed via ICP-OES (see below).

Controls Methods

For Swipe Control samples, 0.05 g of contaminant was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O) for 24 h and analyzed via ICP-OES (see below).

For DeconGel Control samples, a respectable amount of contaminant (0.10 or 0.05g) and approximately 6.0 g of dry DeconGel 1101 (pre-poured gel on the respective un-contaminated substrate and let to dry for 24-48 h) was suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O) for 24 h and analyzed via ICP-OES (see below).

Reagents and Standards

Reagent grade Cadmium Chloride, $CdCl_2$, (CAS # 7790-78-5, Fisher Scientific; Fair Lawn, NJ), was used as received. Reagant grade Cadmium Oxide, CdO, (CAS# 1306-19-0, Fisher Scientific; Fair Lawn, NJ), was used as received

A 1000 ppm calibration standard of each contaminant was prepared using the reagent grade cadmium compounds and freshly prepared aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O). DI H_2O was used as the blank sample.

Analytical Instrumentation

A Thermo ICP-OES instrument model radial iCap 6300 was used to determine cadmium concentration (ppm) of all samples and controls using a freshly prepared 1000 ppm calibration standard.

Analyte (cadmium) analyzed at 228.8 nm

Pump Speed: 0.5 mL/min

6.11. Technical Report for Cyanide (Potassium Ferricyanide)

TITLE: Surface Decontamination of Cyanide Compounds (Potassium Ferricyanide ($K_3[Fe(CN)_6]$)) by DeconGel 1101

AUTHOR: Andreas Mylonakis PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with potassium ferricyanide using ICP-OES (Inductively Coupled Plasma-Optical Emission Spectroscopy) following Environmental Protection Agency (EPA) SW-846 Methods 3005A (sampling) and 6010C (analysis).

HAZARDOUS MATERIALS RELEVANCE: Cyanide can cause acute toxicity through skin contact, ingestion, and inhalation. Potassium ferricyanide ($K_3[Fe(CN)_6]$) is an iron-containing compound possessing similar physical characteristics to potassium cyanide (KCN), a water soluble form of cyanide. Cyanide is utilized in gold and silver mining and electroplating industrial applications. Both ACGIH[4] and NIOSH[5] have set maximum exposure limits for cyanide compounds. Potassium ferricyanide was chosen as a representative cyanide compound for evaluating DeconGel's efficacy; DeconGel is expected to have similar efficacy towards the wide range of cyanide compounds.

SUMMARY RESULTS:

- Excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with cyanide compounds resulting in encapsulation of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 98.2% (on concrete) to 98.9% (on carbon steel) to 99.4% (on stainless steel) as determined by swipe analysis, and from 92.0% (on concrete) to 94.4% (on carbon steel) to 95.7% (on stainless steel) as determined by direct DeconGel analysis for potassium ferricyanide.

- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of inorganic compounds in aqueous samples. When deemed necessary, experimental methods were customized to afford complete

---

[4] American Conference of Industrial Hygienists (ACGIH); http://www.acgih.org/home.htm (2010)
[5] National Institute for Occupational Safety and Health (NIOSH); http://www.cdc.gov/niosh/ (2010)

dissolution of inorganic contaminants, and to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 and 2 show the decontamination efficacies of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with cyanide as determined by residual swipe testing and direct DeconGel analysis respectively.

Table 1. Decontamination efficacies of DeconGel against potassium ferricyanide contaminated stainless steel, carbon steel and concrete surfaces as determined by residue swipe testing method.

Swipe Testing (ppm)		**Formulation**
		DeconGel 1101
Stainless Steel	Control	3.31 ± 0.01
	Residual	0.020 ± 0.007
	Decon. Efficacy (%)	**99.4 ± 4.2**
Carbon Steel	Control	3.45 ± 0.06
	Residual	0.036 ± 0.018
	Decon. Efficacy (%)	**98.9 ± 3.4**
Concrete	Control	3.13 ± 0.07
	Residual	0.057 ± 0.045
	Decon. Efficacy (%)	**98.2 ± 3.2**

1400000x dilution factor for samples and controls

Table 2. Decontamination efficacies of DeconGel against potassium ferricyanide contaminated stainless steel, carbon steel, and concrete surfaces as determined by direct DeconGel analysis.

Direct DegonGel Analysis (ppm)		**Formulation**
		DeconGel 1101
Stainless Steel	Control	3.37 ± 0.03
	Encapsulated in Gel	3.23 ± 0.09
	Decon. Efficacy (%)	**95.7 ± 2.4**
Carbon Steel	Control	3.45 ± 0.06
	Encapsulated in Gel	3.25 ± 0.09
	Decon. Efficacy (%)	**94.4 ± 3.0**
Concrete	Control	3.34 ± 0.12
	Encapsulated in Gel	3.07 ± 0.14
	Decon. Efficacy (%)	**92.0 ± 5.4**

1400000x dilution factor for samples and controls

METHOD DEVELOPMENT OPTIMIZATIONS/OBSERVATIONS:

- We noted that amounts of contaminant solid greater than 0.05 g were difficult to spread evenly throughout coupon surfaces, forming clumps and piles which tended to overload poured DeconGel, significantly limiting the ability of DeconGel to encapsulate/emulsify surface contamination. This issue was circumvented by applying a modest amount (0.05 g) of contaminant solid throughout the surface of interest. Application of a homogenous, thin layer of contaminant (a modest amount of 0.05g) on the respective coupon substrates facilitated an optimized interaction between contaminant and DeconGel as well as an accurate measure of DeconGel's decontamination efficacy in a scaled-down yet real-world setting. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel. Potassium ferricyanide solvated in acidic aqueous solutions readily liberates toxic cyanide gas, therefore $KFe[Fe(CN)_6]$ was dissolved in deionized water (DI H_2O ($\geq$17 M-Ohm)). DI water was used to prepare all samples and controls.

- ASTM method E1728-03 (a standardized swipe testing method used for the sampling of inorganic contaminants) was the integral method used to accurately evaluate DeconGel's decontamination efficacy. GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes (pre-wetted with DI H_2O) were utilized in this swipe testing method.

- Direct chemical analysis of the dried peeled DeconGel samples was also utilized to provide an improved understanding of DeconGel's decontamination efficacy.

- Standardized EPA SW-846 Sampling Method 3005A "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy" was followed as a guideline to prepare all samples and controls. To reduce the viscosity of samples and controls, all samples and controls were diluted by 100x. All samples, controls, and standards were prepared using the same dissolution solution and experimental conditions to ensure both correct instrument calibration and accurate analytical results.

- ICP-OES instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of inorganic compounds. Standardized EPA SW-846 Analytical Method 6010C "Inductively Coupled Plasma-Atomic Emission Spectrometry" was followed as a guideline to prepare all samples and controls.

- To ensure accurate determination of DeconGel decontamination efficacy, calibration standards of the analyte of interest were prepared using either a sufficiently pure analyte or an appropriate ICP-MS Standard (Ricca Chemical Company; Arlington, TX); the respective standards were diluted to a known concentration (ppm) using the same digestion method as that used for samples and controls. Instrument blank controls were DI H_2O ($\geq$17 M-Ohm).

- Prussian blue (potassium ferrous ferricyanide, $KFe[Fe(CN)_6],H_2O$) is an insoluble pigment which was initially considered for DeconGel decontamination efficacy evaluations, however Prussian blue is insoluble in both water and in acidic and/or basic environments. Prussian blue was disregarded from further evaluation tests.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

Decontamination Efficacy (DeconGel Testing) =

(Contaminant (ppm) of DeconGel Direct/Contaminant (ppm) of DeconGel Control) x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.05 g of contaminant was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24-48 hours. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe and gel samples were suspended in 100 mL of DI H_2O and shaken vigorously. After 24 hours, the samples were shaken again prior to a 1:100 dilution in DI H_2O. The samples were then shaken again and analyzed via ICP-OES (see below).

Controls Methods

For Swipe Control samples, 0.05 g of contaminant was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 100 mL DI H_2O and shaken vigorously. After 24 hours, the samples were shaken again prior to a 1:100 dilution in DI H_2O. The control samples were then shaken again and analyzed via ICP-OES.

For DeconGel Control samples, 0.05g of contaminant and approximately 6.0 g of dry DeconGel 1101 (pre-poured gel on the respective un-contaminated substrate and let to dry for 24-48 hours) was suspended in 100 mL DI for 24 h and shaken vigorously. After 24 hours, the samples were shaken again prior to a 1:100 dilution in DI H_2O. The control samples were then shaken again and analyzed via ICP-OES (see below).

Reagents and Standards

Potassium ferricyanide, ($K_3[Fe(CN)_6]$), (CAS# 13746-66-2, Fisher Scientific; Fair Lawn, NJ), was used as received.

A 10.0 ppm calibration standard was prepared by dilution and iron ICP-MS Standard (Ricca Chemical Company; Arlington, TX) to the proper concentration. DI H_2O ($\geq$ 17 M-Ohm) was used as the blank sample.

Analytical Instrumentation

A Thermo ICP-OES instrument model radial iCap 6300 was used to determine iron concentration (ppm) of all samples and controls using a freshly prepared 1000 ppm calibration standard.

Analyte (iron) was analyzed at 271.4 nm

Pump Speed: 0.5 mL/min

6.12. Technical Report for Halogenated Solvents (Tetrachloroethylene)

TITLE: Surface Decontamination of Halogenated Solvents (Tetrachloroethylene) by DeconGel 1101 and 1102

AUTHOR: Geoff Nadolski PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel formulation 1101 and 1102 on stainless steel, floor tile, and concrete surfaces contaminated with Halogenated Solvents (tetrachloroethylene (TCE)) using LC/MS (Liquid Chromatography/Mass Spectrometry) following Environmental Protection Agency (EPA) SW-846 Methods 3500C (Sampling) and 8321B (Analysis).

HAZARDOUS MATERIALS RELEVANCE: TCE is a volatile chlorinated hydrocarbon widely used as a solvent, dry-cleaning fluid, and degreaser. TCE is a common soil contaminant and is an environmentally persistent pollutant. TCE is classified as a carcinogen, and is a skin irritant and central nervous system depressant. TCE was chosen as a representative halogenated solvent; DeconGel is expected to have similar efficacy towards the wide range of halogenated solvents.

SUMMARY RESULTS:

- Excellent surface decontamination was achieved by applying both DeconGel 1101 and 1102, both via brushing or pouring (non-brushed) onto contaminated surfaces, resulting in encapsulation of TCE contaminant by DeconGel's active components. Decontamination efficacies of poured DeconGel 1101 ranged from 100% (on concrete) to 100% (on floor tile) to 99.6% (on stainless steel), brushed DeconGel 1101 ranged from 100% (on concrete) to 100% (on floor tile) to 100% (on stainless steel), as determined by residual swipe analysis. Decontamination efficacies of poured DeconGel 1102 ranged from 100% (on concrete) to 100% (on floor tile) to 99.5% (on stainless steel), brushed DeconGel 1102 ranged from 100% (on concrete) to 100% (on floor tile) to 100% (on stainless steel), as determined by residual swipe analysis. Decontamination efficacies of DeconGel 1101 ranged from 80.5% (on concrete) to 95.8% (on stainless steel) to 95.9% (on floor tile), DeconGel 1102 ranged from 78.5% (on concrete) to 95.9% (on stainless steel) to 95.9% (on floor tile), as determined by direct DeconGel analysis.
- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of organic compounds dissolved in a suitable solvent able to completely solvate TCE as well as DeconGel components. When deemed

necessary, experimental methods were customized to afford complete dissolution of organic contaminants. Additionally, analytical methods and associated equipment (LC column, LC gradient program, MS sample ionization parameters) were appropriately developed to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 and 2 show the decontamination efficacies of DeconGel 1101 and 1102 on stainless steel, floor tile, and concrete surfaces contaminated with Halogentated Solvents (TCE) as determined by residual swipe testing and direct DeconGel analysis.

Table 1. Decontamination efficacies of DeconGel 1101 and 1102 on TCE Halogenated Solvent contaminated stainless steel, floor tile, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation	
		DeconGel 1101	**DeconGel 1102**
Stainless Steel	Control	936.24 ± 2.55	936.24 ± 2.55
	Residual (non-brushed)	4.20 ± 0.05	5.02 ± 0.05
	Residual (brushed)	ND*	ND
	Decon. Efficacy (non-brushed) (%)	**99.6 ± 0.01**	**99.5 ± 0.0**
	Decon. Efficacy (brushed) (%)	**100****	**100****
Floor Tile	Control	934.98 ± 1.52	934.98 ± 1.52
	Residual (non-brushed)	ND	ND
	Residual (brushed)	ND	ND
	Decon. Efficacy (non-brushed) (%)	**100****	**100****
	Decon. Efficacy (brushed) (%)	**100****	**100****
Concrete	Control	813.67 ± 2.92	813.67 ± 2.92
	Residual (non-brushed)	ND	ND
	Residual (brushed)	ND	ND
	Decon. Efficacy (non-brushed) (%)	**100****	**100****
	Decon. Efficacy (brushed) (%)	**100****	**100****

678x dilution factor for samples and controls

* ND: not detected, limit of detection (LOD) for TCE approximates 250 ppb

** An ND value for residual experiments results in a Decontamination Efficacy of 100%

Table 2. Decontamination efficacies of DeconGel 1101 and 1102 on TCE Halogenated Solvent contaminated stainless steel, floor tile, and concrete surfaces as determined by direct DeconGel analysis.

Direct DeconGel Testing (ppm)		**Formulation**	
		DeconGel 1101	**DeconGel 1102**
Stainless Steel	Control	973.08 ± 1.53	973.08 ± 1.53
	Encapsulated in Gel (non-brushed)	932.63 ± 1.06	932.76 ± 1.81
	Decon. Efficacy (non-brushed) (%)*	**95.8 ± 0.22**	**95.9 ± 0.32**
Floor Tile	Control	972.83 ± 0.79	972.83 ± 0.79
	Encapsulated in Gel (non-brushed)	932.98 ± 0.76	932.64 ± 0.93
	Decon. Efficacy (non-brushed) (%)	**95.9 ± 0.10**	**95.9 ± 0.16**
Concrete	Control	972.71 ± 0.67	972.71 ± 0.67
	Encapsulated in Gel (non-brushed)	782.67 ± 0.73	763.21 ± 1.39
	Decon. Efficacy (non-brushed) (%)	**80.5 ± 0.01**	**78.5 ± 0.27**

678x dilution factor for samples and controls

* Decontamination Efficacy by direct DeconGel testing was determined only for non-brushed films, such that brushing DeconGel onto contaminated surfaces results in a loss of some portion of contaminant on brushing tool.

METHOD DEVELOPMENT OPTIMIZATIONS/OBSERVATIONS:

- We noted that amounts of contaminant liquid greater than 100 uL were difficult to spread evenly throughout coupon surfaces, forming zones of liquid saturation which tended to overload poured DeconGel, significantly limiting the ability of DeconGel to encapsulate/emulsify surface contamination. This issue was circumvented by applying a modest amount (50 uL) of contaminant liquid throughout the surface of interest using a small brush. Application of a homogenous, thin layer of contaminant on the respective substrate facilitated an optimized interaction between contaminant and DeconGel, and provided an accurate measure of DeconGel's decontamination efficacy in a scaled-down yet real-world setting. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel. TCE readily dissolves in DMSO (50 mL) used to prepare all analytical samples and controls.

- Acceptable direct DeconGel decontamination efficacies ranging from 80.5 to 95.9% for DeconGel 1101, and decontamination efficacies ranging from 78.5 to 95.9% for DeconGel 1102 were achieved, however, because TCE is prone to evaporate over the required 24 h drying time needed by DeconGel once applied to a contaminated surface, this could contribute to lower than expected DeconGel decontamination efficacies determined by the direct DeconGel testing method.

- ASTM method E1728-03, a standardized swipe testing method used for sampling of contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. Air-dried GhostWipe™

(Environmental Express; Mt. Pleasant, SC) swipes were utilized in this swipe testing method.

- Direct chemical analysis of the dried peeled DeconGel samples was also utilized to provide an improved understanding of DeconGel's decontamination efficacy. Decontamination efficacy was determined only for non-brushed (poured) DeconGel films, because brushing DeconGel onto contaminated surfaces resulted in a loss of some portion of contaminant on the brushing tool used, and as such accurate decontamination efficacies for brushed DeconGel 1101 and 1102 could not be determined. For brushed films, accurate decontamination efficacies were best determined utilizing swipe testing.
- Standardized EPA SW-846 Sampling Method 3500C "Organic Extraction and Sample Preparation" was followed as a guideline to prepare all samples and controls. All samples, controls, and standards were prepared using the same solvent and appropriate experimental conditions to ensure accurate and LC/MS instrumental analysis.
- LC/MS instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of organic compounds. Standardized EPA SW-846 Analytical Method 8321B "Solvent-Extractable Nonvolatile Compounds by High-Performance Liquid Chromatography/Thermospray/Mass Spectrometry (HPLC/TS/MS) or Ultraviolet (UV) Detection" was followed as a guideline to prepare all samples and controls.
- To ensure accurate determination of DeconGel decontamination efficacy, a standard curve of the analyte of interest was prepared using sufficiently pure analyte; the respective standards were diluted to a known concentration (ppm) using the same solvent as used for samples and controls.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

Decontamination Efficacy (DeconGel Testing) =

(Contaminant (ppm) of DeconGel Direct/Contaminant (ppm) of DeconGel Control) x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 50 uL of contaminant was evenly applied via brushing on 1) stainless steel (surface area: 56.3 cm^2), 2) floor tile (surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe and gel samples were suspended in 50 mL DMSO for 24 h. Samples were then analyzed via LC/MS (see below).

Controls Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied via brushing on 1) stainless steel (surface area: 56.3 cm^2), 2) floor tile (surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 50 mL DMSO for 24 h and analyzed via LC/MS (see below).

For DeconGel Control samples, a respectable amount of contaminant and approximately 6.0 g of dry DeconGel 1101 (pre-poured gel on the respective uncontaminated substrate and let to dry for 24 h) was suspended in 50 mL DMSO for 24 h and analyzed via LC/MS (see below).

Reagents and Standards

Tetrachloroethylene (TCE) (tetrachloroethene) (CAS# 127-18-4, Fisher Scientific; Fair Lawn, NJ) was used as received.

Analytical Instrumentation

A Thermo LCQ LC/MS with autosampler in positive mode was used to determine toluene concentration (ppm, wt/wt) of all samples and controls, using a Grace Davison (Deerfield, IL) C18-Select analytical column (150 x 4.6 mm, 5 um).

A 9-point standard curve derived from three independently prepared stock solutions was prepared using DMSO as the working solvent.

LC method using A= water (0.1% formic acid), B=acetonitrile (0.1% formic acid); start at 95%A to 3 min at 70%A, to 6 min at 50%A, to 12 min at 30%A, to 15 min at 90%B, to 16 min at 100%B, hold until 22 min at 100% B, to 23 min at 95%A, hold until 24 min.

TCE LC/MS data: 15.4 min; lambda max = 238 nm; M^+ = 165.

6.13. Technical Report for Hydrocarbon Petroleum Distillates (Kerosene)

TITLE: Surface Decontamination of Hydrocarbon Petroleum Distillates (Kerosene) by DeconGel 1101 and 1102

AUTHOR: Geoff Nadolski PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel formulation 1101 and 1102 on stainless steel, aluminum, and concrete surfaces contaminated with Hydrocarbon Petroleum Distillates (kerosene) using GC/MS (Gas Chromatography/Mass Spectrometry) following Environmental Protection Agency (EPA) SW-846 Methods 3500C (Sampling) and 8270C (Analysis).

HAZARDOUS MATERIALS RELEVANCE: Kerosene is a highly flammable, volatile hydrocarbon liquid petroleum distillate mixture containing between six to sixteen carbon atom molecules. Kerosene is used as a heating fuel, propellant, and solvent and thinner. Ingestion of kerosene is harmful and in sufficient quantities can be fatal. Kerosene was chosen as a representative petroleum distillate; DeconGel is expected to have similar efficacy towards the wide range of hydrocarbon petroleum distillates.

SUMMARY RESULTS:

- Excellent surface decontamination was achieved by applying both DeconGel 1101 and 1102, both via brushing or pouring (non-brushed) onto contaminated surfaces, resulting in encapsulation of kerosene by DeconGel's active components. Decontamination efficacies of poured DeconGel 1101 ranged from 92.8% (on concrete) to 95.8% (on stainless steel) to 96.0% (on aluminum), brushed DeconGel 1101 ranged from 100% (on concrete) to 99.8% (on stainless steel) to 99.8% (on aluminum), as determined by residual swipe analysis. Decontamination efficacies of poured DeconGel 1102 ranged from 100% (on concrete) to 100% (on aluminum) to 100% (on stainless steel), brushed DeconGel 1102 ranged from 100% (on concrete) to 100% (on aluminum) to 100% (on stainless steel), as determined by residual swipe analysis. Decontamination efficacies of DeconGel 1101 ranged from 74.8% (on concrete) to 90.1% (on aluminum) to 90.9% (on stainless steel), DeconGel 1102 ranged from 76.8% (on concrete) to 92.2% (on stainless steel) to 92.7% (on aluminum), as determined by direct DeconGel analysis.

- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of organic compounds dissolved in a suitable solvent able to completely solvate kerosene as well as DeconGel components. When necessary, experimental methods were customized to afford complete dissolution of organic contaminants. Additionally, analytical methods and associated equipment (GC column, GC temperature gradient program, MS sample ionization parameters) were appropriately utilized to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 and 2 show the decontamination efficacies of DeconGel 1101 and 1102 on stainless steel, aluminum, and concrete surfaces contaminated with Hydrocarbon Petroleum Distillates (kerosene) as determined by residual swipe testing and direct DeconGel analysis.

Table 1. Decontamination efficacies of DeconGel 1101 and 1102 on Kerosene Hydrocarbon Petroleum Distillate contaminated stainless steel, aluminum, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation	
		DeconGel 1101	**DeconGel 1102**
Stainless Steel	Control	89.31 ± 0.18	89.31 ± 0.18
	Residual (non-brushed)	3.76 ± 0.07	ND*
	Residual (brushed)	0.112 ± 0.001	ND
	Decon. Efficacy (non-brushed) (%)	**95.8 ± 0.12**	**100****
	Decon. Efficacy (brushed) (%)	**99.8 ± 0.06**	**100****
Aluminum	Control	89.34 ± 0.30	89.34 ± 0.30
	Residual (non-brushed)	3.59 ± 0.10	ND
	Residual (brushed)	0.111 ± 0.002	ND
	Decon. Efficacy (non-brushed) (%)	**96.0 ± 0.10**	**100****
	Decon. Efficacy (brushed) (%)	**99.8 ± 0.06**	**100****
Concrete	Control	70.38 ± 1.37	70.38 ± 1.37
	Residual (non-brushed)	5.10 ± 0.02	ND
	Residual (brushed)	ND	ND
	Decon. Efficacy (non-brushed) (%)	**92.8 ± 0.16**	**100****
	Decon. Efficacy (brushed) (%)	**100****	**100****

412x dilution factor for samples and controls

* ND: not detected, limit of detection (LOD) for kerosene approximates 250 ppb

** An ND value for residual experiments results in a Decontamination Efficacy of 100%

Table 2. Decontamination efficacies of DeconGel 1101 and 1102 on Kerosene Hydrocarbon Petroleum Distillate contaminated stainless steel, aluminum, and concrete surfaces as determined by direct DeconGel analysis.

Direct DeconGel Testing (ppm)		Formulation	
		DeconGel 1101	**DeconGel 1102**
Stainless Steel	Control	114.86 ± 1.16	114.61 ± 1.20
	Encapsulated in Gel (non-brushed)	104.45 ± 1.10	105.91 ± 0.68
	Decon. Efficacy (non-brushed) (%)*	**90.9 ± 0.86**	**92.2 ± 1.53**
Aluminum	Control	114.86 ± 1.16	114.61 ± 1.20
	Encapsulated in Gel (non-brushed)	103.48 ± 0.44	106.48 ± 0.44
	Decon. Efficacy (non-brushed) (%)	**90.1 ± 0.89**	**92.7 ± 0.86**
Concrete	Control	114.86 ± 1.16	114.61 ± 1.20
	Encapsulated in Gel (non-brushed)	85.92 ± 0.78	88.13 ± 0.14
	Decon. Efficacy (non-brushed) (%)	**74.8 ± 1.39**	**76.8 ± 1.02**

412x dilution factor for samples and controls

* Decontamination Efficacy by direct DeconGel testing was determined only for non-brushed films, such that brushing DeconGel onto contaminated surfaces results in a loss of some portion of contaminant on brushing tool.

METHOD DEVELOPMENT OPTIMIZATIONS/OBSERVATIONS:

- We noted that amounts of contaminant liquid greater than 100 uL were difficult to spread evenly throughout coupon surfaces, forming zones of liquid saturation which tended to overload poured DeconGel, significantly limiting the ability of DeconGel to encapsulate/emulsify surface contamination. This issue was circumvented by applying a modest amount (50 uL) of contaminant liquid throughout the surface of interest using a small brush. Application of a homogenous, thin layer of contaminant on the respective substrate facilitated an optimized interaction between contaminant and DeconGel, and provided an accurate measure of DeconGel's decontamination efficacy in a scaled-down yet real-world setting. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel. Kerosene readily dissolves in hexanes (50 mL) used to prepare all analytical samples and controls.

- Acceptable direct DeconGel decontamination efficacies ranging from 74.8 to 90.9% for DeconGel 1101, and decontamination efficacies ranging from 76.8 to 92.7% for DeconGel 1102 were achieved, however, because kerosene is prone to evaporate over the required 24 h drying time needed by DeconGel once applied to a contaminated surface, this could contribute to lower than expected DeconGel decontamination efficacies determined by the direct DeconGel testing method.

- ASTM method E1728-03, a standardized swipe testing method used for sampling of contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. Air-dried GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes were utilized in this swipe testing method.

- Direct chemical analysis of the dried peeled DeconGel samples was also utilized to provide an improved understanding of DeconGel's decontamination efficacy. Decontamination efficacy was determined only for non-brushed (poured) DeconGel films, because brushing DeconGel onto contaminated surfaces resulted in a loss of some portion of contaminant on the brushing tool used, and as such accurate decontamination efficacies for brushed DeconGel 1101 and 1102 could not be determined. For brushed films, accurate decontamination efficacies were best determined utilizing a residual swipe testing method.

- Standardized EPA SW-846 Sampling Method 3500C "Organic Extraction and Sample Preparation" was followed as a guideline to prepare all samples and controls. All samples, controls, and standards were prepared using the same solvent and appropriate experimental conditions to ensure accurate and GC/MS instrumental analysis.

- GC/MS instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of organic compounds. Standardized EPA SW-846 Analytical Method 8270C "SEMIVOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)" was followed as a guideline to prepare all samples and controls.

- To ensure accurate determination of DeconGel decontamination efficacy, a standard curve of the analyte of interest was prepared using sufficiently pure analyte; the respective standards were diluted to a known concentration (ppm) using the same solvent as used for samples and controls.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

Decontamination Efficacy (DeconGel Testing) =

(Contaminant (ppm) of DeconGel Direct/Contaminant (ppm) of DeconGel Control) x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 100 uL of contaminant was evenly applied via brushing on 1) stainless steel (commercial grade, surface area: 56.3 cm^2), 2) aluminum (commercial grade, surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 50 mL hexane, and gel samples were first suspended in 20 mL water and then 20 mL hexane was added, and let to stand for 24 h. For gel samples, the water layer was extracted two more times with first 20 mL hexane and then 10 mL hexane, such that the hexane layers (50 mL total) were combined. Samples were then analyzed via GC/MS (see below).

Controls Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied via brushing on 1) stainless steel (commercial grade, surface area: 56.3 cm^2), 2) floor tile (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 50 mL hexane for 24 h and analyzed via GC/MS (see below).

For DeconGel Control samples, a respectable amount of contaminant and approximately 6.0 g of dry DeconGel 1101 (pre-poured gel on the respective uncontaminated substrate and let to dry for 24 h) was first suspended in 20 mL water and then 20 mL hexane was added, and let to stand for 24 h. The water layer was extracted two more times with first 20 mL hexane and then 10 mL hexane, such that the hexane layers (50 mL total) were combined and analyzed via GC/MS (see below).

Reagents and Standards

Kerosene, odorless (CAS# 64742-14-9, Acros; NJ) was used as received.

Analytical Instrumentation

A Thermo DSQII GC/MS with autosampler in positive mode was used to determine EG concentration (ppm, wt/wt) of all samples and controls, using a Restek (Bellefonte, PA) Rtx-5 capillary column (30 m x 0.25 mm, 0.5 um). A 9-point standard curve derived from three independently prepared stock solutions was prepared using DMSO as the working solvent.

GC method: start at 45 ºC, hold for 7 min, ramp at 30 ºC/min to 320 ºC, hold 10 min.

Kerosene GC/MS data: 11.5 min; M+ = 170 (C12H26)

6.14. Technical Report for Iodine (Potassium Iodide)

TITLE: Surface Decontamination of Iodine Compounds (Potassium Iodide (KI)) by DeconGel 1101

AUTHOR: Andreas Mylonakis PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel 1101 on stainless steel, carbon steel, floor tile, and concrete surfaces contaminated with Potassium Iodide (KI) using ICP-OES (Inductively Coupled Plasma-Optical Emission Spectroscopy) following Environmental Protection Agency (EPA) SW-846 Methods 3005A (sampling) and 6010C (analysis).

HAZARDOUS MATERIALS RELEVANCE: Iodine and its compounds are used in medicine, photography, and the dye industry. Iodides are soluble in water and are concentrated in seawater. Iodine is an oxidizing irritant and allergen and is poisonous if taken orally in large amounts. Potassium iodide was chosen as a representative iodine compound for evaluating DeconGel's efficacy; DeconGel is expected to have similar efficacy towards the wide range of iodine compounds.

SUMMARY RESULTS:

- Excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with iodine compounds resulting in encapsulation of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 98.6% (on floor tile) to 98.7% (on concrete) to 98.8% (on carbon steel) to 99.5% (on stainless steel) as determined by residual swipe analysis, and from 87.3% (on concrete) to 95.1% (on carbon steel) to 96.0% (on floor tile) to 99.5% (on stainless steel) as determined by direct DeconGel analysis.
- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for the determination of inorganic compounds in aqueous samples. When necessary, experimental methods were customized to afford complete dissolution of inorganic contaminants, and to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 and 2 show the decontamination efficacies of DeconGel 1101 on stainless steel, carbon steel, floor tile and concrete surfaces contaminated with potassium iodide as determined by residual swipe testing and direct DeconGel testing respectively.

Table 1. Decontamination efficacy of DeconGel 1101 on KI contaminated stainless steel, carbon steel, floor tile, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation DeconGel 1101
Stainless Steel	Control	531.3 ± 2.5
	Residual	8.9 ± 4.1
	Decon. Efficacy (%)	**99.5 ± 2.9**
Carbon Steel	Control	521.8 ± 11.6
	Residual	6.2 ± 4.8
	Decon. Efficacy (%)	**98.8 ± 6.8**
Floor Tile	Control	293.3 ± 19.8
	Residual	4.14 ± 3.30
	Decon. Efficacy (%)	**98.6 ± 1.1**
Concrete	Control	514.4 ± 11.5
	Residual	6.7 ± 2.8
	Decon. Efficacy (%)	**98.7 ± 3.2**

21880x dilution factor for samples and controls

Table 2. Decontamination efficacy of DeconGel 1101 on KI on stainless steel, carbon steel, floor tile, and concrete surfaces as determined by direct DeconGel analysis.

Direct DeconGel Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	512.3 ± 3.7
	Encapsulated in Gel	509.9 ± 14.4
	Decon. Efficacy (%)	**99.5 ± 2.9**
Carbon Steel	Control	512.4 ± 6.9
	Encapsulated in Gel	487.5 ± 25.2
	Decon. Efficacy (%)	**95.1 ± 5.1**
Floor Tile	Control	446.8 ± 6.4
	Encapsulated in Gel	428.9 ± 25.8
	Decon. Efficacy (%)	**96.0 + 5.8**
Concrete	Control	514.9 ± 19.1
	Encapsulated in Gel	449.6 ± 21.2
	Decon. Efficacy (%)	**87.3 ± 5.2**

21880x dilution factor for samples and controls

METHOD DEVELOPMENT OPTIMIZATIONS/OBSERVATIONS:

- We noted that amounts of contaminant solid greater than 0.05 g were difficult to spread evenly throughout coupon surfaces, forming clumps and piles which tended to overload poured DeconGel, significantly limiting the ability of DeconGel to encapsulate/emulsify surface contamination. This issue was circumvented by applying a modest amount (0.05 g) of KI solid throughout the test surfaces by sprinkling KI granules immediately upon weighing them out.

- Application of a homogenous, thin layer of contaminant on the respective substrate facilitated an optimized interaction between contaminant and DeconGel, and provided an accurate measure of DeconGel's decontamination efficacy in a scaled-down yet real-world setting. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel. Solid KI readily dissolves in aqueous acidic solutions used to prepare all analytical samples and controls (15% HCl, 15% HNO_3, 70% DI H_2O).

- Evaluation of the decontamination efficacy of DeconGel on KI on waxed floor tile was conducted to simulate an iodine/iodide spill as evidenced in a medical/clinical setting, such that the formed KI deposit is intended to simulate a radioactive and/or pharmaceutical form of iodine/iodide. DeconGel effectively decontaminated several forms of iodide contamination (from loose residual solids to dense deposits) on multiple types of surfaces (from non-porous, inert metals to waxy, chemically active plastics, to porous substrates).

- ASTM method E1728-03, a standardized swipe testing method used for sampling of inorganic contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes (pre-wetted with DI H_2O) were utilized in this swipe testing method.

- Direct chemical analysis of the dried peeled DeconGel samples was also utilized to provide an improved understanding of DeconGel's decontamination efficacy.

- Standardized EPA SW-846 Sampling Method 3005A "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy" was followed as a guideline to prepare all samples and controls. When necessary, digestion methods were customized to afford complete dissolution of inorganic contaminants by increasing hydrochloric and nitric acid concentrations from 20% to 30% wt, and/or by heating samples to higher temperatures using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC) All samples, controls, and standards were prepared using the same dissolution solution and experimental conditions to ensure both correct instrument calibration and accurate analytical results.

- ICP-OES instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of inorganic compounds. Standardized EPA SW-846 Analytical Method 6010C "Inductively Coupled Plasma-Atomic Emission Spectrometry" was followed as a guideline to prepare all samples and controls.

- To ensure accurate determination of DeconGel decontamination efficacy, calibration standards of the analyte of interest were prepared using either a sufficiently pure analyte or an appropriate ICP-MS Standard (Ricca Chemical Company; Arlington, TX); the respective standards were diluted to a known concentration (ppm) using the same digestion method as the one used for samples and controls. Instrument blank controls were DI H_2O ($\geq$17 M-Ohm).

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(KI (ppm) of Swipe Control) – (KI (ppm) of Residual Swipe)/KI (ppm) of Swipe Control] x 100%

Decontamination Efficacy (DeconGel Testing) =

(KI (ppm) of DeconGel Direct/KI (ppm) of DeconGel Control) x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.05 g KI was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons; for waxed floor tile (surface area: 221.9 cm^2), 1.0 mL aqueous KI (50 mg/mL) was homogeneously pipetted onto the coupon surface and let to stand for one week until all water had evaporated and a KI deposit had formed. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24-48 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe and gel samples were suspended in 100 mL aqueous acidic solution (15% HCl, 15% HNO_3, 70% deionized (DI) H_2O) for 24 h. When deemed necessary, samples were heated to 90 ^oC for 8-24 h to afford complete dissolution of inorganics using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC). Samples were allowed to cool to room temperature and were then analyzed via ICP-OES (see below).

Controls Methods

For Swipe Control samples, 0.05 g KI was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons: for waxed floor tile (surface area: 221.9 cm^2), 1.0 mL aqueous KI (50 mg/mL) was homogeneously pipetted onto the coupon surface and let to stand for one week until all water had evaporated and a KI deposit had formed. Coupon surfaces were swipe tested (ASTM method 1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 100 mL aqueous acidic solution (15% HCl, 15% HNO_3, 70% DI H_2O) for 24 h and analyzed via ICP-OES (see below).

For DeconGel Control samples, 0.05 g KI and approximately 6.0 g dry DeconGel (pre-poured gel on the respective un-contaminated substrate and let to dry for 24-48 h) was suspended in 100 mL aqueous acidic solution (15% HCl, 15% HNO_3, 70% DI H_2O) for 24 h and analyzed via ICP-OES (see below).

Reagents and Standards

Reagent grade potassium iodide (KI) (CAS# 7681-11-0, Fisher Scientific; Fair Lawn, NJ) was used as received. A 10.0 ppm calibration standard was prepared using reagent grade granular KI and freshly prepared aqueous acidic solution (15% HCl, 15% HNO_3, 70% DI H_2O). DI H_2O was used as the blank sample.

Analytical Instrumentation

A Thermo ICP-OES instrument model Radial iCap 6300 was used to determine KI concentration (ppm) of all samples and controls using a freshly prepared 10.0 ppm calibration standard.

Analyte (iodine) analyzed at 183.0 nm. Pump Speed: 0.5 mL/min

6.15. Technical Report for Industrial Coolants (Ethylene Glycol)

TITLE: Surface Decontamination of Industrial Coolants (Ethylene Glycol) by DeconGel 1101

AUTHOR: Geoff Nadolski PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel formulation 1101 on stainless steel, aluminum, and concrete surfaces contaminated with Industrial Coolants (ethylene glycol (EG)) using GC/MS (Gas Chromatography/Mass Spectrometry) following Environmental Protection Agency (EPA) SW-846 Methods 3500C (Sampling) and 8270C (Analysis).

HAZARDOUS MATERIALS RELEVANCE: EG is viscous odorless liquid used widely as a coolant/heat transfer agent. EG is also used as a dessicant, and as a polymer precursor, and is moderately orally toxic to humans. Ingestion of large amounts of EG can be fatal if untreated. EG was chosen as a representative industrial coolant; DeconGel is expected to have similar efficacy towards the wide range of industrial coolants.

SUMMARY RESULTS:

- Excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with industrial coolants (EG) resulting in encapsulation of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 93.6% (on concrete) to 95.5% (on stainless steel) to 95.5% (on aluminum) for EG determined by residual swipe analysis, and 79.5% (on concrete) to 97.4% (on stainless steel) to 97.5% (on aluminum) for EG as determined by direct DeconGel analysis.

- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of organic compounds dissolved in a suitable solvent able to completely solvate aniline as well as DeconGel components. When necessary, experimental methods were customized to afford complete dissolution of organic contaminants. Additionally, analytical methods and associated equipment (GC column, GC temperature gradient program, MS sample ionization parameters) were appropriately utilized to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 and 2 show the decontamination efficacies of DeconGel 1101 on stainless steel, aluminum, and concrete surfaces contaminated with

Industrial Coolants (EG) as determined by residual swipe testing and direct DeconGel analysis.

Table 1. Decontamination efficacy of DeconGel 1101 on Industrial Coolant Ethylene Glycol contaminated stainless steel, aluminum, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	203.43 ± 0.29
	Residual	9.08 ± 0.01
	Decon. Efficacy (%)	**95.5 ± 0.01**
Aluminum	Control	203.61 ± 1.03
	Residual	9.08 ± 0.02
	Decon. Efficacy (%)	**95.5 ± 0.01**
Concrete	Control	155.31 ± 0.74
	Residual	9.97 ± 0.03
	Decon. Efficacy (%)	**93.6 ± 0.01**

1422x dilution factor for samples and controls

Table 2. Decontamination efficacy of DeconGel 1101 on Industrial Coolant Ethylene Glycol contaminated stainless steel, aluminum, and concrete surfaces as determined by direct DeconGel analysis.

Direct DeconGel Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	207.73 ± 0.55
	Encapsulated in Gel	202.33 ± 0.32
	Decon. Efficacy (%)	**97.4 ± 0.41**
Aluminum	Control	206.92 ± 0.57
	Encapsulated in Gel	201.69 ± 0.73
	Decon. Efficacy (%)	**97.5 ± 0.16**
Concrete	Control	206.77 ± 1.55
	Encapsulated in Gel	164.34 ± 0.70
	Decon. Efficacy (%)	**79.5 ± 0.91**

1422x dilution factor for samples and controls

METHOD DEVELOPMENT OPTIMIZATIONS/OBSERVATIONS:

- We noted that amounts of contaminant liquid greater than 100 uL were difficult to spread evenly throughout coupon surfaces, forming zones of liquid saturation which tended to overload poured DeconGel, significantly limiting the ability of DeconGel to encapsulate/emulsify surface contamination. This issue was circumvented by applying a modest amount (35 uL) of contaminant liquid throughout the surface of interest using a small brush. Application of a homogenous, thin layer of contaminant on the respective substrate facilitated an optimized interaction between contaminant and DeconGel, and provided an accurate measure of DeconGel's decontamination efficacy in a scaled-down yet real-world setting. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel. EG readily dissolves in 60% methanol in water (50 mL) used to prepare all analytical samples and controls.

- ASTM method E1728-03, a standardized swipe testing method used for sampling of contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. Air-dried GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes were utilized in this swipe testing method.

- Direct chemical analysis of the dried peeled DeconGel samples was also utilized to provide an improved understanding of DeconGel's decontamination efficacy.

- Standardized EPA SW-846 Sampling Method 3500C "Organic Extraction and Sample Preparation" was followed as a guideline to prepare all samples and controls. All samples, controls, and standards were prepared using the same solvent and appropriate experimental conditions to ensure accurate and GC/MS instrumental analysis.

- GC/MS instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of organic compounds. Standardized EPA SW-846 Analytical Method 8270C "SEMIVOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)" was followed as a guideline to prepare all samples and controls.

- To ensure accurate determination of DeconGel decontamination efficacy, a standard curve of the analyte of interest was prepared using sufficiently pure analyte; the respective standards were diluted to a known concentration (ppm) using the same solvent as used for samples and controls.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

Decontamination Efficacy (DeconGel Testing) =

(Contaminant (ppm) of DeconGel Direct/Contaminant (ppm) of DeconGel Control) x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 25 uL of contaminant was evenly applied via brushing on 1) aluminum (commercial grade, surface area: 56.3 cm^2), 2) stainless steel (commercial grade, surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe and gel samples were suspended in 50 mL 60% methanol in water for 24 h. Samples were then analyzed via GC/MS (see below).

Controls Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied via brushing on 1) aluminum (commercial grade, surface area: 56.3 cm^2), 2) stainless steel (commercial grade, surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 50 mL 60% methanol in water for 24 h and analyzed via GC/MS (see below).

For DeconGel Control samples, a respectable amount of contaminant and approximately 6.0 g of dry DeconGel 1101 (pre-poured gel on the respective uncontaminated substrate and let to dry for 24 h) was suspended in 50 mL 60% methanol in water for 24 h and analyzed via GC/MS (see below).

Reagents and Standards

Ethylene glycol (EG) (ethane-1,2-diol) (CAS# 107-21-1, Fisher Scientific; Fair Lawn, NJ) was used as received.

Analytical Instrumentation

A Thermo DSQII GC/MS with autosampler in positive mode was used to determine EG concentration (ppm, wt/wt) of all samples and controls, using a Restek (Bellefonte, PA) Rtx-5 capillary column (30 m x 0.25 mm, 0.5 um).

A 7-point standard curve derived from three independently prepared stock solutions was prepared using DMSO as the working solvent.

GC method: start at 45 ºC, hold for 1 min, ramp at 30 ºC/min to 320 ºC, hold 10 min.

EG GC/MS data: 2.8 min; $M^+ = 62$.

6.16. Technical Report for Industrial Solvents (*m*-Cresol)

TITLE: Surface Decontamination of Industrial Solvents (*m*-Cresol) by DeconGel 1101

ANALYST: Geoff Nadolski PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel formulation 1101 on stainless steel, aluminum, and concrete surfaces contaminated with Industrial Solvents (*m*-Cresol) using LC/MS (Liquid Chromatography/Mass Spectrometry) following Environmental Protection Agency (EPA) SW-846 Methods 3500C (Sampling) and 8321B (Analysis).

HAZARDOUS MATERIALS RELEVANCE: Meta-Cresol is a methylated aromatic phenolic liquid used as a disinfectant, deodorizer, precursor to insecticides, and as an industrial solvent. Cresol is a phenolic irritant that can cause a severe burning of sensitive tissues, and if ingested or absorbed at high levels can damage the kidneys, liver, brain, and lungs. Cresol was chosen as a representative industrial solvent for evaluating DeconGel's efficacy; DeconGel is expected to have similar efficacy towards the wide range of industrial solvents.

SUMMARY RESULTS:

- Excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with industrial solvents (*m*-cresol) resulting in encapsulation of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 99.4% (on concrete) to 99.8% (on aluminum) to 99.8% (on stainless steel) for *m*-Cresol determined by residual swipe analysis, and 83.8% (on concrete) to 96.4% (on stainless steel) to 96.8% (on aluminum) for *m*-Cresol as determined by direct DeconGel analysis.

- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of organic compounds dissolved in a suitable solvent able to completely solvate nicotine as well as DeconGel components. When necessary, experimental methods were customized to afford complete dissolution of organic contaminants. Additionally, analytical methods and associated equipment (LC column, LC gradient program, MS sample ionization parameters) were appropriately utilized to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 and 2 show the decontamination efficacies of DeconGel 1101 on stainless steel, aluminum, and concrete surfaces contaminated with Industrial Solvents (*m*-cresol) as determined by residual swipe testing and direct DeconGel analysis.

Table 1. Decontamination efficacy of DeconGel 1101 on Cresol Industrial Solvent contaminated stainless steel, aluminum, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	524.38 ± 1.98
	Residual	1.23 ± 0.01
	Decon. Efficacy (%)	**99.8 ± 0.0**
Aluminum	Control	523.94 ± 1.16
	Residual	1.24 ± 0.003
	Decon. Efficacy (%)	**99.8 ± 0.0**
Concrete	Control	410.48 ± 2.37
	Residual	2.40 ± 0.03
	Decon. Efficacy (%)	**99.4 ± 0.0**

1375x dilution factor for samples and controls

Table 2. Decontamination efficacy of DeconGel 1101 on Cresol Industrial Solvent contaminated stainless steel, aluminum, and concrete surfaces as determined by direct DeconGel analysis.

Direct DeconGel Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	550.24 ± 1.02
	Encapsulated in Gel	530.36 ± 2.45
	Decon. Efficacy (%)	**96.4 ± 0.42**
Aluminum	Control	550.24 ± 1.02
	Encapsulated in Gel	532.55 ± 0.98
	Decon. Efficacy (%)	**96.8 ± 0.39**
Concrete	Control	550.24 ± 1.02
	Encapsulated in Gel	460.94 ± 1.76
	Decon. Efficacy (%)	**83.8 ± 0.42**

1375x dilution factor for samples and controls

METHOD DEVELOPMENT OPTIMIZATIONS/OBSERVATIONS:

- We noted that amounts of contaminant liquid greater than 100 uL were difficult to spread evenly throughout coupon surfaces, forming zones of liquid saturation which tended to overload poured DeconGel, significantly limiting the ability of DeconGel to encapsulate/emulsify surface contamination. This issue was circumvented by applying a modest amount (25 uL) of contaminant liquid throughout the surface of interest using a small brush. Application of a homogenous, thin layer of contaminant on the respective substrate facilitated an optimized interaction between contaminant and DeconGel, and provided an accurate measure of DeconGel's decontamination efficacy in a scaled-down yet real-world setting. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel. Cresol readily dissolves in DMSO (50 mL) used to prepare all analytical samples and controls.

- ASTM method E1728-03, a standardized swipe testing method used for sampling of contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. Air-dried GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes were utilized in this swipe testing method.

- Direct chemical analysis of the dried peeled DeconGel samples was also utilized to provide an improved understanding of DeconGel's decontamination efficacy.

- Standardized EPA SW-846 Sampling Method 3500C "Organic Extraction and Sample Preparation" was followed as a guideline to prepare all samples and controls. All samples, controls, and standards were prepared using the same solvent and appropriate experimental conditions to ensure accurate and LC/MS instrumental analysis.

- LC/MS instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of organic compounds. Standardized EPA SW-846 Analytical Method 8321B "Solvent-Extractable Nonvolatile Compounds by High-Performance Liquid Chromatography/Thermospray/Mass Spectrometry (HPLC/TS/MS) or Ultraviolet (UV) Detection" was followed as a guideline to prepare all samples and controls.

- To ensure accurate determination of DeconGel decontamination efficacy, a standard curve of the analyte of interest was prepared using sufficiently pure analyte; the respective standards were diluted to a known concentration (ppm) using the same solvent as used for samples and controls.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

Decontamination Efficacy (DeconGel Testing) =

(Contaminant (ppm) of DeconGel Direct/Contaminant (ppm) of DeconGel Control) x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 40 uL of contaminant was evenly applied via brushing on 1) stainless steel (commercial grade, surface area: 56.3 cm^2), 2) aluminum (commercial grade, surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe and gel samples were suspended in 50 mL DMSO for 24 h. Samples were then analyzed via LC/MS (see below).

Controls Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied via brushing on 1) stainless steel (surface area: 56.3 cm^2), 2) aluminum (surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 50 mL DMSO for 24 h and analyzed via LC/MS (see below).

For DeconGel Control samples, a respectable amount of contaminant and approximately 6.0 g of dry DeconGel 1101 (pre-poured gel on the respective uncontaminated substrate and let to dry for 24 h) was suspended in 50 mL DMSO for 24 h and analyzed via LC/MS (see below).

Reagents and Standards

Meta-Cresol (3-methylphenol) (CAS# 108-39-4, Fisher Scientific; Fair Lawn, NJ) was used as received.

Analytical Instrumentation

A Thermo LCQ LC/MS with autosampler in positive mode was used to determine nicotine concentration (ppm, wt/wt) of all samples and controls, using a Grace Davison (Deerfield, IL) C18-Select analytical column (150 x 4.6 mm, 5 um).

A 7-point standard curve derived from three independently prepared stock solutions was prepared using DMSO as the working solvent.

LC method using A= water (0.1% formic acid), B=acetonitrile (0.1% formic acid); start at 95%A to 3 min at 70%A, to 6 min at 50%A, to 12 min at 30%A, to 15 min at 90%B, to 16 min at 100%B, hold until 22 min at 100% B, to 23 min at 95%A, hold until 24 min.

Meta-Cresol LC/MS data: 9.8 min; lambda max = 261, 272, 283 nm; M^+ = 108.

6.17. Technical Report for Iron (Iron Chloride)

TITLE: Surface Decontamination of Iron Compounds (Iron (II) Chloride ($FeCl_2$)) by DeconGel 1101

AUTHOR: Andreas Mylonakis PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel 1101 on stainless steel, carbon steel, aluminum and concrete surfaces contaminated with Iron (II) Chloride ($FeCl_2$) using ICP-OES (Inductively Coupled Plasma-Optical Emission Spectroscopy) following Environmental Protection Agency (EPA) SW-846 Methods 3005A (sampling) and 6010C (analysis).

HAZARDOUS MATERIALS RELEVANCE: While iron itself is non-hazardous, iron dust can be considered as an inhalation hazard. Compounds of iron are often corrosive and harmful; one such compound is iron (II) chloride ($FeCl_2$) or ferrous chloride which is often found in the waste water treatment arena and in laboratory settings as a reducing agent. Ferrous chloride was chosen as a representative iron compound for evaluating DeconGel's efficacy; DeconGel is expected to have similar efficacy towards the wide range of iron compounds.

SUMMARY RESULTS:

- Excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with iron compounds resulting in encapsulation of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 98.8% (on concrete) to 99.5% (on carbon steel) to 99.5% (on stainless steel) as determined by residual swipe analysis, and from 86.0% (on concrete) to 97.9% (on carbon steel) to 100.0% (on stainless steel) as determined by direct DeconGel analysis.
- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of inorganics in aqueous samples. When deemed necessary, experimental methods were customized to afford complete dissolution of inorganic contaminants, and to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 and 2 show the decontamination efficacies of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with ferrous chloride as determined by residual swipe testing and direct DeconGel analysis respectively.

Table 1. Decontamination efficacies of DeconGel 1101 against ferrous chloride ($FeCl_2$) contaminated stainless steel, carbon steel,

and concrete surfaces as determined by residual swipe testing method.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	574.5 ± 19.8
	Residual	2.60 ± 0.51
	Decon. Efficacy (%)	**99.5 ± 5.1**
Carbon Steel	Control	582.6 ± 19.9
	Residual	2.66 ± 1.1
	Decon. Efficacy (%)	**99.5 ± 6.3**
Concrete	Control	536.2 ± 20.4
	Residual	6.20 ± 3.4
	Decon. Efficacy (%)	**98.8 ± 6.5**

21880x dilution factor for samples and controls

Table 2. Decontamination efficacies of DeconGel 1101 against ferrous chloride ($FeCl_2$) contaminated stainless steel, carbon steel, and concrete surfaces as determined by direct DeconGel testing method.

Direct DeconGelTesting (ppm)		Formulation		
		DeconGel 1101[a]	**DeconGel 1101[b]**	**DeconGel 1101[c]**
Stainless Steel	Control	1360 ± 10.0	NA	572.1 ± 11.2
	Encapsulated in Gel	1337 ± 49.6	NA	599.5 ± 20.2
	Decon. Efficacy (%)	**98.3 ± 3.6**	**NA**	**104.8 ± 4.1**
Aluminum	Control	1360 ± 10.0	NA	NA
	Encapsulated in Gel	1248 ± 36.3	NA	NA
	Decon. Efficacy (%)	**91.8 ± 2.7**	NA	NA
Carbon Steel	Control	NA	NA	569.2 ± 18.4
	Encapsulated in Gel	NA	NA	557.2 ± 15.0
	Decon. Efficacy (%)	NA	NA	**97.9 ± 4.1**
Concrete	Control	1360 ± 10	1257 ± 23	565.5 ± 12.3
	Encapsulated in Gel	743.1 ± 110.0	546.7 ± 159	486.3 ± 15.4
	Decon. Efficacy (%)	**54.6 ± 8.1**	**43.5 ± 12.7**	**86.0 ± 3.3**

21880x dilution factor for samples and controls

a. 100 mg was applied to the surface, prior to application of DeconGel. Dry time was 24 hours.
b. 100 mg was applied to the surface, prior to application of DeconGel. Dry time was 72 hours.
c. 50 mg was applied to the surface and left to dry overnight. DeconGel was applied after 24 hours and left to dry for 24 hours.

METHOD DEVELOPMENT OPTIMIZATIONS/OBSERVATIONS:

- We noted that amounts of contaminant solid greater than 0.05 g were difficult to spread evenly throughout coupon surfaces, forming clumps and piles which tended to overload poured DeconGel, significantly limiting the ability of DeconGel to encapsulate/emulsify surface contamination. This issue was circumvented by applying a modest amount (0.05 g) of contaminant solid throughout the surface of interest. Ferrous chloride is a hygroscopic, paramagnetic solid that was difficult to weigh which led to significantly high deviations as evidenced by obtaining a decontamination efficacy on steel of over 100% (104.8 ± 4.1) as determined by the direct DeconGel test method. Application of a homogenous, thin layer of contaminant on the respective substrate facilitated an optimized interaction between contaminant and DeconGel, and provided an accurate measure of DeconGel's decontamination efficacy in a scaled-down yet real-world setting. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel. $FeCl_2$ readily dissolves in aqueous acidic solutions used to prepare all samples and controls (15% HCl, 15% HNO_3, 70% DI H_2O).

- ASTM method E1728-03, a standardized swipe testing method used for inorganic contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes (pre-wetted with DI H_2O) were utilized in this swipe testing method.

- Direct chemical analysis of the dried peeled DeconGel samples was also utilized to provide an improved understanding of DeconGel's decontamination efficacy.

- Standardized EPA SW-846 Sampling Method 3005A "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy" was followed as a guideline to prepare all samples and controls. When deemed necessary, digestion methods were customized by increasing hydrochloric and nitric acid concentrations from 20% to 30% wt, and/or by heating samples to higher temperatures using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC) for the complete digestion/dissolution of the inorganic contaminants. All samples, controls, and standards were prepared using the same dissolution solution and experimental conditions to ensure both correct instrument calibration and accurate analytical results.

- ICP-OES instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of inorganic compounds. Standardized EPA SW-846 Analytical Method 6010C

"Inductively Coupled Plasma-Atomic Emission Spectrometry" was followed as a guideline to prepare all samples and controls.

- To ensure accurate determination of DeconGel decontamination efficacy, calibration standards of the analyte of interest were prepared using either a sufficiently pure analyte or an appropriate ICP-MS Standard (Ricca Chemical Company; Arlington, TX); the respective standards were diluted to a known concentration (ppm) using the same digestion method as the one used for samples and controls. Instrument blank controls were DI H_2O ($\geq$17 M-Ohm).

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

Decontamination Efficacy (DeconGel Testing) =

(Contaminant (ppm) of DeconGel Direct/Contaminant (ppm) of DeconGel Control) x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.05 g contaminant was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), 3) aluminum (commercial grade, surface area: 56.3 cm^2) or 4) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24-48 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe and gel samples were suspended in 100 mL aqueous acidic solution (15% HCl, 15% HNO_3, 70% deionized (DI) H_2O) for 24 h.

When deemed necessary, samples were heated to 94 ^oC for 4-14 h to effectively complete dissolution of inorganics using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC). Samples were allowed to cool to room temperature and were then analyzed via ICP-OES (see below).

Controls Methods

For Swipe Controls, 0.05 g contaminant was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 100 mL aqueous acidic solution (15% HCl, 15% HNO_3, 70% DI H_2O) for 24 h and analyzed via ICP-OES (see below).

For DeconGel Controls, 0.05 g contaminant and approximately 6.0 g dry DeconGel (pre-poured gel on the respective un-contaminated substrate and let to dry for 24-48 h) was suspended in 100 mL aqueous acidic solution (15% HCl, 15% HNO_3, 70% DI H_2O) for 24 h and analyzed via ICP-OES (see below).

Reagents and Standards

Reagent grade ferrous chloride, Iron (II) Chloride, $FeCl_2$, (CAS# 13478-10-9, Fisher Scientific; Fair Lawn, NJ) was used as received.

A 1000 ppm calibration standard was prepared using reagent grade ferrous chloride and freshly prepared aqueous acidic solution (15% HCl, 15% HNO_3, 70% DI H_2O). DI H_2O was used as the blank sample.

Analytical Instrumentation

A Thermo ICP-OES instrument model radial iCap 6300 was used to determine $FeCl_2$ concentration (ppm) of all samples and controls using a freshly prepared 1000 ppm calibration standard.

Analyte (iron) analyzed at 259.9 nm

Pump Speed: 0.5 mL/min

6.18. Technical Report for Mercury Compounds (Mercury (II) Chloride, Mercury (II) Oxide)

TITLE: Surface Decontamination of Mercury Compounds (Mercury (II) Chloride ($HgCl_2$) and Mercury Oxide (HgO)) by DeconGel 1101

AUTHOR: Andreas Mylonakis PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel formulation 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with mercury(II) chloride ($HgCl_2$) and mercury(II) oxide (HgO) using ICP-OES (Inductively Coupled Plasma-Optical Emission Spectroscopy) following Environmental Protection Agency (EPA) SW-846 Methods 3005A (sampling) and 6010C (analysis).

HAZARDOUS MATERIALS RELEVANCE: Mercury compounds (mercury (II) chloride ($HgCl_2$) and mercury (II) oxide (HgO)) are commonly used for a variety of industrial applications. Mercury oxide is often decomposed to produce elemental mercury and is used in the production of mercuric batteries. Mercury (II) chloride is a well known reagent in analytical and organic chemistry and is also used as a depolarizer in batteries. Both compounds are highly toxic if ingested or inhaled. Mercury (II) chloride and mercury (II) oxide were chosen as representative mercury compounds for evaluating DeconGel's efficacy; DeconGel is expected to have similar efficacy towards the wide range of mercury compounds.

SUMMARY RESULTS:

- Acceptable to excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with mercury compounds resulting in encapsulation of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from greater than 95.3% (on concrete) to greater than 57.7% (carbon steel) to greater than 64.6% (on stainless steel) as determined by residual swipe analysis, and from greater than 76.7% (on concrete) to greater than 57.7% (on carbon steel) and to greater than 49.0% (on stainless steel) as determined by direct DeconGel analysis.
- Due to the corrosive nature of mercury(II) chloride, DeconGel surface decontamination was not found to be exceptional on steel (carbon and stainless steel) surfaces due to mercury chloride's ability to react with these surfaces, forming a fixed residue that was not able to be completely removed

by DeconGel. Nevertheless, DeconGel showed acceptable decontamination efficacy of loose mercury chloride contamination from such surfaces.

- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of inorganic compounds in aqueous samples. When deemed necessary, experimental methods were customized to afford complete dissolution of inorganic contaminants, and to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 through 4 show the decontamination efficacies of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with mercury compounds as determined by residual swipe testing and direct DeconGel analysis.

Table 1. Decontamination efficacies of DeconGel 1101 against mercury(II) chloride ($HgCl_2$) contaminated stainless steel, carbon steel, and concrete surfaces as determined by residual swipe testing method.

Swipe Testing* (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	568.9 ± 32.6
	Residual	31.9 ± 38.2
	Decon. Efficacy (%)	**64.6 ± 13.8**
Carbon Steel	Control	584.9 ± 32.6
	Residual	97.2 ± 75.3
	Decon. Efficacy (%)	**57.7 ± 8.1**
Concrete	Control	525.0 ± 21.3
	Residual	24.6 ± 12.0
	Decon. Efficacy (%)	**95.3 ± 4.8**

21880x dilution factor for samples and controls

* Mercury chloride is corrosive and reacted with all the surfaces evaluated, resulting in a fixed residue on the contaminated surface that could not be completely removed by DeconGel.

Table 2. Decontamination efficacies of DeconGel 1101 against mercury(II) chloride ($HgCl_2$) contaminated stainless steel, carbon steel, and concrete surfaces as determined by direct DeconGel analysis.

Direct DeconGel Testing* (ppm)		Formulation	
		DeconGel 1101[1]	DeconGel 1101[1]
Stainless Steel	Control	634.2 ± 5.9	NA
	Encapsulated in Gel	310.5 ± 67.8	NA
	Decon. Efficacy (%)	**49.0 ± 10.7**	NA
Carbon Steel	Control	626.1 ± 11.9	NA
	Encapsulated in Gel	361.2 ± 50.5	NA
	Decon. Efficacy (%)	**57.7 ± 8.1**	NA
Concrete[1]	Control	529.2 ± 15.1	565.8 ± 23.1
	Encapsulated in Gel	405.8 ± 39.0	352.8 ± 93.3
	Decon. Efficacy (%)	**76.7 ± 7.7**	**62.4 ± 16.7**

21880x dilution factor for samples and controls

[1] Experiment was run in duplicate

* Mercury chloride is corrosive and reacted with all the surfaces evaluated, resulting in a fixed residue on the contaminated surface that could not be completely removed by DeconGel.

Table 3. Decontamination efficacies of DeconGel 1101 against mercury(II) oxide (HgO) contaminated stainless steel, carbon steel, and concrete surfaces as determined by residual swipe testing method.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	517.5 ± 33.4
	Residual residual	0.47 ± 0.19
	Decon. Efficacy (%)	**99.9 ± 11.5**
Carbon Steel	Control	511.4 ± 22.6
	Residual	0.67 ± 0.27
	Decon. Efficacy (%)	**99.9 ± 7.4**
Concrete	Control	490.6 ± 32.3
	Residual	1.21 ± 0.91
	Decon. Efficacy (%)	**99.8 ± 5.4**

21880x dilution factor for samples and controls

Table 4. Decontamination efficacies of DeconGel against mercury(II) oxide (HgO) contaminated stainless steel, carbon steel, and concrete surfaces as determined by direct DeconGel analysis.

Direct DeconGel Testing (ppm)		Formulation	
		DeconGel 1101[1]	**DeconGel 1101[1]**
Stainless Steel	Control	605.4 ± 9.1	481.1 ± 5.3
	Encapsulated in Gel	596.8 ± 24.6	479.1 ± 26.4
	Decon. Efficacy (%)	**98.6 ± 4.1**	**99.6 ± 5.6**
Carbon Steel	Control	592.7 ± 5.5	495.6 ± 36.8
	Encapsulated in Gel	599.0 ± 13.7	497.5 ± 16.7
	Decon. Efficacy (%)	**101.1 ± 2.3**	**100.4 ± 8.2**
Concrete	Control	569.9 ± 3.5	497.2 ± 8.3
	Encapsulated in Gel	562.7 ± 9.7	487.2 ± 17.5
	Decon. Efficacy (%)	**98.7 ± 1.7**	**98.0 ± 3.9**

21880x dilution factor for samples and controls

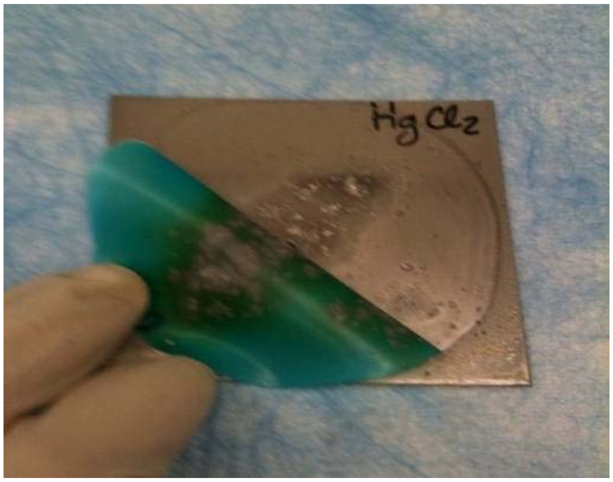
[1] Experiments were run in duplicate

METHOD DEVELOPMENT OPTIMIZATIONS/OBSERVATIONS:

- We noted that amounts of contaminant solid greater than 0.05 g were difficult to spread evenly throughout coupon surfaces, forming clumps and piles which tended to overload poured DeconGel, significantly limiting the ability of DeconGel to encapsulate/emulsify surface contamination. This issue was circumvented by applying a modest amount (0.05 g) of contaminant solid throughout the surface of interest. Application of a homogenous, thin layer of contaminant on the respective substrate facilitated an optimized interaction between contaminant and DeconGel, and provided an accurate measure of DeconGel 1101's decontamination efficacy in a scaled-down yet real-world setting. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel. Mercury(II) chloride and mercury(II) oxide readily dissolve in aqueous acidic solutions used to prepare all samples and controls (20% HCl, 15% HNO_3, 65% DI H_2O).

- Mercury chloride underwent a chemical reaction with stainless steel and carbon steel surfaces (Figure 1) which prevented some contamination from being sampled (swipe testing) and encapsulated into dry DeconGel (direct gel testing). Nevertheless DeconGel showed acceptable decontamination efficacy against loose mercury chloride contamination.

Figure 1. Mercury chloride reacted with the stainless steel surface creating a fixed surface deposit.



- Mercurous(I) Chloride (Hg_2Cl_2) was also evaluated via direct DeconGel analysis but failed to digest/dissolve in multiple digestion solutions comprised of various concentrations of nitric, hydrochloric, and sulfuric acids. Samples were also heated to up to 95ºC utilizing a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC) for varying amounts of time (1-12 hrs), yet complete Hg_2Cl_2 dissolution was not afforded and this mercury contaminant was disregarded from the present study. Nevertheless as evidenced through visual inspection, DeconGel appeared to be very efficient in decontaminating mercurous(I) chloride (Hg_2Cl_2) from a variety of surfaces (including stainless steel, aluminum and concrete). In place of Hg_2Cl_2, use of the more common chemical mercury(II) chloride was employed for DeconGel efficacy evaluations.

- ASTM method E1728-03, a standardized swipe testing method used for sampling of inorganic contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes (pre-wetted with DI H_2O) were utilized in this swipe testing method.

- Direct chemical analysis of the dried peeled DeconGel samples was also utilized to provide an improved understanding of DeconGel's decontamination efficacy.

- Standardized EPA SW-846 Sampling Method 3005A "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy" was followed as a guideline to prepare all samples and controls. When deemed necessary, digestion methods were customized by increasing hydrochloric and nitric acid concentrations from 25% to 35% wt, and/or by heating samples to higher temperatures using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC) to afford the complete digestion of the inorganic contaminants. All samples, controls, and standards were prepared using the same dissolution solution and experimental conditions to ensure both correct instrument calibration and accurate analytical results.

- ICP-OES instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of inorganic compounds. Standardized EPA SW-846 Analytical Method 6010C "Inductively Coupled Plasma-Atomic Emission Spectrometry" was followed as a guideline to prepare all samples and controls.
- To ensure accurate determination of DeconGel decontamination efficacy, calibration standards of the analyte of interest were prepared using either a sufficiently pure analyte or an appropriate ICP-MS Standard (Ricca Chemical Company; Arlington, TX); the respective standards were diluted to a known concentration (ppm) using the same digestion method as the one used for samples and controls. Instrument blank controls were DI H_2O ($\geq$17 M-Ohm).

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

Decontamination Efficacy (DeconGel Testing) =

(Contaminant (ppm) of DeconGel Direct/Contaminant (ppm) of DeconGel Control) x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.05 g contaminant was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24-48 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe and gel samples were suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% deionized (DI) H_2O) for 24 h. When deemed necessary, samples were heated to 94°C for 4-14 h to effectively complete dissolution of inorganics using a HotBlock™ Sample Heater (Environmental Express; Mt.

Pleasant, SC). Samples were allowed to cool to room temperature and were then analyzed via ICP-OES (see below).

Controls Methods

For Swipe Controls, 0.05 g of contaminant was evenly applied on 1) stainless steel (56.3 cm^2), 2) carbon steel (commercial grade, 100 cm^2), or 3) concrete (industrial grade, 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O) for 24 h and analyzed via ICP-OES.

For DeconGel Control samples, 0.05 g of contaminant and approximately 6.0 g dry DeconGel 1101 (pre-poured gel on the respective un-contaminated substrate and let to dry for 24-48 h) was suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O) for 24 h and analyzed via ICP-OES.

Reagents and Standards

Reagent grade Mercury(II) Chloride, $HgCl_2$, (CAS# 7487-94-7, Fisher Scientific; Fair Lawn, NJ) was used as received.

Reagent grade Mercury(II) Oxide, HgO, (CAS # 21508-53-2, Fisher Scientific; Fair Lawn, NJ), was used as received.

1000 ppm calibration standards were prepared using reagent grade HgO and $HgCl_2$ in freshly prepared aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O). DI H_2O was used as the blank sample.

Analytical Instrumentation

A Thermo ICP-OES instrument model radial iCap 6300 was used to determine mercury compound concentration (ppm) of all samples and controls using a freshly prepared 1000 ppm calibration standard.

Analyte (mercury) analyzed at 184.9 nm

Pump Speed: 0.5 mL/min

6.19. Technical Report for Nickel (Nickel Nitrate)

TITLE: Surface Decontamination of Nickel Compounds (Nickel Nitrate ($Ni(NO_3)_2$)) by DeconGel 1101

AUTHOR: Andreas Mylonakis PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with nickel nitrate ($Ni(NO_3)_2$) using ICP-OES (Inductively Coupled Plasma-Optical Emission Spectroscopy) following Environmental Protection Agency (EPA) SW-846 Methods 3005A (sampling) and 6010C (analysis).

HAZARDOUS MATERIALS RELEVANCE: Nickel is corrosion-resistant and is used in alloys and platings, and in the manufacturing of rechargeable batteries, magnets, and coins. Nickel and its compounds can cause contact allergy and are believed to be carcinogenic. Nickel nitrate was chosen as a representative nickel compound for evaluating DeconGel's efficacy; DeconGel is expected to have similar efficacy towards the wide range of nickel compounds.

SUMMARY RESULTS:

- Excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with nickel compounds facilitating encapsulation/emulsification of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 98.3% (on concrete) to 99.3% (on carbon steel) to 99.9% (on stainless steel) as determined by residual swipe analysis, and from 97.9% (on concrete) to 94.1% (on carbon steel) to 87.6% (on stainless steel) as determined by direct DeconGel analysis.

- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of inorganic compounds in aqueous samples. When deemed necessary, experimental methods were customized to facilitate complete dissolution of inorganic contaminants, and to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 and 2 show the decontamination efficacies of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with nickel nitrate as determined by residual swipe testing and direct DeconGel testing respectively.

Table 1. Decontamination efficacy of DeconGel 1101 on $Ni(NO_3)_2$ contaminated stainless steel, carbon steel, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		**Formulation**
		DeconGel 1101
Stainless Steel	Control	565.7 ± 7.3
	Residual	0.67 ± 0.22
	Decon. Efficacy (%)	**99.9 ± 7.3**
Carbon Steel	Control	574.2 ± 37.4
	Residual	3.99 ± 0.63
	Decon. Efficacy (%)	**99.3 ± 8.5**
Concrete	Control	533.7 ± 17.9
	Residual	9.14 ± 2.72
	Decon. Efficacy (%)	**98.3 ± 4.6**

2280x dilution factor for samples and controls

Table 2. Decontamination efficacy of DeconGel 1101 on $Ni(NO_3)_2$ contaminated stainless steel, carbon steel, and concrete surfaces as determined by direct DeconGel analysis.

Direct DeconGel Analysis (ppm)		**Formulation**
		DeconGel 1101
Stainless Steel	Control	542.4 ± 4.7
	Encapsulated in Gel	530.9 ± 11.4
	Decon. Efficacy (%)	**97.9 ± 2.3**
Carbon Steel	Control	565.3 ± 20.4
	Encapsulated in Gel	532.0 ± 18.4
	Decon. Efficacy (%)	**94.1 ± 4.7**
Concrete	Control	563.0 ± 11.2
	Encapsulated in Gel	493.4 ± 14.7
	Decon. Efficacy (%)	**87.6 ± 3.1**

2280x dilution factor for samples and controls

METHOD DEVELOPMENT OPTIMIZATIONS/OBSERVATIONS:

- We noted that amounts of contaminant solid greater than 0.05 g were difficult to spread evenly throughout coupon surfaces, forming clumps and piles which tended to overload poured DeconGel, significantly limiting the ability of DeconGel to encapsulate/emulsify surface contamination. This issue was circumvented by applying a modest amount (0.05 g) of contaminant solid throughout the surface of interest. Nickel nitrate is hygroscopic and as such is difficult to precisely weigh out. Modest amounts (0.05 g) of solid $NiNO_3$ contaminant were used for all the surfaces evaluated. Application of a homogenous, thin layer of a modest amount of contaminant on the respective substrate facilitated an optimized interaction between contaminant and DeconGel, and provided an accurate measure of DeconGel's decontamination efficacy in a scaled-down yet real-world setting. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel. $Ni(NO_3)_2$ readily dissolves in aqueous acidic solutions used to prepare all samples and controls (20% HCl, 15% HNO_3, 65% DI H_2O).

- ASTM method E1728-03, a standardized swipe testing method used for sampling of inorganic contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes (pre-wetted with DI H_2O) were utilized in this swipe testing method.

- Direct chemical analysis of the dried peeled DeconGel samples was also utilized to provide an improved understanding of DeconGel's decontamination efficacy.

- Standardized EPA SW-846 Sampling Method 3005A "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy" was followed as a guideline to prepare all samples and controls. When deemed necessary, digestion methods were customized by increasing hydrochloric and nitric acid concentrations from 20% to 30% wt, and/or by heating samples to higher temperatures using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC) for the complete digestion of the inorganic contaminants. All samples, controls, and standards were prepared using the same dissolution solution and experimental conditions to ensure both correct instrument calibration and accurate analytical results.

- ICP-OES instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of inorganic compounds. Standardized EPA SW-846 Analytical Method 6010C "Inductively Coupled Plasma-Atomic Emission Spectrometry" was followed as a guideline to prepare all samples and controls.

- To ensure accurate determination of DeconGel decontamination efficacy, calibration standards of the analyte of interest were prepared using either a sufficiently pure analyte or an appropriate ICP-MS Standard (Ricca Chemical Company; Arlington, TX); the respective standards were diluted to a known concentration (ppm) using the same digestion method as the one used for samples and controls. Instrument blank controls were DI H_2O ($\geq$17 M-Ohm).

- Nickel sulfate ($NiSO_4$) was also initially considered as a nickel compound contaminant for this study. Dissolution of gel samples containing nickel sulfate in acidic aqueous solutions formed thick, viscous precipitates that inhibited analytical analysis utilizing ICP-OES due to instrument clogging; as such, nickel sulfate was not evaluated in this study.

- Due to the pyrophoric and toxic nature of elemental nickel powder, elemental nickel was not evaluated in this study.

CALCULATION:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

Decontamination Efficacy (DeconGel Testing) =

(Contaminant (ppm) of DeconGel Direct/Contaminant (ppm) of DeconGel Control) x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.05 g $Ni(NO_3)_2$ was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24-48 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe and gel samples were suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% deionized (DI) H_2O) for 24 h. When deemed necessary, samples were heated to 94 ^oC for 4-14 hours to effectively complete digestion of inorganics using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC). Samples were allowed to cool to room temperature and were then analyzed via ICP-OES (see below).

Controls Methods

For Swipe Control samples, 0.05 g $Ni(NO_3)_2$ was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 100 mL aqueous acidic solution (15% HCl, 15% HNO_3, 70% DI H_2O) for 24 h and analyzed via ICP-OES (see below).

For DeconGel Control samples, 0.05 g $Ni(NO_3)_2$ and approximately 6.0 g of dry DeconGel 1101 (pre-poured gel on the respective un-contaminated substrate and let to dry for 24-48 h) was suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O) for 24 h and analyzed via ICP-OES (see below).

Reagents and Standards

Reagent Grade Nickel Nitrate, $Ni(NO_3)_2$, (CAS# 13478-00-7, Fisher Scientific; Fair Lawn, NJ), was used as received.

A 1000 ppm calibration standard was prepared using reagent grade powdered $Ni(NO_3)_2$ and freshly prepared aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O). DI H_2O was used as the blank sample.

Analytical Instrumentation

A Thermo ICP-OES instrument model radial iCap 6300 was used to determine $Ni(NO_3)_2$ concentration (ppm) of all samples and controls using a freshly prepared 1000 ppm calibration standard.

Analyte (nickel) analyzed at 221.6 nm;

Pump Speed: 0.5 mL/min

6.20. Technical Report for Organic Acids (Benzoic Acid)

TITLE: Surface Decontamination of Organic Acids (Benzoic Acid) by DeconGel 1101

ANALYST: Geoff Nadolski PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel formulation 1101 on stainless steel, aluminum, and concrete surfaces contaminated with Organic Acids (benzoic acid (BA)) using LC/MS (Liquid Chromatography/Mass Spectrometry) following Environmental Protection Agency (EPA) SW-846 Methods 3500C (Sampling) and 8321B (Analysis).

HAZARDOUS MATERIALS RELEVANCE: BA is an aromatic carboxylic acid used as a food preservative and as industrial chemical feedstock used to prepare flavors, insect repellents, and plasticizers. As with other organic acids, benzoic acid is acidic, an irritant, and can be corrosive to sensitive tissues including mucous membranes and the gastrointestinal tract. Benzoic acid was chosen as a representative organic acid; DeconGel is expected to have similar efficacy towards the wide range of organic acids.

SUMMARY RESULTS:

- Excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with organic acids (benzoic acid) resulting in encapsulation of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 98.4% (on concrete) to 99.7% (on stainless steel) to 99.8% (on aluminum) for BA determined by residual swipe analysis, and 88.6% (on concrete) to 98.6% (on stainless steel) to 99.0% (on aluminum) for DDT as determined by direct DeconGel analysis.

- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of organic compounds dissolved in a suitable solvent able to completely solvate BA as well as DeconGel components. When deemed necessary, experimental methods were customized to afford complete dissolution of organic contaminants. Additionally, analytical methods and associated equipment (LC column, LC gradient program, MS sample ionization parameters) were appropriately developed to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 and 2 show the decontamination efficacies of DeconGel 1101 on stainless steel, aluminum, and concrete surfaces contaminated with Organic Acids (BA) as determined by residual swipe testing and direct DeconGel analysis.

Table 1. Decontamination efficacy of DeconGel 1101 on BA Organic Acid contaminated stainless steel, aluminum, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	426.77 ± 1.85
	Residual	1.17 ± 0.06
	Decon. Efficacy (%)	**99.7 ± 0.01**
Aluminum	Control	427.01 ± 1.61
	Residual	1.07 ± 0.04
	Decon. Efficacy (%)	**99.8 ± 0.0**
Concrete	Control	350.63 ± 4.19
	Residual	5.65 ± 0.40
	Decon. Efficacy (%)	**98.4 ± 0.10**

1571x dilution factor for samples and controls

Table 2. Decontamination efficacy of DeconGel 1101 on BA Organic Acid contaminated stainless steel, aluminum, and concrete surfaces as determined by direct DeconGel analysis.

Direct DeconGel Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	441.04 ± 1.30
	Encapsulated in Gel	435.11 ± 1.55
	Decon. Efficacy (%)	**98.6 ± 0.68**
Aluminum	Control	440.83 ± 1.45
	Encapsulated in Gel	436.39 ± 0.97
	Decon. Efficacy (%)	**99.0 ± 0.53**
Concrete	Control	441.41 ± 1.55
	Encapsulated in Gel	391.13 ± 1.43
	Decon. Efficacy (%)	**88.6 ± 0.41**

1571x dilution factor for samples and controls

METHOD DEVELOPMENT OPTIMIZATIONS/OBSERVATIONS:

- We noted that amounts of contaminant solid greater than 0.05 g were difficult to spread evenly throughout coupon surfaces, forming clumps and piles which tended to overload poured DeconGel, significantly limiting the ability of DeconGel to encapsulate/emulsify surface contamination. This issue was circumvented by applying a modest amount (0.035 g) of contaminant solid throughout the surface of interest. Application of a homogenous, thin layer of contaminant on the respective substrate facilitated an optimized interaction between contaminant and DeconGel, and provided an accurate measure of DeconGel's decontamination efficacy in a scaled-down yet real-world setting. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel. BA readily dissolves in DMSO (50 mL) used to prepare all analytical samples and controls.

- ASTM method E1728-03, a standardized swipe testing method used for sampling of contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. Air-dried GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes (pre-wetted with DMSO immediately prior to performing swipe analysis) were utilized in this swipe testing method.

- Direct chemical analysis of the dried peeled DeconGel samples was also utilized to provide an improved understanding of DeconGel's decontamination efficacy.

- Standardized EPA SW-846 Sampling Method 3500C "Organic Extraction and Sample Preparation" was followed as a guideline to prepare all samples and controls. All samples, controls, and standards were prepared using the same solvent and appropriate experimental conditions to ensure accurate and LC/MS instrumental analysis.

- LC/MS instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of organic compounds. Standardized EPA SW-846 Analytical Method 8321B "Solvent-Extractable Nonvolatile Compounds by High-Performance Liquid Chromatography/Thermospray/Mass Spectrometry (HPLC/TS/MS) or Ultraviolet (UV) Detection" was followed as a guideline to prepare all samples and controls.

- To ensure accurate determination of DeconGel decontamination efficacy, a standard curve of the analyte of interest was prepared using sufficiently pure analyte; the respective standards were diluted to a known concentration (ppm) using the same solvent as used for samples and controls.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

Decontamination Efficacy (DeconGel Testing) =

(Contaminant (ppm) of DeconGel Direct/Contaminant (ppm) of DeconGel Control) x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.035 g of contaminant was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) aluminum (commercial grade, surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC) wetted with DMSO (2 mL) immediately prior to swipe analysis. Swipe and gel samples were suspended in 50 mL DMSO for 24 h. Samples were then analyzed via LC/MS (see below).

Controls Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) aluminum (commercial grade, surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC) wetted with DMSO (2 mL) immediately prior to swipe analysis. Swipe samples were suspended in 50 mL DMSO for 24 h and analyzed via LC/MS (see below).

For DeconGel Control samples, a respectable amount of contaminant and approximately 6.0 g of dry DeconGel 1101 (pre-poured gel on the respective uncontaminated substrate and let to dry for 24 h) was suspended in 50 mL DMSO for 24 h and analyzed via LC/MS (see below).

Reagents and Standards

Benzoic acid (BA) (CAS# 65-85-0, Fisher Scientific; Fair Lawn, NJ) was used as received.

Analytical Instrumentation

A Thermo LCQ LC/MS with autosampler in positive mode was used to determine BA concentration (ppm, wt/wt) of all samples and controls, using a Grace Davison (Deerfield, IL) C18-Select analytical column (150 x 4.6 mm, 5 um).

A 8-point standard curve derived from three independently prepared stock solutions was prepared using DMSO as the working solvent.

LC method using A= water (0.1% formic acid), B=acetonitrile (0.1% formic acid); start at 95%A to 3 min at 70%A, to 6 min at 50%A, to 12 min at 30%A, to 15 min at 90%B, to 16 min at 100%B, hold until 22 min at 100% B, to 23 min at 95%A, hold until 24 min.

BA LC/MS data: 9.7 min; lambda max = 245, 272 nm; $M^+ = 122$.

6.21. Technical Report for Organic Bases (Triethylamine)

TITLE: Surface Decontamination of Organic Bases (Triethylamine) by DeconGel 1101

ANALYST: Geoff Nadolski PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel formulation 1101 on stainless steel, aluminum, and concrete surfaces contaminated with Organic Bases (Triethylamine (TEA)) using GC/MS (Gas Chromatography/Mass Spectrometry) following Environmental Protection Agency (EPA) SW-846 Methods 3500C (Sampling) and 8270C (Analysis).

HAZARDOUS MATERIALS RELEVANCE: Tertiary amine bases such as TEA are used in the chemical industry as acid scavengers and are used to prepare quaternary ammonium compounds for the textile/dye industries. TEA is volatile and irritating to mucous membranes and skin and possesses an offensive fishy odor. TEA was chosen as a representative organic base compound; DeconGel is expected to have similar efficacy towards the full range of organic bases (amines).

SUMMARY RESULTS:

- Excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with organic bases (TEA) resulting in encapsulation of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 99.7% (on concrete) to 99.9% (on stainless steel) to 99.9% (on aluminum) for TEA as determined by residual swipe analysis and 75.7% (on concrete) to 93.8% (on aluminum) to 93.9% (on aluminum) for TEA as determined by analysis of DeconGel.
- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of organic compounds dissolved in a suitable solvent able to completely solvate triethylamine as well as DeconGel components. When deemed necessary, experimental methods were customized to afford complete dissolution of organic contaminants. Additionally, analytical methods and associated equipment (GC column, GC temperature gradient program, MS sample ionization parameters) were appropriately utilized to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 and 2 show the decontamination efficacies of DeconGel 1101 on stainless steel, aluminum, and concrete surfaces contaminated with

Organic Bases (TEA) as determined by residual swipe testing and direct DeconGel analysis.

Table 1. Decontamination efficacy of DeconGel 1101 on Triethylamine Organic Base contaminated stainless steel, aluminum, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	295.48 ± 1.07
	Residual	0.30 ± 0.01
	Decon. Efficacy (%)	**99.9 ± 0.01**
Aluminum	Control	295.47 ± 0.80
	Residual	0.28 ± 0.004
	Decon. Efficacy (%)	**99.9 ± 0.01**
Concrete	Control	186.42 ± 0.84
	Residual	0.65 ± 0.01
	Decon. Efficacy (%)	**99.7 ± 0.07**

608x dilution factor for samples and controls

Table 2. Decontamination efficacy of DeconGel 1101 on Triethylamine Organic Base on stainless steel, aluminum, and concrete surfaces as determined by direct DeconGel analysis.

Direct DeconGel Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	445.48 ± 0.36
	Encapsulated in Gel	418.17 ± 0.36
	Decon. Efficacy (%)	**93.9 ± 0.21**
Aluminum	Control	445.48 ± 0.36
	Encapsulated in Gel	417.88 ± 0.73
	Decon. Efficacy (%)	**93.8 ± 0.28**
Concrete	Control	445.48 ± 0.36
	Encapsulated in Gel	337.12 ± 0.28
	Decon. Efficacy (%)	**75.7 ± 0.16**

608x dilution factor for samples and controls

METHOD DEVELOPMENT OPTIMIZATIONS/OBSERVATIONS:

- We noted that amounts of contaminant liquid greater than 100 uL were difficult to spread evenly throughout coupon surfaces, forming zones of liquid saturation which tended to overload poured DeconGel, significantly limiting the ability of DeconGel to encapsulate/emulsify surface contamination. This issue was circumvented by applying a modest amount (90 uL, 50% glutaraldehyde in water) of contaminant liquid throughout the surface of interest using a small brush. Application of a homogenous, thin layer of contaminant on the respective substrate facilitated an optimized interaction between contaminant and DeconGel, and provided an accurate measure of DeconGel's decontamination efficacy in a scaled-down yet real-world setting. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel. TEA readily dissolves in 60% methanol in water (50 mL) used to prepare all analytical samples and controls.

- Acceptable direct DeconGel decontamination efficacies ranging from 75.7 to 93.9% were achieved, however, because TEA is prone to evaporate over the required 24 h drying time needed by DeconGel once applied to a contaminated surface, this could contribute to lower than expected DeconGel decontamination efficacies determined by the direct DeconGel testing method.

- ASTM method E1728-03, a standardized swipe testing method used for sampling of contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. Air-dried GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes were utilized in this swipe testing method.

- Direct chemical analysis of the dried peeled DeconGel samples was also utilized to provide an improved understanding of DeconGel's decontamination efficacy.

- Standardized EPA SW-846 Sampling Method 3500C "Organic Extraction and Sample Preparation" was followed as a guideline to prepare all samples and controls. All samples, controls, and standards were prepared using the same solvent and appropriate experimental conditions to ensure accurate and GC/MS instrumental analysis.

- GC/MS instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of organic compounds. Standardized EPA SW-846 Analytical Method 8270C "Semivolatile Organic Compounds by Gas Chromatography/ Mass Spectrometry (GC/MS)" was followed as a guideline to prepare all samples and controls.

- To ensure accurate determination of DeconGel decontamination efficacy, a standard curve of the analyte of interest was prepared using sufficiently pure analyte; the respective standards were diluted to a known concentration (ppm) using the same solvent as used for samples and controls.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

Decontamination Efficacy (DeconGel Testing) =

(Contaminant (ppm) of DeconGel Direct/Contaminant (ppm) of DeconGel Control) x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 90 uL of contaminant was evenly applied via brushing on 1) aluminum (commercial grade, surface area: 56.3 cm^2), 2) stainless steel (commercial grade, surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe and gel samples were suspended in 50 mL 60% methanol in water for 24 h. Samples were then analyzed via GC/MS (see below).

Controls Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied via brushing on 1) aluminum (surface area: 56.3 cm^2), 2) stainless steel (surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 50 mL 60% methanol in water for 24 h and analyzed via GC/MS (see below).

For DeconGel Control samples, a respectable amount of contaminant and approximately 6.0 g of dry DeconGel 1101 (pre-poured gel on the respective uncontaminated substrate and let to dry for 24 h) was suspended in 50 mL 60% methanol in water for 24 h and analyzed via GC/MS (see below).

Reagents and Standards

Triethylamine (TEA) (CAS# 121-44-8, Fisher Scientific; Fair Lawn, NJ) was used as received.

Analytical Instrumentation

A Thermo DSQII GC/MS with autosampler in positive mode was used to determine glutaraldehyde concentration (ppm, wt/wt) of all samples and controls, using a Restek (Bellefonte, PA) Rtx-5 capillary column (30 m x 0.25 mm, 0.5 um).

A 7-point standard curve derived from three independently prepared stock solutions was prepared using DMSO as the working solvent.

GC method: start at 45 ºC, hold for 7 min, ramp at 30 ºC/min to 320 ºC, hold 10 min.

Triethylamine GC/MS data: 3.5 min; M^+ = 101.

6.22. Technical Report for Pesticides (DDT)

TITLE: Surface Decontamination of Pesticides (DDT) by DeconGel 1101

ANALYST: Geoff Nadolski PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel formulation 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with Pesticides (dichlorodiphenyltrichloroethane (DDT)) using LC/MS (Liquid Chromatography/Mass Spectrometry) following Environmental Protection Agency (EPA) SW-846 Methods 3500C (Sampling) and 8321B (Analysis).

HAZARDOUS MATERIALS RELEVANCE: Pesticides can be dangerous to consumers and workers during manufacture, transport, or during and after use. DDT is a chlorinated aromatic pesticide that is a restricted persistent organic pollutant. DDT is a reproductive toxicant to birds, possesses endocrine disrupting activity in animals, and is considered moderately hazardous to humans by the World Health Organization (WHO). DDT was chosen as a representative pesticide; DeconGel is expected to have similar efficacy towards the wide range of pesticides.

SUMMARY RESULTS:

- Excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with pesticides (DDT) resulting in encapsulation of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 96.1% (on concrete) to 98.7% (on carbon steel) to 98.7% (on stainless steel) for DDT determined by residual swipe analysis, and 88.9% (on concrete) to 97.6% (on stainless steel) to 97.8% (on carbon steel) for DDT as determined by direct DeconGel analysis.
- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of organic compounds dissolved in a suitable solvent able to completely solvate DDT as well as DeconGel components. When necessary, experimental methods were customized to afford complete dissolution of organic contaminants. Additionally, analytical methods and associated equipment (LC column, LC gradient program, MS sample ionization parameters) were appropriately utilized to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 and 2 show the decontamination efficacies of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with Pesticides (DDT) as determined by residual swipe testing and direct DeconGel analysis.

Table 1. Decontamination efficacy of DeconGel 1101 on DDT Pesticide contaminated stainless steel, carbon steel, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	188.49 ± 0.94
	Residual	2.48 ± 0.04
	Decon. Efficacy (%)	**98.7 ± 0.0**
Carbon Steel	Control	190.20 ± 1.73
	Residual	2.51 ± 0.04
	Decon. Efficacy (%)	**98.7 ± 0.0**
Concrete	Control	168.32 ± 0.15
	Residual	6.61 ± 0.15
	Decon. Efficacy (%)	**96.1 ± 0.13**

2200x dilution factor for samples and controls

Table 2. Decontamination efficacy of DeconGel 1101 on DDT Pesticide contaminated stainless steel, carbon steel and concrete surfaces as determined by direct DeconGel analysis.

Direct DeconGel Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	198.55 ± 0.35
	Encapsulated in Gel	193.83 ± 0.23
	Decon. Efficacy (%)	**97.6 ± 0.27**
Carbon Steel	Control	198.15 ± 0.64
	Encapsulated in Gel	193.91 ± 0.22
	Decon. Efficacy (%)	**97.8 ± 0.42**
Concrete	Control	197.83 ± 0.41
	Encapsulated in Gel	175.94 ± 0.95
	Decon. Efficacy (%)	**88.9 ± 0.57**

2200x dilution factor for samples and controls

METHOD DEVELOPMENT OPTIMIZATIONS/OBSERVATIONS:

- We noted that amounts of contaminant solid greater than 0.05 g were difficult to spread evenly throughout coupon surfaces, forming clumps and piles which tended to overload poured DeconGel, significantly limiting the ability of DeconGel to encapsulate/emulsify surface contamination. This issue was circumvented by applying a modest amount (0.025 g) of contaminant solid throughout the surface of interest. Application of a homogenous, thin layer of contaminant on the respective substrate facilitated an optimized interaction between contaminant and DeconGel, and provided an accurate measure of DeconGel's decontamination efficacy in a scaled-down yet real-world setting. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel. DDT readily dissolves in DMSO (50 mL) used to prepare all analytical samples and controls.

- ASTM method E1728-03, a standardized swipe testing method used for sampling of contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. Air-dried GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes (pre-wetted with DMSO immediately prior to performing swipe analysis) were utilized in this swipe testing method.

- Direct chemical analysis of the dried peeled DeconGel samples was also utilized to provide an improved understanding of DeconGel's decontamination efficacy.

- Standardized EPA SW-846 Sampling Method 3500C "Organic Extraction and Sample Preparation" was followed as a guideline to prepare all samples and controls. All samples, controls, and standards were prepared using the same solvent and appropriate experimental conditions to ensure accurate and LC/MS instrumental analysis.

- LC/MS instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of organic compounds. Standardized EPA SW-846 Analytical Method 8321B "Solvent-Extractable Nonvolatile Compounds by High-Performance Liquid Chromatography/Thermospray/Mass Spectrometry (HPLC/TS/MS) or Ultraviolet (UV) Detection" was followed as a guideline to prepare all samples and controls.

- To ensure accurate determination of DeconGel decontamination efficacy, a standard curve of the analyte of interest was prepared using sufficiently pure analyte; the respective standards were diluted to a known concentration (ppm) using the same solvent as used for samples and controls.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

Decontamination Efficacy (DeconGel Testing) =

(Contaminant (ppm) of DeconGel Direct/Contaminant (ppm) of DeconGel Control) x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.025 g of contaminant was evenly applied on 1) stainless steel (commercial grade, surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC) wetted with DMSO (2 mL) immediately prior to swipe analysis. Swipe and gel samples were suspended in 50 mL DMSO for 24 h. Samples were then analyzed via LC/MS (see below).

Controls Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC) wetted with DMSO (2 mL) immediately prior to swipe analysis. Swipe samples were suspended in 50 mL DMSO for 24 h and analyzed via LC/MS (see below).

For DeconGel Control samples, a respectable amount of contaminant and approximately 6.0 g of dry DeconGel 1101 (pre-poured gel on the respective uncontaminated substrate and let to dry for 24 h) was suspended in 50 mL DMSO for 24 h and analyzed via LC/MS (see below).

Reagents and Standards

DDT (1,1,1-trichloro-2,2-di(4-chlorophenyl)ethane) (CAS# 50-29-3, Fisher Scientific; Fair Lawn, NJ) was used as received.

Analytical Instrumentation

A Thermo LCQ LC/MS with autosampler in positive mode was used to determine DDT concentration (ppm, wt/wt) of all samples and controls, using a Grace Davison (Deerfield, IL) C18-Select analytical column (150 x 4.6 mm, 5 um).

A 6-point standard curve derived from three independently prepared stock solutions was prepared using DMSO as the working solvent.

LC method using A= water (0.1% formic acid), B=acetonitrile (0.1% formic acid); start at 90%A to 2 min at 70%A, to 3 min at 50%A, to 5 min at 30%A, to 6 min at 100%B, hold until 16 min at 90% A hold until 17 min.

DDT LC/MS data: 9.9 min; lambda max = 244, 266 nm; M+ = 354.

6.23. Technical Report for Phenolic Compounds (Catechol)

TITLE: Surface Decontamination of Phenolic Compounds (Catechol) by DeconGel 1101

ANALYST: Geoff Nadolski PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel formulation 1101 on aluminum, carbon steel, and concrete surfaces contaminated with Phenolic Compounds (Catechol) using LC/MS (Liquid Chromatography/Mass Spectrometry) following Environmental Protection Agency (EPA) SW-846 Methods 3500C (Sampling) and 8321B (Analysis).

HAZARDOUS MATERIALS RELEVANCE: Catechol is an aromatic phenolic compound used as a precursor to produce pesticides, perfumes, and pharmaceuticals. Catechol shares similar chemical reactivity with phenol – an acidic aromatic irritant and corrosive capable of producing tissue-damaging phenoxyl radicals. Catechol was chosen as a representative phenolic compound for evaluating DeconGel's efficacy; DeconGel is expected to have similar efficacy towards the wide range of phenolic compounds.

SUMMARY RESULTS:

- Excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with phenolic compounds (catechol) resulting in encapsulation of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 97.3% (on concrete) to 99.2% (on carbon steel) to 99.2% (on aluminum) for Catechol determined by residual swipe analysis, and 90.4% (on concrete) to 96.1% (on carbon steel) to 96.7% (on aluminum) for Catechol as determined by direct DeconGel analysis.

- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of organic compounds dissolved in a suitable solvent able to completely solvate catechol as well as DeconGel components. When necessary, experimental methods were customized to afford complete dissolution of organic contaminants. Additionally, analytical methods and associated equipment (LC column, LC gradient program, MS sample ionization parameters) were appropriately utilized to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 and 2 show the decontamination efficacies of DeconGel 1101 on aluminum, carbon steel, and concrete surfaces contaminated with Phenolic Compounds (catechol) as determined by residual swipe testing and direct DeconGel analysis.

Table 1. Decontamination efficacy of DeconGel 1101 on Catechol Phenolic Compound contaminated aluminum, carbon steel, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Aluminum	Control	481.68 ± 1.51
	Residual	4.02 ± 0.40
	Decon. Efficacy (%)	**99.2 ± 0.01**
Carbon Steel	Control	480.42 ± 2.68
	Residual	3.82 ± 0.34
	Decon. Efficacy (%)	**99.2 ± 0.12**
Concrete	Control	433.26 ± 4.02
	Residual	11.90 ± 0.20
	Decon. Efficacy (%)	**97.3 ± 0.21**

1571x dilution factor for samples and controls

Table 2. Decontamination efficacy of DeconGel 1101 on Catechol Phenolic Compound contaminated aluminum, carbon steel, and concrete surfaces as determined by direct DeconGel analysis.

Direct DeconGel Testing (ppm)		Formulation
		DeconGel 1101
Aluminum	Control	484.11 ± 2.25
	Encapsulated in Gel	467.94 ± 1.55
	Decon. Efficacy (%)	**96.7 ± 0.36**
Carbon Steel	Control	485.89 ± 1.71
	Encapsulated in Gel	465.48 ± 2.69
	Decon. Efficacy (%)	**96.1 ± 0.01**
Concrete	Control	485.73 ± 3.56
	Encapsulated in Gel	439.23 ± 1.75
	Decon. Efficacy (%)	**90.4 ± 0.58**

1571x dilution factor for samples and controls

METHOD DEVELOPMENT OPTIMIZATIONS/OBSERVATIONS:

- We noted that amounts of contaminant solid greater than 0.05 g were difficult to spread evenly throughout coupon surfaces, forming clumps and piles which tended to overload poured DeconGel, significantly limiting the ability of DeconGel to encapsulate/emulsify surface contamination. This issue was circumvented by applying a modest amount (0.035 g) of contaminant solid throughout the surface of interest. Application of a homogenous, thin layer of contaminant on the respective substrate facilitated an optimized interaction between contaminant and DeconGel, and provided an accurate measure of DeconGel's decontamination efficacy in a scaled-down yet real-world setting. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel. Catechol readily dissolves in DMSO (50 mL) used to prepare all analytical samples and controls.

- Acceptable direct DeconGel decontamination efficacies ranging from 90.4 to 96.7% were achieved, however, catechol is prone to oxidize at ambient temperature over the required 24 h drying time needed by DeconGel once applied to a contaminated surface, this could contribute to lower than expected DeconGel decontamination efficacies determined by the direct DeconGel testing method.

- ASTM method E1728-03, a standardized swipe testing method used for sampling of contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. Air-dried GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes (pre-wetted with DMSO immediately prior to performing swipe analysis) were utilized in this swipe testing method.

- Direct chemical analysis of the dried peeled DeconGel samples was also utilized to provide an improved understanding of DeconGel's decontamination efficacy.

- Standardized EPA SW-846 Sampling Method 3500C "Organic Extraction and Sample Preparation" was followed as a guideline to prepare all samples and controls. All samples, controls, and standards were prepared using the same solvent and appropriate experimental conditions to ensure accurate and LC/MS instrumental analysis.

- LC/MS instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of organic compounds. Standardized EPA SW-846 Analytical Method 8321B "Solvent-Extractable Nonvolatile Compounds by High-Performance Liquid Chromatography/Thermospray/Mass Spectrometry (HPLC/TS/MS) or Ultraviolet (UV) Detection" was followed as a guideline to prepare all samples and controls.

- To ensure accurate determination of DeconGel decontamination efficacy, a standard curve of the analyte of interest was prepared using sufficiently pure analyte; the respective standards were diluted to a known concentration (ppm) using the same solvent as used for samples and controls.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

Decontamination Efficacy (DeconGel Testing) =

(Contaminant (ppm) of DeconGel Direct/Contaminant (ppm) of DeconGel Control) x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.035 g of contaminant was evenly applied on 1) aluminum (commercial grade, surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC) wetted with DMSO (2 mL) immediately prior to swipe analysis. Swipe and gel samples were suspended in 50 mL DMSO for 24 h. Samples were then analyzed via LC/MS (see below).

Controls Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied on 1) stainless steel (commercial grade, surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC) wetted with DMSO (2 mL) immediately prior to swipe analysis. Swipe samples were suspended in 50 mL DMSO for 24 h and analyzed via LC/MS (see below).

For DeconGel Control samples, a respectable amount of contaminant and approximately 6.0 g of dry DeconGel 1101 (pre-poured gel on the respective uncontaminated substrate and let to dry for 24 h) was suspended in 50 mL DMSO for 24 h and analyzed via LC/MS (see below).

Reagents and Standards

Catechol (benzene-1,2-diol) (CAS# 120-80-9, Fisher Scientific; Fair Lawn, NJ) was used as received.

Analytical Instrumentation

A Thermo LCQ LC/MS with autosampler in positive mode was used to determine catechol concentration (ppm, wt/wt) of all samples and controls, using a Grace Davison (Deerfield, IL) C18-Select analytical column (150 x 4.6 mm, 5 um).

A 7-point standard curve derived from three independently prepared stock solutions was prepared using DMSO as the working solvent.

LC method using A= water (0.1% formic acid), B=acetonitrile (0.1% formic acid); start at 95%A to 3 min at 70%A, to 6 min at 50%A, to 12 min at 30%A, to 15 min at 90%B, to 16 min at 100%B, hold until 22 min at 100% B, to 23 min at 95%A, hold until 24 min.

Catechol LC/MS data: 6.9 min; lambda max = 235, 262 nm; M^+ = 110.

6.24. Technical Report for Selenium Compounds (Selenium powder)

TITLE: Surface Decontamination of Selenium Compounds (Selenium powder) by DeconGel 1101

AUTHOR: Andreas Mylonakis PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with Selenium powder using ICP-OES (Inductively Coupled Plasma-Optical Emission Spectroscopy) following Environmental Protection Agency (EPA) SW-846 Methods 3005A (sampling) and 6010C (analysis).

HAZARDOUS MATERIALS RELEVANCE: Selenium is used in industrial applications including steel alloying and rubber compounding. It is also used to produce printers and copier drums. Selenium is hazardous by contact, ingestion, and inhalation as defined by OSHA.[6] Selenium powder was chosen as a representative selenium compound for evaluating DeconGel's efficacy; DeconGel is expected to have similar efficacy towards the wide range of selenium compounds.

SUMMARY RESULTS:

- Excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with selenium resulting in encapsulation of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 96.2% (on concrete) to 99.8% (on carbon steel) to 99.2% (on stainless steel) as determined by swipe analysis, and from 88.2% (on concrete) to 94.4% (on carbon steel) to 100.5% (on stainless steel) as determined by direct DeconGel analysis.
- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of inorganic compounds in aqueous samples. When necessary, experimental methods were customized to afford complete dissolution of inorganic contaminants, and to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 and 2 show the decontamination efficacies of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with

---

[6] Occupational Safety & Health Administration (OSHA); http://www.osha.gov/ (2010)

selenium as determined by residual swipe testing and direct DeconGel analysis respectively.

Table 1. Decontamination efficacy of DeconGel 1101 on selenium powder contaminated stainless steel, carbon steel and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		**Formulation**
		DeconGel 1101
Stainless Steel	Control	447.8 ± 6.8
	Residual	3.64 ± 4.72
	Decon. Efficacy (%)	**99.2 ± 2.8**
Carbon Steel	Control	455.8 ± 32.1
	Residual	0.88 ± 0.85
	Decon. Efficacy (%)	**99.8 ± 7.1**
Concrete	Control	412.3 ± 20.9
	Residual	15.9 ± 6.2
	Decon. Efficacy (%)	**96.2 ± 7.4**

21880x dilution factor for samples and controls

Table 2. Decontamination efficacy of DeconGel 1101 on selenium powder contaminated stainless steel, carbon steel and concrete surfaces as determined by direct DeconGel testing.

Direct DeconGel Testing (ppm)		**Formulation**
		DeconGel 1101
Stainless Steel	Control	456.9 ± 18.9
	Encapsulated in Gel	459.3 ± 10.5
	Decon. Efficacy (%)	**100.5 ± 4.7**
Carbon Steel	Control	459.2 ± 10.6
	Encapsulated in Gel	433.6 ± 10.1
	Decon. Efficacy (%)	**94.4 ± 3.1**
Concrete	Control	424.1 ± 4.0
	Encapsulated in Gel	374.0 ± 25.6
	Decon. Efficacy (%)	**88.2 ± 6.1**

21880x dilution factor for samples and controls

METHOD DEVELOPMENT OPTIMIZATIONS/OBSERVATIONS:

- We noted that amounts of contaminant solid greater than 0.05 g were difficult to spread evenly throughout coupon surfaces, forming clumps and piles which tended to overload poured DeconGel, significantly limiting the ability of DeconGel to encapsulate/emulsify surface contamination. This issue was circumvented by applying a modest amount (0.05 g) of contaminant solid throughout the surface of interest. Application of a homogenous, thin layer of contaminant (0.05g) on the substrate of interest facilitated an optimized interaction between contaminant and DeconGel, and provided an accurate measure of DeconGel's decontamination efficacy in a scaled-down yet real-world setting. No less than 6.0 g of DeconGel 1101 was used for each experiment to allow an optimized interaction between contaminant and DeconGel. Selenium readily dissolves in aqueous acidic solutions used to prepare all samples and controls (20% HCl, 15% HNO_3, 65% DI H_2O).

- ASTM method E1728-03 (a standardized swipe testing method used for sampling of inorganic contaminants) was the integral method used to accurately evaluate DeconGel's decontamination efficacy. GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes (pre-wetted with DI H_2O) were utilized in this swipe testing method.

- Direct chemical analysis of the dried peeled DeconGel samples was also utilized to provide an improved understanding of DeconGel's decontamination efficacy.

- Standardized EPA SW-846 Sampling Method 3005A "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy" was followed as a guideline to prepare all samples and controls. When necessary, digestion methods were customized by increasing hydrochloric and nitric acid concentrations from 25% to 35% wt, and/or by heating samples to higher temperatures using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC) to result in complete digestion of the inorganic contaminants. All samples, controls, and standards were prepared using the same dissolution solution and experimental conditions to ensure both correct instrument calibration and accurate analytical results.

- ICP-OES instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of inorganic compounds. Standardized EPA SW-846 Analytical Method 6010C "Inductively Coupled Plasma-Atomic Emission Spectrometry" was followed as a guideline to prepare all samples and controls.

- To ensure accurate determination of DeconGel decontamination efficacy, calibration standards of the analyte of interest were prepared using either a sufficiently pure analyte or an appropriate ICP-MS Standard (Ricca Chemical Company; Arlington, TX); the respective standards were diluted to a known concentration (ppm) using the same digestion method as the one used for samples and controls. Instrument blank controls were DI H_2O ($\geq$17 M-Ohm).

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(contaminant (ppm) of Swipe Control) – (contaminant (ppm) of Residual Swipe)/contaminant (ppm) of Swipe Control] x 100%

Decontamination Efficacy (DeconGel Testing) =

(Contaminant (ppm) of DeconGel Direct/contaminant (ppm) of DeconGel Control) x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.05 g of contaminant was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), and 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24-48 hours. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe and gel samples were suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% deionized (DI) H_2O) for 24 hours. When deemed necessary, samples were heated to 94°C for 4-14 hours to facilitate complete dissolution of inorganics using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC). Samples were allowed to cool to room temperature and were then analyzed via ICP-OES (see below).

Controls Methods

For Swipe Control samples, 0.05 g of contaminant was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), and 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O) for 24 hours and analyzed via ICP-OES (see below).

For DeconGel Control samples, 0.05 g Se and approximately 6.0 g of dry DeconGel 1101 (pre-poured gel on the respective un-contaminated substrate and let to dry for 24-48 hours) was suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O) for 24 hours and analyzed via ICP-OES (see below).

Reagents and Standards

Selenium, Se metal powder, (CAS# 7782-49-2, 200 mesh, Fisher Scientific; Fair Lawn, NJ), was used as received.

A 10.0 ppm calibration standard was prepared using reagent grade powdered Se and freshly prepared aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O). DI H_2O was used as the blank sample.

Analytical Instrumentation

A Thermo ICP-OES instrument model radial iCap 6300 was used to determine Se concentration (ppm) of all samples and controls using a freshly prepared 10.0 ppm calibration standard.

Analyte (selenium) analyzed at 196.0 nm

Pump Speed: 0.5 mL/min

6.25. Technical Report for Zinc Compounds (Zinc powder, Zinc Oxide, Zinc Acetate)

TITLE: Surface Decontamination of Zinc Compounds (Elemental Zinc Powder (Zn), Zinc Oxide (ZnO), and Zinc Acetate ($Zn(O_2CCH_3)_2$) by DeconGel 1101

AUTHOR: Andreas Mylonakis PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with zinc compounds (elemental zinc powder (Zn), zinc oxide (ZnO), and zinc acetate ($Zn(O_2CCH_3)_2$)) using ICP-OES (Inductively Coupled Plasma-Optical Emission Spectroscopy) following Environmental Protection Agency (EPA) SW-846 Methods 3005A (sampling) and 6010C (analysis).

HAZARDOUS MATERIALS RELEVANCE: Zinc compounds are common industrial compounds and are used as anti-corrosive agents and in the semi conductors and batteries Industries. Zinc compounds are irritants if inhaled and may cause flu-like symptoms known as "metal fume fever". Zinc powder, zinc oxide, and zinc acetate were chosen as representative zinc compound for evaluating DeconGel's efficacy; DeconGel is expected to have similar efficacy towards the wide range of zinc compounds.

SUMMARY RESULTS:

- Acceptable to excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with zinc compounds resulting in encapsulation of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from greater than 86.5% (on concrete) to greater than 66.9% (on carbon steel) to greater than 77.3% (on stainless steel) as determined by residual swipe analysis, and greater than 87.7% (on concrete) to greater than 78.3% (on carbon steel) to greater than 86.9% (on stainless steel) as determined by direct DeconGel analysis.
- Due to the corrosive nature of zinc powder, DeconGel surface decontamination was not found to be exceptional on steel (carbon and stainless steel) surfaces due to zinc powder's ability to react with these surfaces, forming a fixed residue that was not able to be completely removed by DeconGel. Nevertheless, DeconGel showed acceptable decontamination efficacy of loose zinc powder contamination from such surfaces.

- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of inorganic compounds in aqueous samples. When deemed necessary, experimental methods were customized to afford complete dissolution of inorganic contaminants, and to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 through 6 show the decontamination efficacies of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with elemental zinc powder (Zn), zinc oxide (ZnO), and zinc acetate ($Zn(O_2CCH_3)_2$) as determined by residual swipe testing and direct DeconGel analysis.

Table 1. Decontamination efficacy of DeconGel 1101 on zinc powder (Zn) contaminated stainless steel, carbon steel, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel*	Control	330.6 ± 14.4
	Residual	74.9 ± 17.7
	Decon. Efficacy (%)	**77.3 ± 6.4**
Carbon Steel*	Control	327.2 ± 7.0
	Residual	108.4 ± 8.0
	Decon. Efficacy (%)	**66.9 ± 2.9**
Concrete	Control	292.1 ± 16.1
	Residual	39.3 ± 13.8
	Decon. Efficacy (%)	**86.5 ± 7.7**

21880x dilution factor for samples and controls

* Zinc powder is corrosive and reacted with all the surfaces evaluated, resulting in a fixed residue on the contaminated surface that could not be completely removed by DeconGel.

Table 2. Decontamination efficacy of DeconGel 1101 on zinc powder (Zn) contaminated stainless steel, carbon steel, and concrete surfaces as determined by direct DeconGel analysis.

Direct DeconGel Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel*	Control	330.1 ± 9.2
	Encapsulated in Gel	286.8 ± 5.3
	Decon. Efficacy (%)	**86.9 ± 2.9**
Carbon Steel*	Control	317.0 ± 13.1
	Encapsulated in Gel	248.1 ± 14.8
	Decon. Efficacy (%)	**78.3 ± 5.7**
Concrete	Control	310.1 ± 16.1
	Encapsulated in Gel	271.9 ± 12.1
	Decon. Efficacy (%)	**87.7 ± 6.0**

21880x dilution factor for samples and controls

* Zinc powder is corrosive and reacted with all the surfaces evaluated, resulting in a fixed residue on the contaminated surface that could not be completely removed by DeconGel.

Table 3. Decontamination efficacy of DeconGel 1101 on zinc oxide (ZnO) contaminated stainless steel, carbon steel, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	553.4 ± 33.2
	Residual	0.086 ± 0.075
	Decon. Efficacy (%)	**100.0 ± 0.2**
Carbon Steel	Control	578.8 ± 14.3
	Residual	0.204 ± 0.104
	Decon. Efficacy (%)	**100.0 ± 0.4**
Concrete	Control	543.2 ± 22.4
	Residual	8.52 ± 7.23
	Decon. Efficacy (%)	**98.4 ± 0.2**

21880x dilution factor for samples and controls

Table 4. Decontamination efficacy of DeconGel 1101 on zinc oxide (ZnO) contaminated stainless steel, carbon steel and concrete surfaces as determined by direct DeconGel analysis.

Direct DeconGel Testing (ppm)		Formulation	
		DeconGel 1101[a]	DeconGel 1101[b]
Stainless Steel	Control	611.5 ± 5.1	596.1 ± 30.7
	Encapsulated in Gel	426.9 ± 58.2	594.2 ± 19.9
	Decon. Efficacy (%)	**69.8 ± 9.5**	**99.7 ± 6.1**
Carbon Steel	Control	678.7 ± 20.7	601.2 ± 16.0
	Encapsulated in Gel	581.8 ± 49.4	575.4 ± 10.4
	Decon. Efficacy (%)	**85.7 ± 7.3**	**95.7 ± 3.1**
Concrete	Control	578.5 ± 10.2	613.4 ± 11.5
	Encapsulated in Gel	477.3 ± 149.7	529.5 ± 6.2
	Decon. Efficacy (%)	**82.5 ± 25.9**	**86.3 ± 1.9**

21880x dilution factor for samples and controls

a. 100 mg of contaminant was applied on the substrate of interest and the samples were digested in 1% HNO_3 aqueous solution. Partial clogging of the ICP-OES nebulizer due to incomplete digestion of the inorganic contaminants after 72hrs was noted.

b. 50mg of contaminant was applied on the substrate of interest. Samples were digested in a concentrated acidic solution (15% HNO_3, 20%HCl, and 65% H_2O).

Table 5. Decontamination efficacy of DeconGel 1101 on zinc acetate ($Zn(O_2CCH_3)_2$) contaminated stainless steel, carbon steel, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	468.9 ± 25.3
	Residual	8.58 ± 1.38
	Decon. Efficacy (%)	**98.2 ± 6.0**
Carbon Steel	Control	474.4 ± 17.5
	Residual	7.71 ± 0.14
	Decon. Efficacy (%)	**98.4 ± 3.9**
Concrete	Control	477.1 ± 19.6
	Residual	11.4 ± 5.8
	Decon. Efficacy (%)	**97.6 ± 4.4**

21880x dilution factor for samples and controls

Table 6. Decontamination efficacy of DeconGel 1101 on zinc acetate ($Zn(O_2CCH_3)_2$) contaminated stainless steel, carbon steel, and concrete surfaces as determined by direct DeconGel analysis.

Direct Gel Testing (ppm)		**Formulation**	
		DeconGel 1101[a]	**DeconGel 1101[b]**
Stainless Steel	Control	490.7 ± 3.0	483.0 ± 30.4
	Encapsulated in Gel	489.8 ± 41.5	498.3 ± 25.7
	Decon. Efficacy (%)	**99.8 ± 8.5**	**103.2 ± 8.4**
Carbon Steel	Control	514.7 ± 0.8	502.0 ± 11.0
	Encapsulated in Gel	413.8 ± 91.1	502.7 ± 13.5
	Decon. Efficacy (%)	**80.4 ± 17.7**	**100.1 ± 3.5**
Concrete	Control	503.6 ± 3.1	NC
	Encapsulated in Gel	458.1 ± 35.7	NC
	Decon. Efficacy (%)	**91.0 ± 7.1**	NC

21880x dilution factor for samples and controls

c. Contaminant was spread evenly throughout the surface of interest. DeconGel 1101 was applied directly on top of the contaminated area without agitation.

d. Contaminant was spread evenly throughout the surface of interest. Methanol drops were placed on top of the contaminant to facilitate the formation of a thin layer of a contaminant deposit. After 30 minutes, DeconGel 1101 was applied directly on top of the contaminated area without agitation.

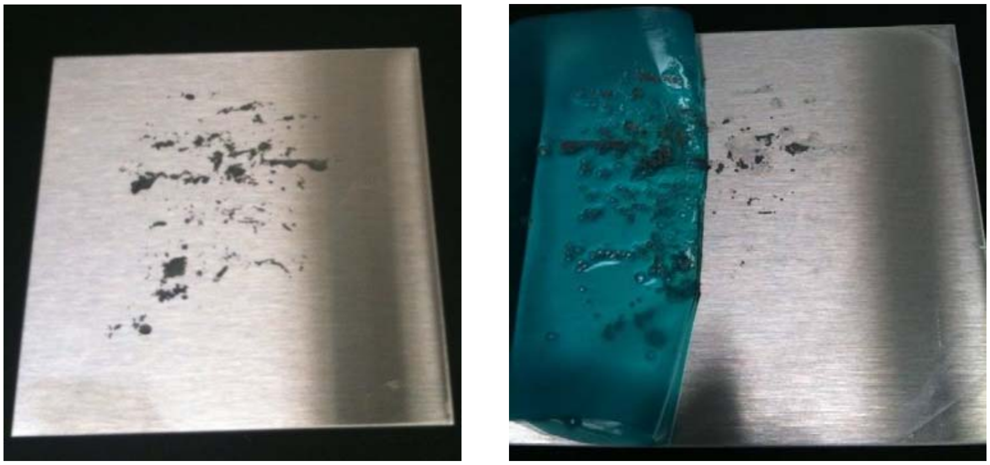
NC: not conducted

METHOD DEVELOPMENT OPTIMIZATIONS/OBSERVATIONS:

- We noted that amounts of contaminant solid greater than 0.05 g were difficult to spread evenly throughout coupon surfaces, forming clumps and piles which tended to overload poured DeconGel, significantly limiting the ability of DeconGel to encapsulate/emulsify surface contamination. This issue was circumvented by applying a modest amount (0.05 g) of contaminant solid throughout the surface of interest. Application of a homogenous, thin layer of contaminant on the respective substrate facilitated an optimized interaction between contaminant and DeconGel, and provided an accurate measure of DeconGel's decontamination efficacy in a scaled-down yet real-world setting. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel. Zinc powder, zinc oxide, and zinc acetate readily dissolve in the aqueous acidic solutions used to prepare all analytical samples and controls (20% HCl, 15% HNO_3, 65% DI H_2O).

- Zinc powder underwent a chemical reaction with stainless steel and carbon steel surfaces (Figure 1) which prevented some contamination from being sampled (swipe testing) and encapsulated into dry DeconGel (direct gel testing). Nevertheless DeconGel showed acceptable decontamination efficacy against loose mercury chloride contamination.

Figure 1. Zinc powder reacted with the carbon steel surface creating a fixed surface deposit.



- ASTM method E1728-03, a standardized swipe testing method used for sampling of inorganic contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes (pre-wetted with DI H_2O) were utilized in this swipe testing method.

- Direct chemical analysis of the dried peeled DeconGel samples was also utilized to provide an improved understanding of DeconGel's decontamination efficacy.

- Standardized EPA SW-846 Sampling Method 3005A "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy" was followed as a guideline to prepare all samples and controls. When deemed necessary, digestion methods were customized by increasing hydrochloric and nitric acid concentrations from 25% to 35% wt, and/or by heating samples to higher temperatures using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC) to afford the complete digestion/dissolution of the inorganic contaminants. All samples, controls, and standards were prepared using the same dissolution solution and experimental conditions to ensure both correct instrument calibration and accurate analytical results.

- ICP-OES instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of inorganic compounds. Standardized EPA SW-846 Analytical Method 6010C "Inductively Coupled Plasma-Atomic Emission Spectrometry" was followed as a guideline to prepare all samples and controls.

- To ensure accurate determination of DeconGel decontamination efficacy, calibration standards of the analyte of interest were prepared using either a sufficiently pure analyte or an appropriate ICP-MS Standard (Ricca Chemical Company; Arlington, TX); the respective standards were diluted to a known concentration (ppm) using the same digestion method as the one used for samples and controls. Instrument blank controls were DI H_2O ($\geq$17 M-Ohm).

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

Decontamination Efficacy (DeconGel Testing) =

(Contaminant (ppm) of DeconGel Direct/Contaminant (ppm) of DeconGel Control) x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.05 g of contaminant was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24-48 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe and gel samples were suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% deionized (DI) H_2O) for 24 h. When deemed necessary, samples were heated to 94 °C for 4-14 h resulting in complete dissolution of inorganic compounds using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC). Samples were allowed to cool to room temperature and were then analyzed via ICP-OES (see below).

Controls Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O) for 24 h and analyzed via ICP-OES (see below).

For DeconGel Control samples, a respectable amount of contaminant and approximately 6.0 g of dry DeconGel 1101 (pre-poured gel on the respective un-contaminated substrate and let to dry for 24-48 h) was suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O) for 24 h and analyzed via ICP-OES (see below).

Reagents and Standards

Zinc metal, Zn, finest powder grade (CAS# 7440-66-6, Fisher Scientific; Fair Lawn, NJ) was used as received

Zinc oxide, ZnO (CAS# 1314-13-2, Fisher Scientific; Fair Lawn, NJ) was used as receivedZinc acetate, $Zn(O_2CCH_3)_2$ (CAS# 5970-45-6, Fisher Scientific; Fair Lawn, NJ) was used as received.

A 1000 ppm calibration standard of each contaminant was prepared using the zinc and zinc compounds in freshly prepared aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O). DI H_2O was used as the blank sample.

Analytical Instrumentation

A Thermo ICP-OES instrument model radial iCap 6300 was used to determine zinc concentration (ppm) of all samples and controls using a freshly prepared 1000 ppm calibration standard.

Analyte (zinc) analyzed at 213.8 nm

Pump Speed: 0.5 mL/min

6.26. End-User Report for Aldehydes (Glutaraldehyde)

ANALYST: Geoff Nadolski PhD, Senior Polymer Scientist, CBI Polymers, Inc.

TITLE: Surface Decontamination of Aldehydes (Glutaraldehyde) by DeconGel 1101

OBJECTIVE: Surface decontamination determination of DeconGel formulation 1101 on stainless steel, aluminum, and concrete surfaces contaminated with Aldehydes (glutaraldehyde) using GC/MS (Gas Chromatography/Mass Spectrometry) following Environmental Protection Agency (EPA) SW-846 Methods 3500C (Sampling) and 8270C (Analysis).

HAZARDOUS MATERIALS RELEVANCE: Glutaraldehyde is a volatile liquid aldehyde used as a disinfectant, fixative, and plastics crosslinker. Glutaraldehyde is a strong and toxic disinfectant and can cause severe mucosal membrane irritation. Glutaraldehyde was chosen as a representative aldehyde; DeconGel is expected to have similar efficacy towards the wide range of aldehydes.

SUMMARY RESULTS:

- Excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with aldehydes (Glutaraldehyde) resulting in encapsulation of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 100% (on concrete) to 100% (on stainless steel) to 100% (on aluminum) for Glutaraldehyde determined by residual swipe analysis.
- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of organic compounds dissolved in a suitable solvent able to completely solvate aniline as well as DeconGel components. When necessary, experimental methods were customized to afford complete dissolution of organic contaminants. Additionally, analytical methods and associated equipment (GC column, GC temperature gradient program, MS sample ionization parameters) were appropriately developed to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Table 1 shows the decontamination efficacies of DeconGel 1101 on stainless steel, aluminum, and concrete surfaces contaminated with Aldehydes (glutaraldehyde) as determined by residual swipe testing.

Table 1. Decontamination efficacy of DeconGel 1101 on Glutaraldehyde Aldehyde contaminated stainless steel, aluminum, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		**Formulation** **DeconGel 1101**
Stainless Steel	Control	805.32 ± 0.91
	Residual	ND*
	Decon. Efficacy (%)	**100****
Aluminum	Control	805.03 ± 2.28
	Residual	ND
	Decon. Efficacy (%)	**100****
Concrete	Control	654.15 ± 3.76
	Residual	ND
	Decon. Efficacy (%)	**100****

1222x dilution factor for samples and controls

* ND: not detected, limit of detection (LOD) for glutaraldehyde approximates 250 ppb

** An ND value for residual experiments results in a Decontamination Efficacy of 100%

NOTES:

- ASTM method E1728-03, a standardized swipe testing method used for sampling of contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. Air-dried GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes were utilized in this swipe testing method.
- Standardized EPA SW-846 Sampling Method 3500C "Organic Extraction and Sample Preparation" was followed as a guideline to prepare all samples and controls. All samples, controls, and standards were prepared using the same solvent and appropriate experimental conditions to ensure accurate and GC/MS instrumental analysis.
- GC/MS instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of organic compounds. Standardized EPA SW-846 Analytical Method 8270C "SEMIVOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)" was followed as a guideline to prepare all samples and controls.

- To ensure accurate determination of DeconGel decontamination efficacy, a standard curve of the analyte of interest was prepared using sufficiently pure analyte; the respective standards were diluted to a known concentration (ppm) using the same solvent as used for samples and controls.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 90 uL (50% glutaraldehyde in water) of contaminant was evenly applied via brushing on 1) aluminum (surface area: 56.3 cm^2), 2) stainless steel (surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe and gel samples were suspended in 50 mL acetonitrile for 24 h. Samples were then analyzed via GC/MS (see below).

Controls Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied via brushing on 1) aluminum (surface area: 56.3 cm^2), 2) stainless steel (surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 50 mL acetonitrile for 24 h and analyzed via GC/MS (see below).

Reagents and Standards

Glutaraldehyde (pentane-1,5-dial) (CAS# 111-30-8, Fisher Scientific; Fair Lawn, NJ) was used as received.

Analytical Instrumentation

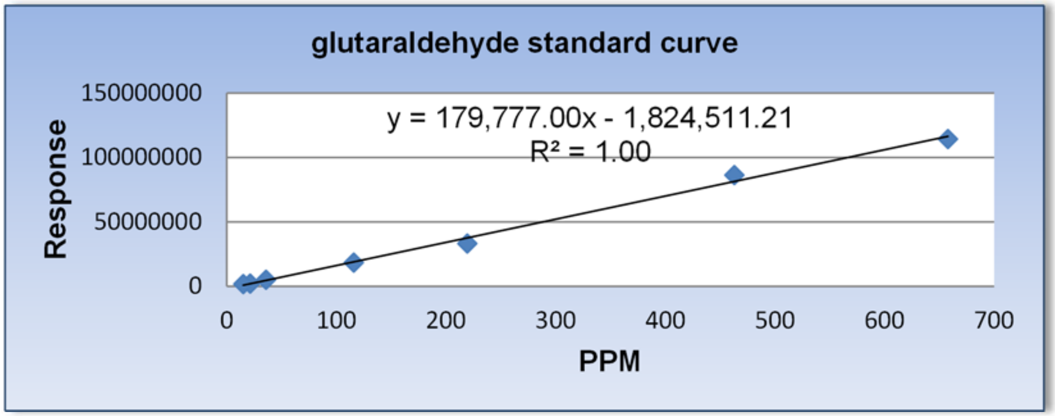
A Thermo DSQII GC/MS with autosampler in positive mode was used to determine glutaraldehyde concentration (ppm, wt/wt) of all samples and controls, using a Restek (Bellefonte, PA) Rtx-5 capillary column (30 m x 0.25 mm, 0.5 um).

A 7-point standard curve derived from three independently prepared stock solutions was prepared using DMSO as the working solvent.

GC method: start at 45 ºC, hold for 1 min, ramp at 30 ºC/min to 320 ºC, hold 10 min.

Glutaraldehyde GC/MS data: 4.3 min; M^+ = 100.

Figure 1. Glutaraldehyde standard calibration curve



APPLICATION INSTRUCTIONS FOR END-USERS:

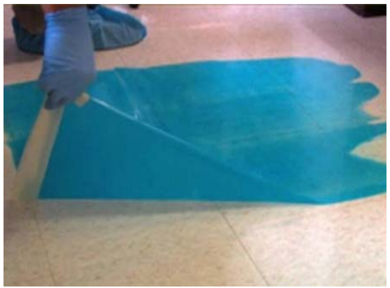
Use product directly as is from container. DO NOT DILUTE. Masking or painters tape can be applied along one edge of the area that is to be decontaminated to aid creating a peeled edge to grip for peeling the dried film. Apply DeconGel using a paint brush, a trowel, a handheld sprayer, or an industrial grade sprayer (use DeconGel 1120 or 1121 for spray application).

The thickness of the gel and the number of coats is dictated by the surface to be decontaminated. Coating thickness required for good peel characteristics varies with substrate and generally increases with substrate porosity. It is recommended that first time customers test DeconGel on a small sample area to confirm the required film thickness and dry time for their specific application. If the film is difficult to peel, please apply an additional coat. A razor blade is useful to start the peel. Lay the blade nearly flat and fillet the edge of the film to create a tab that can be pulled. For surfaces that the gel adheres to well, such as concrete, 12" – 24" strips can be cut in the film resulting in less force being required to peel the film.

- Let DeconGel dry for 24 hours

Dry time will vary depending on humidity, temperature, air flow and thickness of the DeconGel. This can take from as little time as an hour for thin coats in a dry environment with plenty of airflow, to overnight or longer if thicker coats are applied in humid environments. Dry times exceeding 24 hours may sometimes be required for good peel performance on bare concrete, wood and other highly porous substrates and substrates with deep cracks or grooves. However, 18-24 hrs is often sufficient dry time on good quality concrete. It is recommended that users test a small area to determine drying time prior to applying DeconGel for an entire job. Supplemental heat or air circulation will accelerate DeconGel's drying time for any job.

- Peel DeconGel off the surface by starting from one of the edges



When dry, the product locks the contaminants into a polymer matrix. The film containing the encapsulated contamination can then be peeled. DeconGel peels from most non-porous and porous hard surfaces if the dried film is thick enough. If the film is difficult to peel, add another coat, let dry, and peel. In most cases the DeconGel will come off in a single sheet but for odd shaped surfaces you may be required to score DeconGel in order to be able to peel it off.

- Dispose of the dried DeconGel in accordance with the local, state and Federal disposal regulations of the contaminant/substance you are removing. DeconGel itself has no special disposal restrictions.

For more information on application techniques visit our website: www.decongel.com

6.27. End-User Report for Alkaloids (Nicotine)

TITLE: Surface Decontamination of Alkaloids (Nicotine) by DeconGel 1101

ANALYST: Geoff Nadolski PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel formulation 1101 on stainless steel, aluminum, and concrete surfaces contaminated with Alkaloids (nicotine) using LC/MS (Liquid Chromatography/Mass Spectrometry) following Environmental Protection Agency (EPA) SW-846 Methods 3500C (Sampling) and 8321B (Analysis).

HAZARDOUS MATERIALS RELEVANCE: Nicotine is an aromatic (pyridine derivative) alkaloid produced by the nightshade family of plants such as tobacco. Nicotine stimulates production of cytochrome P450 liver enzymes during metabolism and possesses high oral and topical toxicities in comparison to other alkaloids. Nicotine was chosen as a representative alkaloid; DeconGel is expected to have similar efficacy towards the wide range of alkaloids.

SUMMARY RESULTS:

- Excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with alkaloids (nicotine) resulting in encapsulation of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 99.5% (on concrete) to 99.8% (on aluminum) to 99.8% (on stainless steel) for Nicotine determined by residual swipe analysis.
- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of organic compounds dissolved in a suitable solvent able to completely solvate nicotine as well as DeconGel components. When deemed necessary, experimental methods were customized to afford complete dissolution of organic contaminants. Additionally, analytical methods and associated equipment (LC column, LC gradient program, MS sample ionization parameters) were appropriately developed to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Table 1 shows the decontamination efficacies of DeconGel 1101 on stainless steel, aluminum, and concrete surfaces contaminated with Alkaloids (nicotine) as determined by residual swipe testing and direct DeconGel analysis.

Table 1. Decontamination efficacy of DeconGel 1101 on Nicotine Alkaloid contaminated stainless steel, aluminum, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		**Formulation**
		DeconGel 1101
Stainless Steel	Control	209.74 ± 1.49
	Residual	0.36 ± 0.005
	Decon. Efficacy (%)	**99.8 ± 0.0**
Aluminum	Control	209.85 ± 1.75
	Residual	0.30 ± 0.007
	Decon. Efficacy (%)	**99.8 ± 0.0**
Concrete	Control	152.94 ± 1.51
	Residual	0.77 ± 0.001
	Decon. Efficacy (%)	**99.5 ± 0.0**

2200x dilution factor for samples and controls

NOTES:

- ASTM method E1728-03, a standardized swipe testing method used for sampling of contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. Air-dried GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes (pre-wetted with DMSO immediately prior to performing swipe analysis) were utilized in this swipe testing method.

- Standardized EPA SW-846 Sampling Method 3500C "Organic Extraction and Sample Preparation" was followed as a guideline to prepare all samples and controls. All samples, controls, and standards were prepared using the same solvent and appropriate experimental conditions to ensure accurate and LC/MS instrumental analysis.

- LC/MS instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of organic compounds. Standardized EPA SW-846 Analytical Method 8321B "Solvent-Extractable Nonvolatile Compounds by High-Performance Liquid Chromatography/Thermospray/Mass Spectrometry (HPLC/TS/MS) or Ultraviolet (UV) Detection" was followed as a guideline to prepare all samples and controls.

- To ensure accurate determination of DeconGel decontamination efficacy, a standard curve of the analyte of interest was prepared using sufficiently pure analyte; the respective standards were diluted to a known concentration (ppm) using the same solvent as used for samples and controls.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 25 uL of contaminant was evenly applied via brushing on 1) stainless steel (surface area: 56.3 cm^2), 2) aluminum (surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe and gel samples were suspended in 50 mL DMSO for 24 h. Samples were then analyzed via LC/MS (see below).

Controls Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied via brushing on 1) stainless steel (surface area: 56.3 cm^2), 2) aluminum (surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 50 mL DMSO for 24 h and analyzed via LC/MS (see below).

Reagents and Standards

Nicotine (3-[(2S)-1-methylpyrrolidin-2-yl]pyridine) (CAS# 54-11-5, Fisher Scientific; Fair Lawn, NJ) was used as received.

Analytical Instrumentation

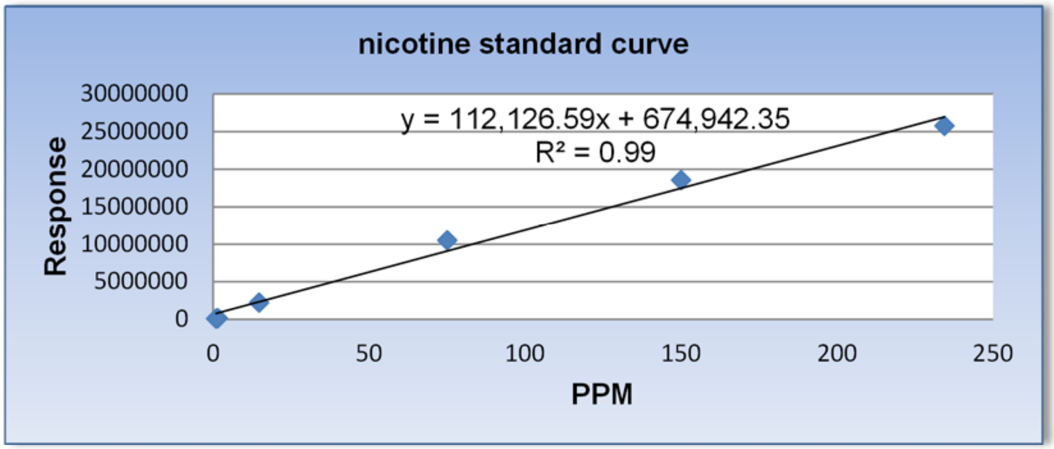
A Thermo LCQ LC/MS with autosampler in positive mode was used to determine nicotine concentration (ppm, wt/wt) of all samples and controls, using a Grace Davison (Deerfield, IL) C18-Select analytical column (150 x 4.6 mm, 5 um).

A 6-point standard curve derived from three independently prepared stock solutions was prepared using DMSO as the working solvent. The calibration curve exhibits a curve fitting as approximated by the coefficient of determination of linear regression R^2, where $R^2 = 0.99$ (see Figure 1).

LC method using A= water (0.1% formic acid), B=acetonitrile (0.1% formic acid); start at 90%A to 2 min at 70%A, to 3 min at 50%A, to 5 min at 30%A, to 6 min at 100%B, hold until 16 min at 90% A hold until 17 min.

Nicotine LC/MS data: 1.4 min; lambda max = 250, 273 nm; $M^+ = 162$.

Figure 1. <u>Nicotine Standard Calibration Curve</u>



APPLICATION INSTRUCTIONS FOR END-USERS:

Use product directly as is from container. DO NOT DILUTE. Masking or painters tape can be applied along one edge of the area that is to be decontaminated to aid creating a peeled edge to grip for peeling the dried film. Apply DeconGel using a paint brush, a trowel, a handheld sprayer, or an industrial grade sprayer (use DeconGel 1120 or 1121 for spray application).

The thickness of the gel and the number of coats is dictated by the surface to be decontaminated. Coating thickness required for good peel characteristics varies with substrate and generally increases with substrate porosity. It is recommended that first time customers test DeconGel on a small sample area to confirm the required film thickness and dry time for their specific application. If the film is difficult to peel, please apply an additional coat. A razor blade is useful to start the peel. Lay the blade nearly flat and fillet the edge of the film to create a tab that can be pulled. For surfaces that the gel adheres to well, such as concrete, 12" – 24" strips can be cut in the film resulting in less force being required to peel the film.

➢ Let DeconGel dry for 24 hours

Dry time will vary depending on humidity, temperature, air flow and thickness of the DeconGel. This can take from as little time as an hour for thin coats in a dry environment with plenty of airflow, to overnight or longer if thicker coats are applied in humid environments. Dry times exceeding 24 hours may sometimes be required for good peel performance on bare concrete, wood and other highly porous substrates and substrates with deep cracks or grooves. However, 18-24 hrs is often sufficient dry time on good quality concrete. It is recommended that users test a small area to determine drying time prior to applying DeconGel for an entire job. Supplemental heat or air circulation will accelerate DeconGel's drying time for any job.

➢ Peel DeconGel off the surface by starting from one of the edges

When dry, the product locks the contaminants into a polymer matrix. The film containing the encapsulated contamination can then be peeled. DeconGel peels from most non-porous and porous hard surfaces if the dried film is thick enough. If the film is difficult to peel, add another coat, let dry, and peel. In most cases the DeconGel will come off in a single sheet but for odd shaped surfaces you may be required to score DeconGel in order to be able to peel it off.

- ➢ Dispose of the dried DeconGel in accordance with the local, state and Federal disposal regulations of the contaminant/substance you are removing. DeconGel itself has no special disposal restrictions.

For more information on application techniques visit our website: www.decongel.com

6.28. End-User Report for Aluminum Compounds (Aluminum powder, Aluminum Oxide, Aluminum Chloride and Aluminum Potassium Sulfate)

TITLE: Surface Decontamination of Aluminum Compounds (Elemental Aluminum Powder (Al), Aluminum Oxide (Al_2O_3), Aluminum Chloride ($AlCl_3$) and Aluminum Potassium Sulfate ($AlK(SO_4)_2$)) by DeconGel 1101

AUTHOR: Andreas Mylonakis PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with Aluminum Compounds (elemental aluminum powder (Al), aluminum oxide (Al_2O_3), aluminum chloride ($AlCl_3$), and aluminum potassium sulfate ($AlK(SO_4)_2$)) using ICP-OES (Inductively Coupled Plasma-Optical Emission Spectroscopy) following Environmental Protection Agency (EPA) SW-846 Methods 3005A (sampling) and 6010C (analysis).

HAZARDOUS MATERIALS RELEVANCE: Aluminum and its compounds are widely used in a number of industries including consumer goods such as cosmetics and aluminum cans. Aluminum compounds can become easily airborne and as such are considered inhalation hazards. Aluminum powder, aluminum oxide, aluminum chloride, and aluminum potassium sulfate were chosen as representative aluminum compounds for evaluating DeconGel's efficacy; DeconGel is expected to have similar efficacy towards the wide range of aluminum compounds.

SUMMARY RESULTS:

- Excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with aluminum compounds resulting in encapsulation of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 for aluminum compounds (except aluminum powder elemental which is corrosive and reacts with the metal surfaces evaluated) ranged from greater than 93.0% (on concrete) to greater than 95.4% (on carbon steel) to greater than 96.4% (on stainless steel) as determined by residual swipe analysis.

- Aluminum powder is reactive to some metal surfaces including carbon steel and stainless steel. DeconGel could not completely remove aluminum elemental powder that reacted with and created a fixed deposit on steel surfaces. Nevertheless, DeconGel showed excellent decontamination efficacy on loose aluminum powder contaminant from such surfaces.

- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of inorganics in aqueous samples. When necessary, the digestion methods were customized to result in the complete dissolution of the inorganic contaminants and to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 through 4 show the decontamination efficacies of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with elemental aluminum powder (Al), aluminum oxide (Al_2O_3), aluminum chloride ($AlCl_3$) and aluminum potassium sulfate ($AlK(SO_4)_2$) as determined by residual swipe testing.

Table 1. Decontamination efficacies of DeconGel 1101 on aluminum powder (Al) contaminated stainless steel, carbon steel, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel*	Control	1113.0 ± 17.0
	Residual	23.3 ± 5.3
	Decon. Efficacy (%)	**97.9 ± 2.0**
Carbon Steel*	Control	944.7 ± 0.2
	Residual	4.7 ± 4.3
	Decon. Efficacy (%)	**99.5 ± 4.6**
Concrete	Control	511.7 ± 12.7
	Residual	1.8 ± 0.2
	Decon. Efficacy (%)	**99.6 ± 3.8**

2280x dilution factor for samples and controls

Table 2. Decontamination efficacies of DeconGel against aluminum oxide (Al_2O_3) contaminated stainless steel, carbon steel, and concrete surfaces as determined by residual swipe testing method.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	420.8 ± 7.0
	Residual	1.4 ± 0.6
	Decon. Efficacy (%)	**99.7 ± 7.7**
Carbon Steel	Control	505.5 ± 22.4
	Residual	0.3 ± 0.3
	Decon. Efficacy (%)	**99.9 ± 14.4**
Concrete	Control	420.2 ± 6.1
	Residual	1.3 ± 0.7
	Decon. Efficacy (%)	**99.7 ± 5.9**

2280x dilution factor for samples and controls

Table 3. Decontamination efficacies of DeconGel 1101 against aluminum chloride ($AlCl_3$) contaminated stainless steel, carbon steel, and concrete surfaces as determined by residual swipe testing method.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	325.0 ± 7.9
	Residual	3.6 ± 2.3
	Decon. Efficacy (%)	**98.9 ± 0.7**
Carbon Steel	Control	319.2 ± 18.4
	Residual	4.9 ± 1.4
	Decon. Efficacy (%)	**98.5 ± 5.9**
Concrete	Control	369.4 ± 73.7
	Residual	25.8 ± 4.4
	Decon. Efficacy (%)	**93.0 ± 18.7**

2280x dilution factor for samples and controls

Table 4. Decontamination efficacies of DeconGel 1101 on aluminum potassium sulfate ($AlK(SO_4)_2$) contaminated stainless steel, carbon steel, and concrete surfaces as determined by residual swipe testing method.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	441.8 ± 20.7
	Residual	15.9 ± 3.9
	Decon. Efficacy (%)	**96.4 ± 4.9**
Carbon Steel	Control	442.0 ± 13.6
	Residual	20.4 ± 7.4
	Decon. Efficacy (%)	**95.4 ± 4.2**
Concrete	Control	399.8 ± 47.5
	Residual	20.1 ± 3.3
	Decon. Efficacy (%)	**95.0 ± 11.4**

2280x dilution factor for samples and controls

NOTES:

- ASTM method E1728-03, a standardized swipe testing method used for the sampling of inorganic contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes (pre-wetted with DI H_2O) were utilized in this swipe testing method.

- Standardized EPA SW-846 Sampling Method 3005A "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy" was followed as a guideline to prepare all samples and controls. When deemed necessary, digestion methods were customized by increasing hydrochloric and nitric acid concentrations from 25% to 35% wt, and/or by heating samples to higher temperatures using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC) to facilitate the complete digestion/dissolution of the inorganic contaminants. All samples, controls, and standards were prepared using the same dissolution solution and experimental conditions to ensure both correct instrument calibration and accurate analytical results.

- ICP-OES instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of inorganic compounds. Standardized EPA SW-846 Analytical Method 6010C "Inductively Coupled Plasma-Atomic Emission Spectrometry" was followed as a guideline to prepare all samples and controls.

- To ensure accurate determination of DeconGel decontamination efficacy, calibration standards of the analyte of interest were prepared using either a sufficiently pure analyte or an appropriate ICP-MS Standard (Ricca Chemical Company; Arlington, TX); the respective standards were diluted to a known concentration (ppm) using the same digestion method as the one used for samples and controls. Instrument blank controls were DI H_2O ($\geq$17 M-Ohm).

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.05 g of contaminant was evenly applied on 1) stainless steel (commercial grade, surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24-48 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% deionized (DI) H_2O) for 24 h and were then analyzed via ICP-OES (see below).

Controls Methods

For Swipe Control samples, the respective amount of contaminant was evenly applied on 1) stainless steel (commercial grade, surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O) for 24 h and analyzed via ICP-OES (see below).

Reagents and Standards

Aluminium metal, Al, finest powder grade, (CAS# 7429-90-5, Fisher Scientific; Fair Lawn, NJ), was used as received.

Aluminium oxide, Al_2O_3, 60-325 Mesh, (CAS# 1344-28-1, Fisher Scientific; Fair Lawn, NJ), was used as received.

Aluminum Chloride, $AlCl_3$, fine crystalline solid, (CAS# 7446-70-7, Fisher Scientific; Fair Lawn, NJ), was used as received.

Aluminum Potassium Sulfate, $AlK(SO_4)_2$, fine crystalline solid, (CAS# 7784-24-9, Fisher Scientific; Fair Lawn, NJ), was used as received

A 1000 ppm calibration standard of each contaminant was prepared using the aluminum and aluminum compounds in freshly prepared aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O). DI H_2O ($\geq$ 17 M-Ohm) was used as the blank sample.

Analytical Instrumentation

A Thermo ICP-OES instrument model radial iCap 6300 was used to determine aluminum concentration (ppm) of all samples and controls using a freshly prepared 1000 ppm calibration standard. Analyte (aluminum) analyzed at 308.2 nm. Pump Speed: 0.5 mL/min

APPLICATION INSTRUCTIONS FOR END-USERS:

Use product directly as is from container. DO NOT DILUTE. Masking or painters tape can be applied along one edge of the area that is to be decontaminated to aid creating a peeled edge to grip for peeling the dried film. Apply DeconGel using a paint brush, a trowel, a handheld sprayer, or an industrial grade sprayer (use DeconGel 1120 or 1121 for spray application).

The thickness of the gel and the number of coats is dictated by the surface to be decontaminated. Coating thickness required for good peel characteristics varies with substrate and generally increases with substrate porosity. It is recommended that first time customers test DeconGel on a small sample area to confirm the required film thickness and dry time for their specific application. If the film is difficult to peel, please apply an additional coat. A razor blade is useful to start the peel. Lay the blade nearly flat and fillet the edge of the film to create a tab that can be pulled. For surfaces that the gel adheres to well, such as concrete, 12" – 24" strips can be cut in the film resulting in less force being required to peel the film.

- Let DeconGel dry for 24 hours

Dry time will vary depending on humidity, temperature, air flow and thickness of the DeconGel. This can take from as little time as an hour for thin coats in a dry environment with plenty of airflow, to overnight or longer if thicker coats are applied in humid environments. Dry times exceeding 24 hours may sometimes be required for good peel performance on bare concrete, wood and other highly porous substrates and substrates with deep cracks or grooves. However, 18-24 hrs is often sufficient dry time on good quality concrete. It is recommended that users test a small area to determine drying time prior to applying DeconGel for an entire job. Supplemental heat or air circulation will accelerate DeconGel's drying time for any job.

- Peel DeconGel off the surface by starting from one of the edges

When dry, the product locks the contaminants into a polymer matrix. The film containing the encapsulated contamination can then be peeled. DeconGel peels from most non-porous and porous hard surfaces if the dried film is thick enough. If the film is difficult to peel, add another coat, let dry, and peel. In most cases the DeconGel will come off in a single sheet but for odd shaped surfaces you may be required to score DeconGel in order to be able to peel it off.

- Dispose of the dried DeconGel in accordance with the local, state and Federal disposal regulations of the contaminant/substance you are removing. DeconGel itself has no special disposal restrictions.

For more information on application techniques visit our website:
www.decongel.com

6.29. End-User Report for Amine Compounds (Aniline)

TITLE: Surface Decontamination of Amine Compounds (Aniline) by DeconGel 1101

ANALYST: Geoff Nadolski PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel formulation 1101 on stainless steel, aluminum, and concrete surfaces contaminated with Amine Compounds (Aniline) using GC/MS (Gas Chromatography/Mass Spectrometry) following Environmental Protection Agency (EPA) SW-846 Methods 3500C (Sampling) and 8270C (Analysis).

HAZARDOUS MATERIALS RELEVANCE: Aniline is a volatile aromatic amine used mainly as a chemical feedstock to produce plastics including polyurethane, and used as a rubber additive. Aniline is acutely toxic, and prolonged exposure can result in hemolytic anemia and tumor formation in the spleen. Aniline was chosen as a representative amine compounds (industrial plastics feedstock); DeconGel is expected to have similar efficacy towards the wide range of amine compounds.

SUMMARY RESULTS:

- Excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with amine compounds (Aniline) resulting in encapsulation of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 100% (on concrete) to 100% (on stainless steel) to 100% (on aluminum) for Aniline determined by residual swipe analysis.
- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of organic compounds dissolved in a suitable solvent able to completely solvate aniline as well as DeconGel components. When deemed necessary, experimental methods were customized to afford complete dissolution of organic contaminants. Additionally, analytical methods and associated equipment (GC column, GC temperature gradient program, MS sample ionization parameters) were appropriately developed to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Table 1 shows the decontamination efficacies of DeconGel 1101 on stainless steel, aluminum, and concrete surfaces contaminated with amine compounds (aniline) as determined by residual swipe testing.

Table 1. Decontamination efficacy of DeconGel 1101 on Aniline contaminated stainless steel, aluminum, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	449.79 ± 2.14
	Residual	ND*
	Decon. Efficacy (%)	**100****
Aluminum	Control	449.88 ± 1.56
	Residual	ND
	Decon. Efficacy (%)	**100****
Concrete	Control	394.92 ± 0.43
	Residual	ND
	Decon. Efficacy (%)	**100****

1142x dilution factor for samples and controls

* ND: not detected, limit of detection (LOD) for aniline approximates 100 ppb

** An ND value for residual experiments results in a Decontamination Efficacy of 100%

NOTES:

- ASTM method E1728-03, a standardized swipe testing method used for sampling of contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. Air-dried GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes were utilized in this swipe testing method.
- Standardized EPA SW-846 Sampling Method 3500C "Organic Extraction and Sample Preparation" was followed as a guideline to prepare all samples and controls. All samples, controls, and standards were prepared using the same solvent and appropriate experimental conditions to ensure accurate and GC/MS instrumental analysis.
- GC/MS instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of organic compounds. Standardized EPA SW-846 Analytical Method 8270C "SEMIVOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)" was followed as a guideline to prepare all samples and controls.
- To ensure accurate determination of DeconGel decontamination efficacy, a standard curve of the analyte of interest was prepared using sufficiently pure analyte; the respective standards were diluted to a known concentration (ppm) using the same solvent as used for samples and controls.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 35 uL of contaminant was evenly applied via brushing on 1) aluminum (surface area: 56.3 cm^2), 2) stainless steel (surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe and gel samples were suspended in 50 mL 60% methanol in water for 24 h. Samples were then analyzed via GC/MS (see below).

Controls Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied via brushing on 1) aluminum (surface area: 56.3 cm^2), 2) stainless steel (surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 50 mL 60% methanol in water for 24 h and analyzed via GC/MS (see below).

Reagents and Standards

Aniline (phenylamine) (CAS# 62-53-3, Fisher Scientific; Fair Lawn, NJ) was used as received.

Analytical Instrumentation

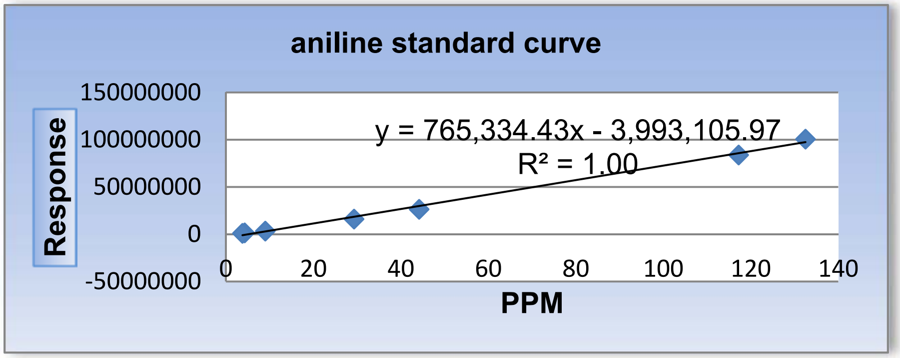
A Thermo DSQII GC/MS with autosampler in positive mode was used to determine aniline concentration (ppm, wt/wt) of all samples and controls, using a Restek (Bellefonte, PA) Rtx-5 capillary column (30 m x 0.25 mm, 0.5 um).

A 7-point standard curve derived from three independently prepared stock solutions was prepared using DMSO as the working solvent.

GC method: start at 45 ºC, hold for 1 min, ramp at 30 ºC/min to 320 ºC, hold 10 min.

Aniline GC/MS data: 4.0 min; $M^+ = 93$.

Figure 1. Aniline Standard Calibration Curve



APPLICATION INSTRUCTIONS FOR END-USERS:

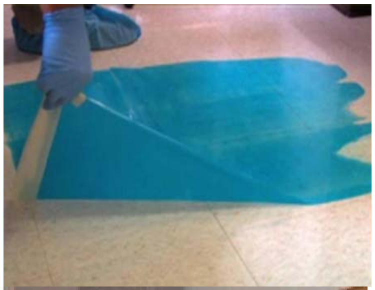
Use product directly as is from container. DO NOT DILUTE. Masking or painters tape can be applied along one edge of the area that is to be decontaminated to aid creating a peeled edge to grip for peeling the dried film. Apply DeconGel using a paint brush, a trowel, a handheld sprayer, or an industrial grade sprayer (use DeconGel 1120 or 1121 for spray application).

The thickness of the gel and the number of coats is dictated by the surface to be decontaminated. Coating thickness required for good peel characteristics varies with substrate and generally increases with substrate porosity. It is recommended that first time customers test DeconGel on a small sample area to confirm the required film thickness and dry time for their specific application. If the film is difficult to peel, please apply an additional coat. A razor blade is useful to start the peel. Lay the blade nearly flat and fillet the edge of the film to create a tab that can be pulled. For surfaces that the gel adheres to well, such as concrete, 12" – 24" strips can be cut in the film resulting in less force being required to peel the film.

- Let DeconGel dry for 24 hours

Dry time will vary depending on humidity, temperature, air flow and thickness of the DeconGel. This can take from as little time as an hour for thin coats in a dry environment with plenty of airflow, to overnight or longer if thicker coats are applied in humid environments. Dry times exceeding 24 hours may sometimes be required for good peel performance on bare concrete, wood and other highly porous substrates and substrates with deep cracks or grooves. However, 18-24 hrs is often sufficient dry time on good quality concrete. It is recommended that users test a small area to determine drying time prior to applying DeconGel for an entire job. Supplemental heat or air circulation will accelerate DeconGel's drying time for any job.

- Peel DeconGel off the surface by starting from one of the edges



When dry, the product locks the contaminants into a polymer matrix. The film containing the encapsulated contamination can then be peeled. DeconGel peels from most non-porous and porous hard surfaces if the dried film is thick enough. If the film is difficult to peel, add another coat, let dry, and peel. In most cases the DeconGel will come off in a single sheet but for odd shaped surfaces you may be required to score DeconGel in order to be able to peel it off.

- Dispose of the dried DeconGel in accordance with the local, state and Federal disposal regulations of the contaminant/substance you are removing. DeconGel itself has no special disposal restrictions.

For more information on application techniques visit our website:
www.decongel.com

6.30. End-User Report for Aromatic Liquids (Toluene)

TITLE: Surface Decontamination of Aromatic Liquids (Toluene) by DeconGel 1101 and 1102

ANALYST: Geoff Nadolski PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel formulation 1101 and 1102 on stainless steel, floor tile, and concrete surfaces contaminated with Aromatic Liquids (toluene) using LC/MS (Liquid Chromatography/Mass Spectrometry) following Environmental Protection Agency (EPA) SW-846 Methods 3500C (Sampling) and 8321B (Analysis).

HAZARDOUS MATERIALS RELEVANCE: Toluene is a volatile aromatic hydrocarbon widely used as a solvent, industrial feedstock, and as an octane booster in gasoline fuels. If inhaled, ingested, or internalized in large quantities, toluene can cause neurological, liver, and kidney damage, unconsciousness, and death. Toluene was chosen as a representative aromatic liquid; DeconGel is expected to have similar efficacy towards the wide range of aromatic liquids.

SUMMARY RESULTS:

- Excellent surface decontamination was achieved by applying both DeconGel 1101 and 1102, both via brushing or pouring (non-brushed) onto contaminated surfaces, resulting in encapsulation of toluene contaminant by DeconGel's active components. Decontamination efficacies of poured DeconGel 1101 ranged from 99.5% (on concrete) to 99.8% (on floor tile) to 99.8% (on stainless steel), brushed DeconGel 1101 ranged from 100% (on concrete) to 100% (on floor tile) to 100% (on stainless steel), as determined by residual swipe analysis. Decontamination efficacies of poured DeconGel 1102 ranged from 100% (on concrete) to 100% (on floor tile) to 100% (on stainless steel), brushed DeconGel 1102 ranged from 100% (on concrete) to 100% (on floor tile) to 100% (on stainless steel), as determined by residual swipe analysis.
- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of organic compounds dissolved in a suitable solvent able to completely solvate toluene as well as DeconGel components. When deemed necessary, experimental methods were customized to afford complete dissolution of organic contaminants. Additionally, analytical methods and associated equipment (LC column, LC gradient program, MS sample

ionization parameters) were appropriately developed to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 shows the decontamination efficacies of DeconGel 1101 and 1102 on stainless steel, floor tile, and concrete surfaces contaminated with Aromatic Liquids (toluene) as determined by residual swipe testing.

Table 1. Decontamination efficacies of DeconGel 1101 and 1102 on Toluene Aromatic Liquid contaminated stainless steel, floor tile, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		**Formulation**	
		DeconGel 1101	**DeconGel 1102**
Stainless Steel	Control	658.42 ± 2.86	658.42 ± 2.86
	Residual (non-brushed)	1.32 ± 0.10	ND[*]
	Residual (brushed)	ND	ND
	Decon. Efficacy (non-brushed) (%)	**99.8 ± 0.0**	**100****
	Decon. Efficacy (brushed) (%)	**100****	**100****
Floor Tile	Control	631.67 ± 1.07	631.67 ± 1.07
	Residual (non-brushed)	1.33 ± 0.06	ND
	Residual (brushed)	ND	ND
	Decon. Efficacy (non-brushed) (%)	**99.8 ± 0.0**	**100****
	Decon. Efficacy (brushed) (%)	**100****	**100****
Concrete	Control	482.95 ± 0.62	482.95 ± 0.62
	Residual (non-brushed)	2.48 ± 0.07	ND
	Residual (brushed)	ND	ND
	Decon. Efficacy (non-brushed) (%)	**99.5 ± 0.0**	**100****
	Decon. Efficacy (brushed) (%)	**100****	**100****

1571x dilution factor for samples and controls

* ND: not detected, limit of detection (LOD) for toluene approximates 250 ppb

** An ND value for residual experiments results in a Decontamination Efficacy of 100%

NOTES:

- ASTM method E1728-03, a standardized swipe testing method used for sampling of contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. Air-dried GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes were utilized in this swipe testing method.
- Standardized EPA SW-846 Sampling Method 3500C "Organic Extraction and Sample Preparation" was followed as a guideline to prepare all samples and controls. All samples, controls, and standards were prepared using the

same solvent and appropriate experimental conditions to ensure accurate and LC/MS instrumental analysis.

- LC/MS instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of organic compounds. Standardized EPA SW-846 Analytical Method 8321B "Solvent-Extractable Nonvolatile Compounds by High-Performance Liquid Chromatography/Thermospray/Mass Spectrometry (HPLC/TS/MS) or Ultraviolet (UV) Detection" was followed as a guideline to prepare all samples and controls.

- To ensure accurate determination of DeconGel decontamination efficacy, a standard curve of the analyte of interest was prepared using sufficiently pure analyte; the respective standards were diluted to a known concentration (ppm) using the same solvent as used for samples and controls.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 40 uL of contaminant was evenly applied via brushing on 1) stainless steel (surface area: 56.3 cm^2), 2) floor tile (surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe and gel samples were suspended in 50 mL DMSO for 24 h. Samples were then analyzed via LC/MS (see below).

Controls Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied via brushing on 1) stainless steel (surface area: 56.3 cm^2), 2) floor tile (surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 50 mL DMSO for 24 h and analyzed via LC/MS (see below).

Reagents and Standards

Toluene (methylbenzene) (CAS# 108-88-3, Fisher Scientific; Fair Lawn, NJ) was used as received.

Analytical Instrumentation

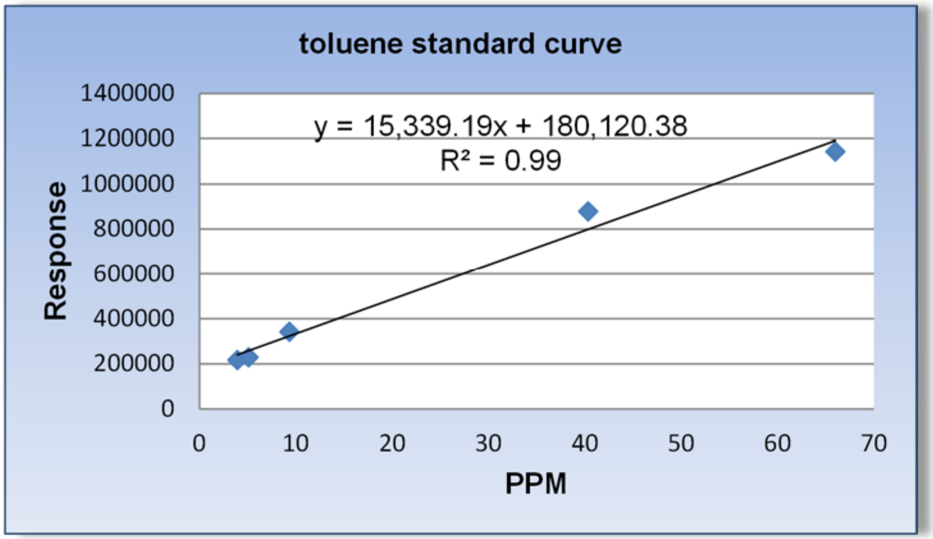
A Thermo LCQ LC/MS with autosampler in positive mode was used to determine toluene concentration (ppm, wt/wt) of all samples and controls, using a Grace Davison (Deerfield, IL) C18-Select analytical column (150 x 4.6 mm, 5 um).

A 5-point standard curve derived from three independently prepared stock solutions was prepared using DMSO as the working solvent. The calibration curve exhibits a curve fitting as approximated by the coefficient of determination of linear regression R^2, where $R^2 = 0.99$ (see Figure 1).

LC method using A= water (0.1% formic acid), B=acetonitrile (0.1% formic acid); start at 95%A to 3 min at 70%A, to 6 min at 50%A, to 12 min at 30%A, to 15 min at 90%B, to 16 min at 100%B, hold until 22 min at 100% B, to 23 min at 95%A, hold until 24 min.

Toluene LC/MS data: 13.3 min; lambda max = 262, 268 nm; $M^+ = 92$.

Figure 1. **Toluene Standard Calibration Curve**



APPLICATION INSTRUCTIONS FOR END-USERS:

Use product directly as is from container. DO NOT DILUTE. Masking or painters tape can be applied along one edge of the area that is to be decontaminated to aid creating a peeled edge to grip for peeling the dried film. Apply DeconGel using a paint brush, a trowel, a handheld sprayer, or an industrial grade sprayer (use DeconGel 1120 or 1121 for spray application).

The thickness of the gel and the number of coats is dictated by the surface to be decontaminated. Coating thickness required for good peel characteristics varies with substrate and generally increases with substrate porosity. It is recommended that first time customers test DeconGel on a small sample area to

confirm the required film thickness and dry time for their specific application. If the film is difficult to peel, please apply an additional coat. A razor blade is useful to start the peel. Lay the blade nearly flat and fillet the edge of the film to create a tab that can be pulled. For surfaces that the gel adheres to well, such as concrete, 12” – 24” strips can be cut in the film resulting in less force being required to peel the film.

- Let DeconGel dry for 24 hours

Dry time will vary depending on humidity, temperature, air flow and thickness of the DeconGel. This can take from as little time as an hour for thin coats in a dry environment with plenty of airflow, to overnight or longer if thicker coats are applied in humid environments. Dry times exceeding 24 hours may sometimes be required for good peel performance on bare concrete, wood and other highly porous substrates and substrates with deep cracks or grooves. However, 18-24 hrs is often sufficient dry time on good quality concrete. It is recommended that users test a small area to determine drying time prior to applying DeconGel for an entire job. Supplemental heat or air circulation will accelerate DeconGel’s drying time for any job.

- Peel DeconGel off the surface by starting from one of the edges

When dry, the product locks the contaminants into a polymer matrix. The film containing the encapsulated contamination can then be peeled. DeconGel peels from most non-porous and porous hard surfaces if the dried film is thick enough. If the film is difficult to peel, add another coat, let dry, and peel. In most cases the DeconGel will come off in a single sheet but for odd shaped surfaces you may be required to score DeconGel in order to be able to peel it off.

- Dispose of the dried DeconGel in accordance with the local, state and Federal disposal regulations of the contaminant/substance you are removing. DeconGel itself has no special disposal restrictions.

For more information on application techniques visit our website:
www.decongel.com

6.31. End-User Report for Aromatic Solids (Naphthalene)

TITLE: Surface Decontamination of Aromatic Solids (Naphthalene) by DeconGel 1101

ANALYST: Geoff Nadolski PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel formulation 1101 on stainless steel, aluminum, and concrete surfaces contaminated with Aromatic Solids (naphthalene) using LC/MS (Liquid Chromatography/Mass Spectrometry) following Environmental Protection Agency (EPA) SW-846 Methods 3500C (Sampling) and 8321B (Analysis).

HAZARDOUS MATERIALS RELEVANCE: Naphthalene is a polycyclic aromatic hydrocarbon (PAH) that is volatile, readily subliming at room temperature. Naphthalene is the most abundant single component of coal tar, and is used as a chemical precursor to other chemicals, as a wetting agent/surfactant, and is used as a fumigant. Exposure to naphthalene may damage red blood cells, and the International Agency for Research on Cancer (IARC) classifies naphthalene as a potential carcinogen to animals and humans. Naphthalene was chosen as a representative solid aromatic compound; DeconGel is expected to have similar efficacy towards the wide range of aromatic solids.

SUMMARY RESULTS:

- Excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with aromatic solids (naphthalene) resulting in encapsulation of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 99.2% (on concrete) to 99.6% (on stainless steel) to 99.2% (on aluminum) for Naphthalene as determined by residual swipe analysis.

- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of organic compounds dissolved in a suitable solvent able to completely solvate naphthalene as well as DeconGel components. When necessary, experimental methods were customized to afford complete dissolution of organic contaminants. Additionally, analytical methods and associated equipment (LC column, LC gradient program, MS sample ionization parameters) were appropriately utilized to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Table 1 shows the decontamination efficacies of DeconGel 1101 on stainless steel, aluminum, and concrete surfaces contaminated with Aromatic Solids (Naphthalene) as determined by residual swipe testing.

Table 1. Decontamination efficacy of DeconGel 1101 on Naphthalene Aromatic Solid contaminated aluminum, stainless steel, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	223.46 ± 0.38
	Residual	0.94 ± 0.01
	Decon. Efficacy (%)	**99.6 ± 0.0**
Aluminum	Control	223.47 ± 0.30
	Residual	0.95 ± 0.01
	Decon. Efficacy (%)	**99.6 ± 0.0**
Concrete	Control	169.0 ± 0.77
	Residual	1.27 ± 0.01
	Decon. Efficacy (%)	**99.2 ± 0.01**

2200x dilution factor for samples and controls

NOTES:

- ASTM method E1728-03, a standardized swipe testing method used for sampling of contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. Air-dried GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes (pre-wetted with DMSO immediately prior to performing swipe analysis) were utilized in this swipe testing method.

- Standardized EPA SW-846 Sampling Method 3500C "Organic Extraction and Sample Preparation" was followed as a guideline to prepare all samples and controls. All samples, controls, and standards were prepared using the same solvent and appropriate experimental conditions to ensure accurate and LC/MS instrumental analysis.

- LC/MS instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of organic compounds. Standardized EPA SW-846 Analytical Method 8321B "Solvent-Extractable Nonvolatile Compounds by High-Performance Liquid Chromatography/Thermospray/Mass Spectrometry (HPLC/TS/MS) or Ultraviolet (UV) Detection" was followed as a guideline to prepare all samples and controls.

- To ensure accurate determination of DeconGel decontamination efficacy, a standard curve of the analyte of interest was prepared using sufficiently pure analyte; the respective standards were diluted to a known concentration (ppm) using the same solvent as used for samples and controls.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.025 g of contaminant was evenly applied on 1) aluminum (surface area: 56.3 cm^2), 2) stainless steel (surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC) wetted with DMSO (2 mL) immediately prior to swipe analysis. Swipe and gel samples were suspended in 50 mL DMSO for 24 h. Samples were then analyzed via LC/MS (see below).

Controls Methods

For Swipe Control samples, a respective amount of contaminant was evenly applied on 1) aluminum (surface area: 56.3 cm^2), 2) stainless steel (surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC) wetted with DMSO (2 mL) immediately prior to swipe analysis. Swipe samples were suspended in 50 mL DMSO for 24 h and analyzed via LC/MS (see below).

Reagents and Standards

Naphthalene (bicyclo[4.4.0]deca-1,3,5,7,9-pentene) (CAS# 91-20-3, Fisher Scientific; Fair Lawn, NJ) was used as received.

Analytical Instrumentation

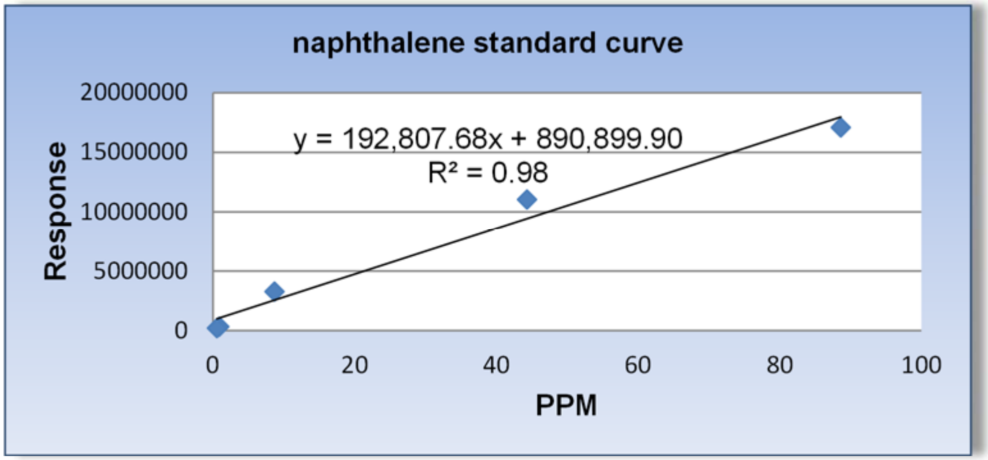
A Thermo LCQ LC/MS with autosampler in positive mode was used to determine naphthalene concentration (ppm, wt/wt) of all samples and controls, using a Grace Davison (Deerfield, IL) C18-Select analytical column (150 x 4.6 mm, 5 um).

A 5-point standard curve derived from three independently prepared stock solutions was prepared using DMSO as the working solvent. The calibration curve exhibits a curve fitting as approximated by the coefficient of determination of linear regression R^2, where $R^2 = 0.98$ (see Figure 1).

LC method using A= water (0.1% formic acid), B=acetonitrile (0.1% formic acid); start at 90%A to 2 min at 70%A, to 3 min at 50%A, to 5 min at 30%A, to 6 min at 100%B, hold until 16 min at 90% A hold until 17 min.

Naphthalene LC/MS data: 8.8 min; lambda max = 250, 313 nm; $M^+ = 128$.

Figure 1. Naphthalene Standard Calibration Curve



APPLICATION INSTRUCTIONS FOR END-USERS:

Use product directly as is from container. DO NOT DILUTE. Masking or painters tape can be applied along one edge of the area that is to be decontaminated to aid creating a peeled edge to grip for peeling the dried film. Apply DeconGel using a paint brush, a trowel, a handheld sprayer, or an industrial grade sprayer (use DeconGel 1120 or 1121 for spray application).

The thickness of the gel and the number of coats is dictated by the surface to be decontaminated. Coating thickness required for good peel characteristics varies with substrate and generally increases with substrate porosity. It is recommended that first time customers test DeconGel on a small sample area to confirm the required film thickness and dry time for their specific application. If the film is difficult to peel, please apply an additional coat. A razor blade is useful to start the peel. Lay the blade nearly flat and fillet the edge of the film to create a tab that can be pulled. For surfaces that the gel adheres to well, such as concrete, 12" – 24" strips can be cut in the film resulting in less force being required to peel the film.

- Let DeconGel dry for 24 hours

Dry time will vary depending on humidity, temperature, air flow and thickness of the DeconGel. This can take from as little time as an hour for thin coats in a dry environment with plenty of airflow, to overnight or longer if thicker coats are applied in humid environments. Dry times exceeding 24 hours may sometimes be required for good peel performance on bare concrete, wood and other highly porous substrates and substrates with deep cracks or grooves. However, 18-24 hrs is often sufficient dry time on good quality concrete. It is recommended that users test a small area to determine drying time prior to applying DeconGel for an entire job. Supplemental heat or air circulation will accelerate DeconGel's drying time for any job.

- Peel DeconGel off the surface by starting from one of the edges

When dry, the product locks the contaminants into a polymer matrix. The film containing the encapsulated contamination can then be peeled. DeconGel peels from most non-porous and porous hard surfaces if the dried film is thick enough. If the film is difficult to peel, add another coat, let dry, and peel. In most cases the DeconGel will come off in a single sheet but for odd shaped surfaces you may be required to score DeconGel in order to be able to peel it off.

- Dispose of the dried DeconGel in accordance with the local, state and Federal disposal regulations of the contaminant/substance you are removing. DeconGel itself has no special disposal restrictions.

For more information on application techniques visit our website: www.decongel.com

6.32. End-User Report for Arsenic (Arsenic Trioxide)

TITLE: Surface Decontamination of Arsenic Compounds (Arsenic Trioxide (As_2O_3)) by DeconGel 1101

AUTHOR: Andreas Mylonakis PhD, Senior Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with Arsenic (Arsenic Trioxide (As_2O_3)) using ICP-OES (Inductively Coupled Plasma-Optical Emission Spectroscopy) following Environmental Protection Agency (EPA) SW-846 Methods 3005A (sampling) and 6010C (analysis).

HAZARDOUS MATERIALS RELEVANCE: Arsenic compounds are used as insecticides and are used to make semiconductors. Arsenic compounds are inhalation hazards and are toxic if ingested. Arsenic Trioxide was chosen as a representative arsenic compound for evaluating DeconGel's efficacy; DeconGel is expected to have similar efficacy towards the wide range of arsenic compounds.

SUMMARY RESULTS:

- Excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with arsenic (arsenic trioxide) resulting in encapsulation of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 99.8% (on concrete) to 99.3% (on carbon steel) to 99.6% (on stainless steel) as determined by swipe analysis.
- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of inorganic compounds in aqueous samples. When necessary, experimental methods were customized to afford complete dissolution of inorganic contaminants, and to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Table 1 shows the decontamination efficacies of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with arsenic trioxide as determined by residual swipe testing.

Table 1. Decontamination efficacy of DeconGel 1101 on As_2O_3 contaminated stainless steel, carbon steel, and concrete surfaces as determined by the residue swipe testing method.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	513.0 ± 37.4
	Residual	1.8 ± 1.3
	Decon. Efficacy (%)	**99.6 ± 7.8**
Carbon Steel	Control	539.4 ± 20.8
	Residual	3.8 ± 3.0
	Decon. Efficacy (%)	**99.3 ± 6.7**
Concrete	Control	487.7 ± 18.0
	Residual	1.1 ± 0.6
	Decon. Efficacy (%)	**99.8 ± 4.0**

2280x dilution factor for samples and controls

NOTES:

- ASTM method E1728-03 (a standardized swipe testing method used for the sampling of inorganic contaminants) was the integral method used to accurately evaluate DeconGel's decontamination efficacy. GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes (pre-wetted with DI H_2O) were utilized in this swipe testing method.

- Standardized EPA SW-846 Sampling Method 3005A "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy" was followed as a guideline to prepare all samples and controls. Because potassium ferricyanide reacts violently with acid releasing poisonous cyanide gas, all samples were digested solely in DI H2O (≥ 17 M-Ohm). To reduce the viscosity of samples, a 1:100 dilution in water took place on all samples and controls after ample soaking and vigorous shaking to incorporate any contaminant contained within swipes and to ensure full dissolution of the gel. All samples, controls, and standards were prepared using the same dissolution solution and experimental conditions to ensure both correct instrument calibration and accurate analytical results.

- To ensure accurate determination of DeconGel decontamination efficacy, calibration standards of the analyte of interest were prepared using either a sufficiently pure analyte or an appropriate ICP-MS Standard (Ricca Chemical Company; Arlington, TX); the respective standards were diluted to a known concentration (ppm) using the same digestion method as that used for samples and controls. Instrument blank controls were DI H_2O (≥17 M-Ohm).

- ICP-OES instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of inorganic compounds. Standardized EPA SW-846 Analytical Method 6010C "Inductively Coupled Plasma-Atomic Emission Spectrometry" was followed as a guideline to prepare all samples and controls. To ensure accurate determination of DeconGel decontamination efficacy, calibration standards of the analyte of interest were prepared using either a sufficiently pure analyte or an appropriate ICP-MS Standard (Ricca Chemical Company; Arlington, TX); the respective standards were diluted to a known concentration (ppm) using the same digestion method as the one used for samples and controls. Instrument blank controls were DI H_2O ($\geq$17 M-Ohm).

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.05 g of contaminant was evenly applied on 1) stainless steel (commercial grade, surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24-48 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipes were suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% deionized (DI) H_2O) for 24 hours and analyzed via ICP-OES (see below).

Controls Methods

For Swipe Controls, 0.05 g As_2O_3 was evenly applied on 1) stainless steel (commercial grade, surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 100 mL aqueous acidic solution (15% HCl, 15% HNO_3, 70% DI H_2O) for 24 h and analyzed via ICP-OES (see below).

Reagents and Standards

Reagent grade Arsenic Trioxide, As_2O_3, (CAS# 1327-53-3, Fisher Scientific; Fair Lawn, NJ), was used as received.

A 1000 ppm calibration standard was prepared using reagent grade powdered As_2O_3 and freshly prepared aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O). DI H_2O ($\geq$ 17 M-Ohm) was used as the blank sample.

Analytical Instrumentation

A Thermo ICP-OES instrument model radial iCap 6300 was used to determine As_2O_3 concentration (ppm) of all samples and controls using a freshly prepared 1000 ppm calibration standard.

Analyte (arsenic) analyzed at 228.8 nm

Pump Speed: 0.5 mL/min

APPLICATION INSTRUCTIONS FOR END-USERS:

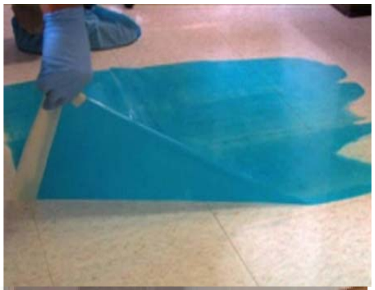
Use product directly as is from container. DO NOT DILUTE. Masking or painters tape can be applied along one edge of the area that is to be decontaminated to aid creating a peeled edge to grip for peeling the dried film. Apply DeconGel using a paint brush, a trowel, a handheld sprayer, or an industrial grade sprayer (use DeconGel 1120 or 1121 for spray application).

The thickness of the gel and the number of coats is dictated by the surface to be decontaminated. Coating thickness required for good peel characteristics varies with substrate and generally increases with substrate porosity. It is recommended that first time customers test DeconGel on a small sample area to confirm the required film thickness and dry time for their specific application. If the film is difficult to peel, please apply an additional coat. A razor blade is useful to start the peel. Lay the blade nearly flat and fillet the edge of the film to create a tab that can be pulled. For surfaces that the gel adheres to well, such as concrete, 12" – 24" strips can be cut in the film resulting in less force being required to peel the film.

- Let DeconGel dry for 24 hours

Dry time will vary depending on humidity, temperature, air flow and thickness of the DeconGel. This can take from as little time as an hour for thin coats in a dry environment with plenty of airflow, to overnight or longer if thicker coats are applied in humid environments. Dry times exceeding 24 hours may sometimes be required for good peel performance on bare concrete, wood and other highly porous substrates and substrates with deep cracks or grooves. However, 18-24 hrs is often sufficient dry time on good quality concrete. It is recommended that users test a small area to determine drying time prior to applying DeconGel for an entire job. Supplemental heat or air circulation will accelerate DeconGel's drying time for any job.

- Peel DeconGel off the surface by starting from one of the edges



When dry, the product locks the contaminants into a polymer matrix. The film containing the encapsulated contamination can then be peeled. DeconGel peels from most non-porous and porous hard surfaces if the dried film is thick enough. If the film is difficult to peel, add another coat, let dry, and peel. In most cases the DeconGel will come off in a single sheet but for odd shaped

surfaces you may be required to score DeconGel in order to be able to peel it off.

- Dispose of the dried DeconGel in accordance with the local, state and Federal disposal regulations of the contaminant/substance you are removing. DeconGel itself has no special disposal restrictions.

For more information on application techniques visit our website: www.decongel.com

6.33. End-User Report for Barium Compounds (Barium Chloride, Barium Carbonate)

TITLE: Surface Decontamination of Barium Compounds (Barium Chloride ($BaCl_2$) and Barium Carbonate ($BaCO_3$)) by DeconGel 1101

AUTHOR: Andreas Mylonakis PhD, Senior Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel 1101 on stainless steel, aluminum, carbon steel, and concrete surfaces contaminated with Barium Compounds (barium chloride ($BaCl_2$) and barium carbonate ($BaCO_3$)) using ICP-OES (Inductively Coupled Plasma-Optical Emission Spectroscopy) following Environmental Protection Agency (EPA) SW-846 Methods 3005A (sampling) and 6010C (analysis).

HAZARDOUS MATERIALS RELEVANCE: Barium and its compounds are utilized in the electronics industry as well as in the production of steel, fireworks, and pigments. Barium chloride is a water soluble salt of barium and is considered a severe health hazard and a poison. Barium carbonate is classified as an A4 carcinogen by ACGIH[7]. Barium and its compounds are considered hazardous and can produce adverse health effects in the case of skin contact, inhalation, or ingestion. Barium chloride and barium carbonate were chosen as representative barium compounds for evaluating DeconGel's efficacy; DeconGel is expected to have similar efficacy towards the wide range of barium compounds.

HIGHLIGHTS:

- Excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with barium compounds facilitating encapsulation/emulsification of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 98.6% and 90.1% (on concrete) to 99.8% and 96.5% (on carbon steel) to 99.3% and 95.0% (on stainless steel) as determined by residual swipe analysis.

- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of inorganic compounds in aqueous samples. When deemed necessary, experimental methods were customized to afford complete dissolution of inorganic contaminants, and to ensure accurate decontamination efficacy determination of DeconGel.

---

[7] American Conference of Industrial Hygienists (ACGIH); http://www.acgih.org/home.htm (2010)

RESULTS: Tables 1 and 2 show the decontamination efficacies of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with barium chloride and barium carbonate as determined by residual swipe testing.

Table 1. Decontamination efficacies of DeconGel 1101 against Barium Chloride ($BaCl_2$) contaminated surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		**Formulation**
		DeconGel 1101
Stainless Steel	Control	5.5 ± 0.2
	Residual	0.035 ± 0.02
	Decon. Efficacy (%)	**99.3 ± 0.3**
Carbon Steel	Control	5.63 ± 0.27
	Residual	0.014 ± 0.01
	Decon. Efficacy (%)	**99.8 ± 5.5**
Concrete	Control	8.69 ± 0.04
	Residual	0.12 ± 0.02
	Decon. Efficacy (%)	**98.6 ± 1.9**

228000x dilution factor for samples and controls

Table 2. Decontamination efficacies of DeconGel 1101 against barium carbonate ($BaCO_3$) contaminated surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation DeconGel 1101
Stainless Steel	Control	5.31 ± 0.13
	Residual	0.26 ± 0.06
	Decon. Efficacy (%)	**95.0 ± 2.8**
Carbon Steel	Control	5.15 ± 0.42
	Residual	0.18 ± 0.06
	Decon. Efficacy (%)	**96.5 ± 8.1**
Concrete	Control	4.95 ± 0.21
	Residual	0.49 ± 0.07
	Decon. Efficacy (%)	**90.1 ± 4.1**

228000x dilution factor for samples and controls

NOTES:

- ASTM method E1728-03, a standardized swipe testing method used for sampling of inorganic contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes (pre-wetted with DI H_2O) were utilized in this swipe testing method.

- Standardized EPA SW-846 Sampling Method 3005A "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy" was followed as a guideline to prepare all samples and controls. When necessary, the digestion methods were customized by increasing hydrochloric and nitric acid concentrations from 25% to 35% wt, and/or by heating samples to higher temperatures using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC) for the complete digestion/dissolution of the inorganic contaminants. All samples, controls, and standards were prepared using the same dissolution solution and experimental conditions to ensure both correct instrument calibration and accurate analytical results.

- To ensure accurate determination of DeconGel decontamination efficacy, calibration standards of the analyte of interest were prepared using either a sufficiently pure analyte or an appropriate ICP-MS Standard (Ricca Chemical Company; Arlington, TX); the respective standards were diluted to a known concentration (ppm) using the same digestion method as the one used for samples and controls. Instrument blank controls were DI H_2O ($\geq$17 M-Ohm).

- ICP-OES instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of inorganic compounds. Standardized EPA SW-846 Analytical Method 6010C “Inductively Coupled Plasma-Atomic Emission Spectrometry” was followed as a guideline to prepare all samples and controls.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.05 g of contaminant was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24-48 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% deionized (DI) H_2O) for 24 h. When deemed necessary, samples were heated to 94 ºC for 4-14 h to effectively complete dissolution of inorganics using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC). Samples were allowed to cool to room temperature, diluted 1:100 with DI H_2O and were then analyzed via ICP-OES (see below).

Controls Methods

For Swipe Controls samples, 0.05 g of contaminant was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O) for 24 h, diluted 1:100 with DI H_2O and analyzed via ICP-OES (see below).

Reagents and Standards

Barium Chloride Dihydrate, $BaCl_2 \cdot 2H_2O$, (CAS# 10326-27-9, Fisher Scientific; Fair Lawn, NJ), was used as received.

Barium Carbonate, $BaCO_3$, (CAS# 513-77-9 Fisher Scientific; Fair Lawn, NJ), was used as received.

A 10.0 ppm calibration standard of each contaminant was prepared using the reagent grade barium compounds and freshly prepared aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O). DI H_2O ($\geq$17 M-Ohm) was used as the blank sample.

Analytical Instrumentation

A Thermo ICP-OES instrument model radial iCap 6300 was used to determine barium concentration (ppm) of all samples and controls using a freshly prepared 10.0 ppm calibration standard.

Analyte (barium) analyzed at 455.4 nm

Pump Speed: 0.5 mL/min

APPLICATION INSTRUCTIONS FOR END-USERS:

Use product directly as is from container. DO NOT DILUTE. Masking or painters tape can be applied along one edge of the area that is to be decontaminated to aid creating a peeled edge to grip for peeling the dried film. Apply DeconGel using a paint brush, a trowel, a handheld sprayer, or an industrial grade sprayer (use DeconGel 1120 or 1121 for spray application).

The thickness of the gel and the number of coats is dictated by the surface to be decontaminated. Coating thickness required for good peel characteristics varies with substrate and generally increases with substrate porosity. It is recommended that first time customers test DeconGel on a small sample area to confirm the required film thickness and dry time for their specific application. If the film is difficult to peel, please apply an additional coat. A razor blade is useful to start the peel. Lay the blade nearly flat and fillet the edge of the film to create a tab that can be pulled. For surfaces that the gel adheres to well, such as concrete, 12” – 24” strips can be cut in the film resulting in less force being required to peel the film.

- Let DeconGel dry for 24 hours

Dry time will vary depending on humidity, temperature, air flow and thickness of the DeconGel. This can take from as little time as an hour for thin coats in a dry environment with plenty of airflow, to overnight or longer if thicker coats are applied in humid environments. Dry times exceeding 24 hours may sometimes be required for good peel performance on bare concrete, wood and other highly porous substrates and substrates with deep cracks or grooves. However, 18-24 hrs is often sufficient dry time on good quality concrete. It is recommended that users test a small area to determine drying time prior to applying DeconGel for an entire job. Supplemental heat or air circulation will accelerate DeconGel’s drying time for any job.

- Peel DeconGel off the surface by starting from one of the edges

When dry, the product locks the contaminants into a polymer matrix. The film containing the encapsulated contamination can then be peeled. DeconGel peels from most non-porous and porous hard surfaces if the dried film is thick enough. If the film is difficult to peel, add another coat, let dry, and peel. In most cases the DeconGel will come off in a single sheet but for odd shaped surfaces you may be required to score DeconGel in order to be able to peel it off.

- Dispose of the dried DeconGel in accordance with the local, state and Federal disposal regulations of the contaminant/substance you are removing. DeconGel itself has no special disposal restrictions.

For more information on application techniques visit our website: www.decongel.com

6.34. End-User Report for Beryllium Compounds (Aluminum powder, Aluminum Oxide)

TITLE: Surface Decontamination of Beryllium Compounds Utilizing Beryllium Surrogates (Elemental Aluminum Powder (Al) and Aluminum Oxide (Al_2O_3)) by DeconGel 1101

AUTHOR: Andreas Mylonakis PhD, Senior Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with Beryllium Surrogate Compounds (aluminum powder (Al) and aluminum oxide (Al_2O_3)) using ICP-OES (Inductively Coupled Plasma-Optical Emission Spectroscopy) following Environmental Protection Agency (EPA) SW-846 Methods 3005A (sampling) and 6010C (analysis).

HAZARDOUS MATERIALS RELEVANCE: Beryllium compounds are used in the aerospace industry to construct lightweight and resilient alloys. Beryllium is directly corrosive to living tissue; beryllium dust is toxic by inhalation and can cause berylliosis, an incurable chronic lung disease. Due to beryllium toxicity, the commonly used beryllium surrogate aluminum was used in this study. Aluminum powder and aluminum oxide were chosen as representative beryllium surrogate compounds for evaluating DeconGel's efficacy; DeconGel is expected to have similar efficacy towards the wide range of beryllium surrogate compounds.

SUMMARY RESULTS:

- Excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with aluminum metal dust and aluminum oxide (surrogates for beryllium metal dust and beryllium oxide respectively) resulting in encapsulation of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 99.6% and 99.7% (on concrete) to 99.5% and 99.9% (on carbon steel) to 97.9% and 99.7% (on stainless steel) for aluminum metal dust and aluminum oxide respectively as determined by residual swipe analysis.

- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guid^{e}lines for determination of inorganic compounds in aqueous samples. When necessary, experimental methods were customized to afford complete dissolution of inorganic contaminants, and to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 and 2 show the decontamination efficacies of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with aluminum powder (Al) and aluminum oxide (Al_2O_3) as determined by residual swipe testing.

Table 1. Decontamination efficacies of DeconGel 1101 on aluminum powder (Al) contaminated stainless steel, carbon steel, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		**Formulation**
		DeconGel 1101
Stainless Steel	Control	1113.0 ± 17.0
	Residual	23.3 ± 5.3
	Decon. Efficacy (%)	**97.9 ± 2.0**
Carbon Steel	Control	944.7 ± 0.2
	Residual	4.7 ± 4.3
	Decon. Efficacy (%)	**99.5 ± 4.6**
Concrete	Control	511.7 ± 12.7
	Residual	1.8 ± 0.2
	Decon. Efficacy (%)	**99.6 ± 3.8**

2280x dilution factor for samples and controls

Table 2. Decontamination efficacies of DeconGel against aluminum oxide (Al_2O_3) contaminated stainless steel, carbon steel, and concrete surfaces as determined by residual swipe testing method.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	420.8 ± 7.0
	Residual	1.4 ± 0.6
	Decon. Efficacy (%)	**99.7 ± 7.7**
Carbon Steel	Control	505.5 ± 22.4
	Residual	0.3 ± 0.3
	Decon. Efficacy (%)	**99.9 ± 4.4**
Concrete	Control	420.2 ± 6.1
	Residual	1.3 ± 0.7
	Decon. Efficacy (%)	**99.7 ± 5.9**

2280x dilution factor for samples and controls

NOTES:

- ASTM method E1728-03, a standardized swipe testing method used for the sampling of inorganic contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes (pre-wetted with DI H_2O) were utilized in this swipe testing method.
- Standardized EPA SW-846 Sampling Method 3005A "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy" was followed as a guideline to prepare all samples and controls. When deemed necessary, digestion methods were customized by increasing hydrochloric and nitric acid concentrations from 25% to 35% wt, and/or by heating samples to higher temperatures using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC) to facilitate the complete digestion/dissolution of the inorganic contaminants. All samples, controls, and standards were prepared using the same dissolution solution and experimental conditions to ensure both correct instrument calibration and accurate analytical results.

- ICP-OES instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of inorganic compounds. Standardized EPA SW-846 Analytical Method 6010C "Inductively Coupled Plasma-Atomic Emission Spectrometry" was followed as a guideline to prepare all samples and controls.

- To ensure accurate determination of DeconGel decontamination efficacy, calibration standards of the analyte of interest were prepared using either a sufficiently pure analyte or an appropriate ICP-MS Standard (Ricca Chemical Company; Arlington, TX); the respective standards were diluted to a known concentration (ppm) using the same digestion method as the one used for samples and controls. Instrument blank controls were DI H_2O ($\geq$17 M-Ohm).

- Beryllium surrogates (aluminium metal powder (Al) and aluminium oxide (Al_2O_3) for beryllium metal powder (Be) and beryllium oxide (BeO) respectively) have been utilized in this evaluation studies due to the high toxicity and carcinogenicity of beryllium metal and beryllium compounds.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.05 g of contaminant was evenly applied on 1) stainless steel (commercial grade, surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24-48 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% deionized (DI) H_2O) for 24 h and were then analyzed via ICP-OES (see below).

Controls Methods

For Swipe Control samples, the respective amount of contaminant was evenly applied on 1) stainless steel (commercial grade, surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O) for 24 h and analyzed via ICP-OES (see below).

Reagents and Standards

Aluminium metal, Al, finest powder grade, (CAS# 7429-90-5, Fisher Scientific; Fair Lawn, NJ), was used as received.

Aluminium oxide, Al_2O_3, 60-325 Mesh, (CAS# 1344-28-1, Fisher Scientific; Fair Lawn, NJ), was used as received.

A 1000 ppm calibration standard of each contaminant was prepared using the beryllium surrogates and freshly prepared aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O). DI H_2O ($\geq$ 17 M-Ohm) was used as the blank sample.

Analytical Instrumentation

A Thermo ICP-OES instrument model radial iCap 6300 was used to determine aluminum concentration (ppm) of all samples and controls using a freshly prepared 1000 ppm calibration standard. Analyte (aluminum) analyzed at 308.2 nm. Pump Speed: 0.5 mL/min

APPLICATION INSTRUCTIONS FOR END-USERS:

Use product directly as is from container. DO NOT DILUTE. Masking or painters tape can be applied along one edge of the area that is to be decontaminated to aid creating a peeled edge to grip for peeling the dried film. Apply DeconGel using a paint brush, a trowel, a handheld sprayer, or an industrial grade sprayer (use DeconGel 1120 or 1121 for spray application).

The thickness of the gel and the number of coats is dictated by the surface to be decontaminated. Coating thickness required for good peel characteristics varies with substrate and generally increases with substrate porosity. It is recommended that first time customers test DeconGel on a small sample area to confirm the required film thickness and dry time for their specific application. If the film is difficult to peel, please apply an additional coat. A razor blade is useful to start the peel. Lay the blade nearly flat and fillet the edge of the film to create a tab that can be pulled. For surfaces that the gel adheres to well, such as concrete, 12" – 24" strips can be cut in the film resulting in less force being required to peel the film.

- Let DeconGel dry for 24 hours

Dry time will vary depending on humidity, temperature, air flow and thickness of the DeconGel. This can take from as little time as an hour for thin coats in a dry environment with plenty of airflow, to overnight or longer if thicker coats are applied in humid environments. Dry times exceeding 24 hours may sometimes be required for good peel performance on bare concrete, wood and other highly porous substrates and substrates with deep cracks or grooves. However, 18-24 hrs is often sufficient dry time on good quality concrete. It is recommended that users test a small area to determine drying time prior to applying DeconGel for an entire job. Supplemental heat or air circulation will accelerate DeconGel's drying time for any job.

- Peel DeconGel off the surface by starting from one of the edges

When dry, the product locks the contaminants into a polymer matrix. The film containing the encapsulated contamination can then be peeled. DeconGel peels from most non-porous and porous hard surfaces if the dried film is thick enough. If the film is difficult to peel, add another coat, let dry, and peel. In most cases the DeconGel will come off in a single sheet but for odd shaped surfaces you may be required to score DeconGel in order to be able to peel it off.

- Dispose of the dried DeconGel in accordance with the local, state and Federal disposal regulations of the contaminant/substance you are removing. DeconGel itself has no special disposal restrictions.

For more information on application techniques visit our website: www.decongel.com

6.35. End-User Report for Cadmium Compounds (Cadmium Chloride, Cadmium Oxide)

TITLE: Surface Decontamination of Cadmium Compounds (Cadmium Chloride ($CdCl_2$) and Cadmium Oxide (CdO)) by DeconGel 1101

AUTHOR: Andreas Mylonakis PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Determine the surface decontamination efficacy of DeconGel 1101 on stainless steel, aluminum, carbon steel, and concrete surfaces contaminated with cadmium compounds (cadmium chloride ($CdCl_2$) and cadmium oxide (CdO)) using ICP-OES (Inductively Coupled Plasma-Optical Emission Spectroscopy) following Environmental Protection Agency (EPA) SW-846 Methods 3005A (sampling) and 6010C (analysis).

HAZARDOUS MATERIALS RELEVANCE: Cadmium metal and cadmium compounds are highly toxic. Cadmium chloride is often used in the process of making cadmium sulfide, a common yellow pigment while cadmium oxide is a conductive material used in the making of photovoltaic cells and photodiodes. Cadmium compounds are known carcinogens (classified A2 by ACGIH[8]) and fatal inhalation hazards. It is important to contain and remove cadmium particles which can become airborne. Cadmium chloride and cadmium oxide were chosen as representative cadmium compounds for evaluating DeconGel's efficacy; DeconGel is expected to have similar efficacy towards the wide range of cadmium compounds.

HIGHLIGHTS:

- Excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with cadmium (cadmium chloride and cadmium oxide) resulting in encapsulation of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 99.5% and 100% (on stainless steel), to 98.5% and 100% (carbon steel) to 97.6% and 98.4% (on concrete) as determined by residue swipe analysis.

- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of inorganic compounds in aqueous samples. When deemed necessary, experimental methods were customized to afford complete dissolution of inorganic contaminants, and to ensure accurate decontamination efficacy determination of DeconGel.

---

[8] A2: suspected human carcinogen; American Conference of Industrial Hygienists (ACGIH); http://www.acgih.org/home.htm (2010)

RESULTS: Tables 1 and 2 show the decontamination efficacies of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with cadmium chloride and cadmium oxide as determined by residual swipe testing.

Table 1. Decontamination efficacies of DeconGel 1101 against Cadmium Chloride ($CdCl_2$) contaminated stainless steel, carbon steel, and concrete surfaces as determined by residual swipe testing method.

Swipe Testing (ppm)		**Formulation**
		DeconGel 1101
Stainless Steel	Control	464.7 ± 7.0
	Residual	2.25 ± 1.81
	Decon. Efficacy (%)	**99.5 ± 1.7**
Carbon Steel	Control	521.1 ± 21.8
	Residual	7.81 ± 1.17
	Decon. Efficacy (%)	**98.5 ± 4.2**
Concrete	Control	483.0 ± 2.5
	Residual	11.63 ± 7.80
	Decon. Efficacy (%)	**97.6 ± 2.2**

2280x dilution factor for samples and controls

Table 2. Decontamination efficacies of DeconGel against Cadmium Oxide (CdO) contaminated stainless steel, carbon steel, and concrete surfaces as determined by residual swipe testing method.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	553.4 ± 33.2
	Residual	0.086 ± 0.075
	Decon. Efficacy (%)	**100.0 ± 0.2**
Carbon Steel	Control	578.8 ± 14.3
	Residual	0.204 ± 0.104
	Decon. Efficacy (%)	**100.0 ± 0.4**
Concrete	Control	543.2 ± 22.4
	Residual	8.52 ± 7.23
	Decon. Efficacy (%)	**98.4 ± 0.2**

2280x dilution factor for samples and controls

NOTES:

- ASTM method E1728-03, a standardized swipe testing method used for sampling inorganic contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes (pre-wetted with DI H_2O) were utilized in this swipe testing method.

- Standardized EPA SW-846 Sampling Method 3005A "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy" was followed as a guideline to prepare all samples and controls. When necessary, digestion methods were customized by increasing hydrochloric and nitric acid concentrations from 25% to 35% wt, and/or by heating samples to higher temperatures using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC) to afford the complete digestion/dissolution of the inorganic contaminants. All samples, controls, and standards were prepared using the same dissolution solution and experimental conditions to ensure both correct instrument calibration and accurate analytical results.

- To ensure accurate determination of DeconGel decontamination efficacy, calibration standards of the analyte of interest were prepared using either a sufficiently pure analyte or an appropriate ICP-MS Standard (Ricca Chemical Company; Arlington, TX); the respective standards were diluted to a known concentration (ppm) using the same digestion method as the one used for samples and controls. Instrument blank controls were DI H_2O ($\geq$17 M-Ohm).

- ICP-OES instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of inorganic compounds. Standardized EPA SW-846 Analytical Method 6010C “Inductively Coupled Plasma-Atomic Emission Spectrometry” was followed as a guideline to prepare all samples and controls.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.05 g of contaminant was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24-48 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipes were suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% deionized (DI) H_2O) for 24 h. When deemed necessary, samples were heated to 94 ^oC for 4-14 h to effectively complete dissolution of inorganics using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC). Samples were allowed to cool to room temperature and were then analyzed via ICP-OES (see below).

Controls Methods

For Swipe Control samples, 0.05 g of contaminant was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O) for 24 h and analyzed via ICP-OES (see below).

Reagents and Standards

Reagent grade Cadmium Chloride, $CdCl_2$, (CAS # 7790-78-5, Fisher Scientific; Fair Lawn, NJ), was used as received.

Reagant grade Cadmium Oxide, CdO, (CAS# 1306-19-0, Fisher Scientific; Fair Lawn, NJ), was used as received

A 1000 ppm calibration standard of each contaminant was prepared using the reagent grade cadmium compounds and freshly prepared aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O). DI H_2O was used as the blank sample.

Analytical Instrumentation

A Thermo ICP-OES instrument model radial iCap 6300 was used to determine cadmium concentration (ppm) of all samples and controls using a freshly prepared 1000 ppm calibration standard.

Analyte (cadmium) analyzed at 228.8 nm

Pump Speed: 0.5 mL/min

APPLICATION INSTRUCTIONS FOR END-USERS:

Use product directly as is from container. DO NOT DILUTE. Masking or painters tape can be applied along one edge of the area that is to be decontaminated to aid creating a peeled edge to grip for peeling the dried film. Apply DeconGel using a paint brush, a trowel, a handheld sprayer, or an industrial grade sprayer (use DeconGel 1120 or 1121 for spray application).

The thickness of the gel and the number of coats is dictated by the surface to be decontaminated. Coating thickness required for good peel characteristics varies with substrate and generally increases with substrate porosity. It is recommended that first time customers test DeconGel on a small sample area to confirm the required film thickness and dry time for their specific application. If the film is difficult to peel, please apply an additional coat. A razor blade is useful to start the peel. Lay the blade nearly flat and fillet the edge of the film to create a tab that can be pulled. For surfaces that the gel adheres to well, such as concrete, 12" – 24" strips can be cut in the film resulting in less force being required to peel the film.

- Let DeconGel dry for 24 hours

Dry time will vary depending on humidity, temperature, air flow and thickness of the DeconGel. This can take from as little time as an hour for thin coats in a dry environment with plenty of airflow, to overnight or longer if thicker coats are applied in humid environments. Dry times exceeding 24 hours may sometimes be required for good peel performance on bare concrete, wood and other highly porous substrates and substrates with deep cracks or grooves. However, 18-24 hrs is often sufficient dry time on good quality concrete. It is recommended that users test a small area to determine drying time prior to applying DeconGel for an entire job. Supplemental heat or air circulation will accelerate DeconGel's drying time for any job.

- Peel DeconGel off the surface by starting from one of the edges

When dry, the product locks the contaminants into a polymer matrix. The film containing the encapsulated contamination can then be peeled. DeconGel peels from most non-porous and porous hard surfaces if the dried film is thick enough. If the film is difficult to peel, add another coat, let dry, and peel. In most cases the DeconGel will come off in a single sheet but for odd shaped surfaces you may be required to score DeconGel in order to be able to peel it off.

- Dispose of the dried DeconGel in accordance with the local, state and Federal disposal regulations of the contaminant/substance you are removing. DeconGel itself has no special disposal restrictions.

For more information on application techniques visit our website: www.decongel.com

6.36. End-User Report for Cyanide Compounds (Potassium Ferricyanide)

TITLE: Surface Decontamination of Cyanide Compounds (Potassium Ferricyanide ($K_3[Fe(CN)_6]$)) by DeconGel 1101

AUTHOR: Andreas Mylonakis PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with potassium ferricyanide using ICP-OES (Inductively Coupled Plasma-Optical Emission Spectroscopy) following Environmental Protection Agency (EPA) SW-846 Methods 3005A (sampling) and 6010C (analysis).

HAZARDOUS MATERIALS RELEVANCE: Cyanide can cause acute toxicity through skin contact, ingestion, and inhalation. Potassium ferricyanide ($K_3[Fe(CN)_6]$) is an iron-containing compound possessing similar physical characteristics to potassium cyanide (KCN), a water soluble form of cyanide. Cyanide is utilized in gold and silver mining and electroplating industrial applications. Both ACGIH[9] and NIOSH[10] have set maximum exposure limits for cyanide compounds. Potassium ferricyanide was chosen as a representative cyanide compound for evaluating DeconGel's efficacy; DeconGel is expected to have similar efficacy towards the wide range of cyanide compounds.

HIGHLIGHTS:

- Excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with cyanide compounds resulting in encapsulation of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 98.2% (on concrete) to 98.9% (on carbon steel) to 99.4% (on stainless steel) as determined by swipe analysis.

- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of inorganic compounds in aqueous samples. When necessary, experimental methods were customized to afford complete dissolution of inorganic contaminants, and to ensure accurate decontamination efficacy determination of DeconGel.

---

[9] American Conference of Industrial Hygienists (ACGIH); http://www.acgih.org/home.htm (2010)
[10] National Institute for Occupational Safety and Health (NIOSH); http://www.cdc.gov/niosh/ (2010)

RESULTS: Table 1 and shows the decontamination efficacies of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with cyanide as determined by residual swipe testing.

Table 1. Decontamination efficacies of DeconGel against potassium ferricyanide contaminated stainless steel, carbon steel, and concrete surfaces as determined by residue swipe testing method.

Swipe Testing (ppm)		**Formulation**
		DeconGel 1101
Stainless Steel	Control	3.31 ± 0.01
	Residual	0.020 ± 0.007
	Decon. Efficacy (%)	**99.4 ± 4.2**
Carbon Steel	Control	3.45 ± 0.06
	Residual	0.036 ± 0.018
	Decon. Efficacy (%)	**98.9 ± 3.4**
Concrete	Control	3.13 ± 0.07
	Residual	0.057 ± 0.045
	Decon. Efficacy (%)	**98.2 ± 3.2**

1400000x dilution factor for samples and controls

NOTES:

- ASTM method E1728-03 (a standardized swipe testing method used for the sampling of inorganic contaminants) was the integral method used to accurately evaluate DeconGel's decontamination efficacy. GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes (pre-wetted with DI H_2O) were utilized in this swipe testing method.

- Standardized EPA SW-846 Sampling Method 3005A "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy" was followed as a guideline to prepare all samples and controls. Because potassium ferricyanide reacts violently with acid releasing poisonous cyanide gas, all samples were digested solely in DI H2O (≥ 17 M-Ohm). To reduce the viscosity of samples, a 1:100 dilution in water took place on all samples and controls after ample soaking and vigorous shaking to incorporate any contaminant contained within swipes and to ensure full dissolution of the gel. All samples, controls, and standards were prepared using the same dissolution solution and experimental conditions to ensure both correct instrument calibration and accurate analytical results.

- To ensure accurate determination of DeconGel decontamination efficacy, calibration standards of the analyte of interest were prepared using either a sufficiently pure analyte or an appropriate ICP-MS Standard (Ricca

Chemical Company; Arlington, TX); the respective standards were diluted to a known concentration (ppm) using the same digestion method as that used for samples and controls. Instrument blank controls were DI H_2O ($\geq$17 M-Ohm).

- ICP-OES instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of inorganic compounds. Standardized EPA SW-846 Analytical Method 6010C "Inductively Coupled Plasma-Atomic Emission Spectrometry" was followed as a guideline to prepare all samples and controls.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.05 g of contaminant was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24-48 hours. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipes were suspended in 100 mL of DI H_2O and shaken vigorously. After 24 hours, the samples were shaken again prior to a 1:100 dilution in DI H_2O. The samples were then shaken again and analyzed via ICP-OES (see below).

Controls Methods

For Swipe Control samples, 0.05 g of contaminant was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 100 mL DI H_2O and shaken vigorously. After 24 hours, the samples were shaken again prior to a 1:100 dilution in DI H_2O. The control samples were then shaken again and analyzed via ICP-OES.

Reagents and Standards

Potassium ferricyanide, ($K_3[Fe(CN)_6]$), (CAS# 13746-66-2, Fisher Scientific; Fair Lawn, NJ), was used as received.

A 10.0 ppm calibration standard was prepared by dilution of iron ICP-MS Standard (Ricca Chemical Company; Arlington, TX) to the proper concentration. DI H_2O ($\geq$ 17 M-Ohm) was used as the blank sample.

Analytical Instrumentation

A Thermo ICP-OES instrument model radial iCap 6300 was used to determine cyanide concentration (ppm) of all samples and controls using a freshly prepared 1000 ppm calibration standard. Analyte (iron) was analyzed at 271.4 nm Pump Speed: 0.5 mL/min

APPLICATION INSTRUCTIONS FOR END-USERS:

Use product directly as is from container. DO NOT DILUTE. Masking or painters tape can be applied along one edge of the area that is to be decontaminated to aid creating a peeled edge to grip for peeling the dried film. Apply DeconGel using a paint brush, a trowel, a handheld sprayer, or an industrial grade sprayer (use DeconGel 1120 or 1121 for spray application).

The thickness of the gel and the number of coats is dictated by the surface to be decontaminated. Coating thickness required for good peel characteristics varies with substrate and generally increases with substrate porosity. It is recommended that first time customers test DeconGel on a small sample area to confirm the required film thickness and dry time for their specific application. If the film is difficult to peel, please apply an additional coat. A razor blade is useful to start the peel. Lay the blade nearly flat and fillet the edge of the film to create a tab that can be pulled. For surfaces that the gel adheres to well, such as concrete, 12” – 24” strips can be cut in the film resulting in less force being required to peel the film.

- Let DeconGel dry for 24 hours

Dry time will vary depending on humidity, temperature, air flow and thickness of the DeconGel. This can take from as little time as an hour for thin coats in a dry environment with plenty of airflow, to overnight or longer if thicker coats are applied in humid environments. Dry times exceeding 24 hours may sometimes be required for good peel performance on bare concrete, wood and other highly porous substrates and substrates with deep cracks or grooves. However, 18-24 hrs is often sufficient dry time on good quality concrete. It is recommended that users test a small area to determine drying time prior to applying DeconGel for an entire job. Supplemental heat or air circulation will accelerate DeconGel’s drying time for any job.

- Peel DeconGel off the surface by starting from one of the edges

When dry, the product locks the contaminants into a polymer matrix. The film containing the encapsulated contamination can then be peeled. DeconGel peels from most non-porous and porous hard surfaces if the dried film is thick enough. If the film is difficult to peel, add another coat, let dry, and peel. In most cases the DeconGel will come off in a single sheet but for odd shaped surfaces you may be required to score DeconGel in order to be able to peel it off.

- Dispose of the dried DeconGel in accordance with the local, state and Federal disposal regulations of the contaminant/substance you are removing. DeconGel itself has no special disposal restrictions.

For more information on application techniques visit our website:
www.decongel.com

6.37. End-User Report for Halogenated Solvents (Tetrachloroethylene)

TITLE: Surface Decontamination of Halogenated Solvents (Tetrachloroethylene) by DeconGel 1101 and 1102

ANALYST: Geoff Nadolski PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel formulation 1101 and 1102 on stainless steel, floor tile, and concrete surfaces contaminated with Halogenated Solvents (tetrachloroethylene (TCE)) using LC/MS (Liquid Chromatography/Mass Spectrometry) following Environmental Protection Agency (EPA) SW-846 Methods 3500C (Sampling) and 8321B (Analysis).

HAZARDOUS MATERIALS RELEVANCE: TCE is a volatile chlorinated hydrocarbon widely used as a solvent, dry-cleaning fluid, and degreaser. TCE is a common soil contaminant and is an environmentally persistent pollutant. TCE is classified as a carcinogen, and is a skin irritant and central nervous system depressant. TCE was chosen as a representative halogenated solvent; DeconGel is expected to have similar efficacy towards the wide range of halogenated solvents.

SUMMARY RESULTS:

- Excellent surface decontamination was achieved by applying both DeconGel 1101 and 1102, both via brushing or pouring (non-brushed) onto contaminated surfaces, resulting in encapsulation of TCE contaminant by DeconGel's active components. Decontamination efficacies of poured DeconGel 1101 ranged from 100% (on concrete) to 100% (on floor tile) to 99.6% (on stainless steel), brushed DeconGel 1101 ranged from 100% (on concrete) to 100% (on floor tile) to 100% (on stainless steel), as determined by residual swipe analysis. Decontamination efficacies of poured DeconGel 1102 ranged from 100% (on concrete) to 100% (on floor tile) to 99.5% (on stainless steel), brushed DeconGel 1102 ranged from 100% (on concrete) to 100% (on floor tile) to 100% (on stainless steel), as determined by residual swipe analysis.
- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of organic compounds dissolved in a suitable solvent able to completely solvate TCE as well as DeconGel components. When deemed necessary, experimental methods were customized to afford complete dissolution of organic contaminants. Additionally, analytical methods and

associated equipment (LC column, LC gradient program, MS sample ionization parameters) were appropriately developed to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 shows the decontamination efficacies of DeconGel 1101 and 1102 on stainless steel, floor tile, and concrete surfaces contaminated with Halogentated Solvents (TCE) as determined by residual swipe testing.

Table 1. Decontamination efficacies of DeconGel 1101 and 1102 on TCE Halogenated Solvent contaminated stainless steel, floor tile, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		**Formulation**	
		DeconGel 1101	**DeconGel 1102**
Stainless Steel	Control	936.24 ± 2.55	936.24 ± 2.55
	Residual (non-brushed)	4.20 ± 0.05	5.02 ± 0.05
	Residual (brushed)	ND*	ND
	Decon. Efficacy (non-brushed) (%)	**99.6 ± 0.01**	**99.5 ± 0.0**
	Decon. Efficacy (brushed) (%)	**100****	**100****
Floor Tile	Control	934.98 ± 1.52	934.98 ± 1.52
	Residual (non-brushed)	ND	ND
	Residual (brushed)	ND	ND
	Decon. Efficacy (non-brushed) (%)	**100****	**100****
	Decon. Efficacy (brushed) (%)	**100****	**100****
Concrete	Control	813.67 ± 2.92	813.67 ± 2.92
	Residual (non-brushed)	ND	ND
	Residual (brushed)	ND	ND
	Decon. Efficacy (non-brushed) (%)	**100****	**100****
	Decon. Efficacy (brushed) (%)	**100****	**100****

678x dilution factor for samples and controls

* ND: not detected, limit of detection (LOD) for TCE approximates 250 ppb

** An ND value for residual experiments results in a Decontamination Efficacy of 100%

NOTES:

- ASTM method E1728-03, a standardized swipe testing method used for sampling of contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. Air-dried GhostWipe™

(Environmental Express; Mt. Pleasant, SC) swipes were utilized in this swipe testing method.

- Standardized EPA SW-846 Sampling Method 3500C “Organic Extraction and Sample Preparation” was followed as a guideline to prepare all samples and controls. All samples, controls, and standards were prepared using the same solvent and appropriate experimental conditions to ensure accurate and LC/MS instrumental analysis.
- LC/MS instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of organic compounds. Standardized EPA SW-846 Analytical Method 8321B “Solvent-Extractable Nonvolatile Compounds by High-Performance Liquid Chromatography/Thermospray/Mass Spectrometry (HPLC/TS/MS) or Ultraviolet (UV) Detection” was followed as a guideline to prepare all samples and controls.
- To ensure accurate determination of DeconGel decontamination efficacy, a standard curve of the analyte of interest was prepared using sufficiently pure analyte; the respective standards were diluted to a known concentration (ppm) using the same solvent as used for samples and controls.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 50 uL of contaminant was evenly applied via brushing on 1) stainless steel (surface area: 56.3 cm^2), 2) floor tile (surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe and gel samples were suspended in 50 mL DMSO for 24 h. Samples were then analyzed via LC/MS (see below).

Controls Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied via brushing on 1) stainless steel (surface area: 56.3 cm^2), 2) floor tile (surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 50 mL DMSO for 24 h and analyzed via LC/MS (see below).

Reagents and Standards

Tetrachloroethylene (TCE) (tetrachloroethene) (CAS# 127-18-4, Fisher Scientific; Fair Lawn, NJ) was used as received.

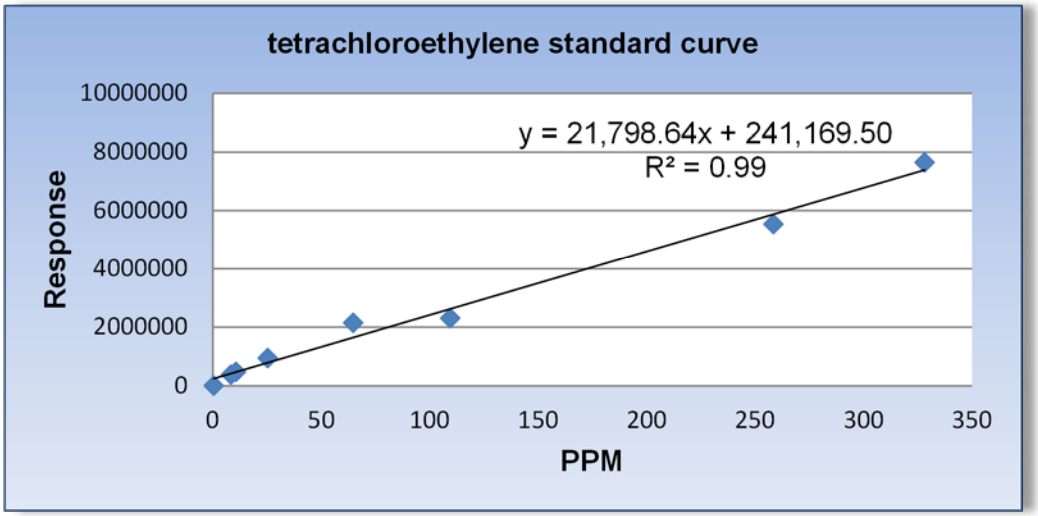
Analytical Instrumentation

A Thermo LCQ LC/MS with autosampler in positive mode was used to determine toluene concentration (ppm, wt/wt) of all samples and controls, using a Grace Davison (Deerfield, IL) C18-Select analytical column (150 x 4.6 mm, 5 um).

A 9-point standard curve derived from three independently prepared stock solutions was prepared using DMSO as the working solvent. The calibration curve exhibits a curve fitting as approximated by the coefficient of determination of linear regression R^2, where $R^2 = 0.99$ (see Figure 1).

LC method using A= water (0.1% formic acid), B=acetonitrile (0.1% formic acid); start at 95%A to 3 min at 70%A, to 6 min at 50%A, to 12 min at 30%A, to 15 min at 90%B, to 16 min at 100%B, hold until 22 min at 100% B, to 23 min at 95%A, hold until 24 min. TCE LC/MS data: 15.4 min; lambda max = 238 nm; $M^+ = 165$.

Figure 1. **TCE Standard Calibration Curve**



APPLICATION INSTRUCTIONS FOR END-USERS:

Use product directly as is from container. DO NOT DILUTE. Masking or painters tape can be applied along one edge of the area that is to be decontaminated to aid creating a peeled edge to grip for peeling the dried film. Apply DeconGel using a paint brush, a trowel, a handheld sprayer, or an industrial grade sprayer (use DeconGel 1120 or 1121 for spray application).

The thickness of the gel and the number of coats is dictated by the surface to be decontaminated. Coating thickness required for good peel characteristics varies with substrate and generally increases with substrate porosity. It is recommended that first time customers test DeconGel on a small sample area to confirm the required film thickness and dry time for their specific application.

If the film is difficult to peel, please apply an additional coat. A razor blade is useful to start the peel. Lay the blade nearly flat and fillet the edge of the film to create a tab that can be pulled. For surfaces that the gel adheres to well, such as concrete, 12" – 24" strips can be cut in the film resulting in less force being required to peel the film.

- Let DeconGel dry for 24 hours

Dry time will vary depending on humidity, temperature, air flow and thickness of the DeconGel. This can take from as little time as an hour for thin coats in a dry environment with plenty of airflow, to overnight or longer if thicker coats are applied in humid environments. Dry times exceeding 24 hours may sometimes be required for good peel performance on bare concrete, wood and other highly porous substrates and substrates with deep cracks or grooves. However, 18-24 hrs is often sufficient dry time on good quality concrete. It is recommended that users test a small area to determine drying time prior to applying DeconGel for an entire job. Supplemental heat or air circulation will accelerate DeconGel's drying time for any job.

- Peel DeconGel off the surface by starting from one of the edges

When dry, the product locks the contaminants into a polymer matrix. The film containing the encapsulated contamination can then be peeled. DeconGel peels from most non-porous and porous hard surfaces if the dried film is thick enough. If the film is difficult to peel, add another coat, let dry, and peel. In most cases the DeconGel will come off in a single sheet but for odd shaped surfaces you may be required to score DeconGel in order to be able to peel it off.

- Dispose of the dried DeconGel in accordance with the local, state and Federal disposal regulations of the contaminant/substance you are removing. DeconGel itself has no special disposal restrictions.

For more information on application techniques visit our website:
www.decongel.com

6.38. End-User Report for Hydrocarbon Petroleum Distillates (Kerosene)

TITLE: Surface Decontamination of Hydrocarbon Petroleum Distillates (Kerosene) by DeconGel 1101 and 1102

ANALYST: Geoff Nadolski PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel formulation 1101 and 1102 on stainless steel, aluminum, and concrete surfaces contaminated with Hydrocarbon Petroleum Distillates (kerosene) using GC/MS (Gas Chromatography/Mass Spectrometry) following Environmental Protection Agency (EPA) SW-846 Methods 3500C (Sampling) and 8270C (Analysis).

HAZARDOUS MATERIALS RELEVANCE: Kerosene is a highly flammable, volatile hydrocarbon liquid petroleum distillate mixture containing between six to sixteen carbon atom molecules. Kerosene is used as a heating fuel, propellant, and solvent and thinner. Ingestion of kerosene is harmful and in sufficient quantities can be fatal. Kerosene was chosen as a representative petroleum distillate; DeconGel is expected to have similar efficacy towards the wide range of hydrocarbon petroleum distillates.

SUMMARY RESULTS:

- Excellent surface decontamination was achieved by applying both DeconGel 1101 and 1102, both via brushing or pouring (non-brushed) onto contaminated surfaces, resulting in encapsulation of kerosene by DeconGel's active components. Decontamination efficacies of poured DeconGel 1101 ranged from 92.8% (on concrete) to 95.8% (on stainless steel) to 96.0% (on aluminum), brushed DeconGel 1101 ranged from 100% (on concrete) to 99.8% (on stainless steel) to 99.8% (on aluminum), as determined by residual swipe analysis. Decontamination efficacies of poured DeconGel 1102 ranged from 100% (on concrete) to 100% (on aluminum) to 100% (on stainless steel), brushed DeconGel 1102 ranged from 100% (on concrete) to 100% (on aluminum) to 100% (on stainless steel), as determined by residual swipe analysis.
- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of organic compounds dissolved in a suitable solvent able to completely solvate kerosene as well as DeconGel components. When necessary, experimental methods were customized to afford complete dissolution of organic contaminants. Additionally, analytical methods and associated equipment (GC column, GC temperature gradient program, MS

sample ionization parameters) were appropriately utilized to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 shows the decontamination efficacies of DeconGel 1101 and 1102 on stainless steel, aluminum, and concrete surfaces contaminated with Hydrocarbon Petroleum Distillates (kerosene) as determined by residual swipe testing.

Table 1. Decontamination efficacies of DeconGel 1101 and 1102 on Kerosene Hydrocarbon Petroleum Distillate contaminated stainless steel, aluminum, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		**Formulation**	
		DeconGel 1101	**DeconGel 1102**
Stainless Steel	Control	89.31 ± 0.18	89.31 ± 0.18
	Residual (non-brushed)	3.76 ± 0.07	ND*
	Residual (brushed)	0.112 ± 0.001	ND
	Decon. Efficacy (non-brushed) (%)	**95.8 ± 0.12**	**100****
	Decon. Efficacy (brushed) (%)	**99.8 ± 0.06**	**100****
Aluminum	Control	89.34 ± 0.30	89.34 ± 0.30
	Residual (non-brushed)	3.59 ± 0.10	ND
	Residual (brushed)	0.111 ± 0.002	ND
	Decon. Efficacy (non-brushed) (%)	**96.0 ± 0.10**	**100****
	Decon. Efficacy (brushed) (%)	**99.8 ± 0.06**	**100****
Concrete	Control	70.38 ± 1.37	70.38 ± 1.37
	Residual (non-brushed)	5.10 ± 0.02	ND
	Residual (brushed)	ND	ND
	Decon. Efficacy (non-brushed) (%)	**92.8 ± 0.16**	**100****
	Decon. Efficacy (brushed) (%)	**100****	**100****

412x dilution factor for samples and controls

* ND: not detected, limit of detection (LOD) for kerosene approximates 250 ppb

** An ND value for residual experiments results in a Decontamination Efficacy of 100%

NOTES:

- ASTM method E1728-03, a standardized swipe testing method used for sampling of contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. Air-dried GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes were utilized in this swipe testing method.

- Standardized EPA SW-846 Sampling Method 3500C “Organic Extraction and Sample Preparation” was followed as a guideline to prepare all samples and controls. All samples, controls, and standards were prepared using the same solvent and appropriate experimental conditions to ensure accurate and GC/MS instrumental analysis.
- GC/MS instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of organic compounds. Standardized EPA SW-846 Analytical Method 8270C “SEMIVOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)” was followed as a guideline to prepare all samples and controls.
- To ensure accurate determination of DeconGel decontamination efficacy, a standard curve of the analyte of interest was prepared using sufficiently pure analyte; the respective standards were diluted to a known concentration (ppm) using the same solvent as used for samples and controls.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 100 uL of contaminant was evenly applied via brushing on 1) stainless steel (commercial grade, surface area: 56.3 cm^2), 2) aluminum (commercial grade, surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 50 mL hexane, and gel samples were first suspended in 20 mL water and then 20 mL hexane was added, and let to stand for 24 h. For gel samples, the water layer was extracted two more times with first 20 mL hexane and then 10 mL hexane, such that the hexane layers (50 mL total) were combined. Samples were then analyzed via GC/MS (see below).

Controls Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied via brushing on 1) stainless steel (commercial grade, surface area: 56.3 cm^2), 2) floor tile (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 50 mL hexane for 24 h and analyzed via GC/MS (see below).

Reagents and Standards

Kerosene, odorless (CAS# 64742-14-9, Acros; NJ) was used as received.

Analytical Instrumentation

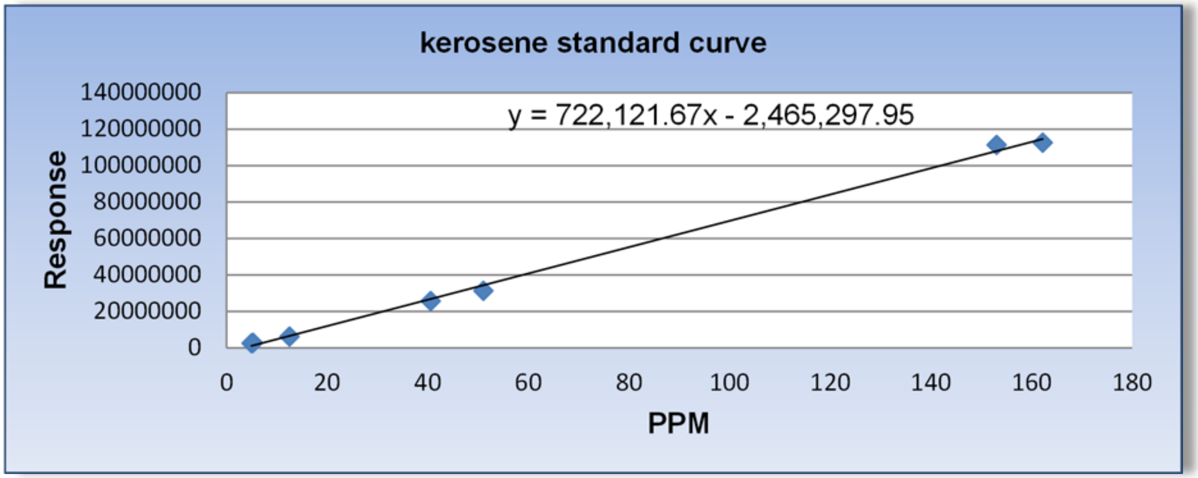
A Thermo DSQII GC/MS with autosampler in positive mode was used to determine EG concentration (ppm, wt/wt) of all samples and controls, using a Restek (Bellefonte, PA) Rtx-5 capillary column (30 m x 0.25 mm, 0.5 um).

A 9-point standard curve derived from three independently prepared stock solutions was prepared using DMSO as the working solvent.

GC method: start at 45 ºC, hold for 7 min, ramp at 30 ºC/min to 320 ºC, hold 10 min.

Kerosene GC/MS data: 11.5 min; $M^+ = 170$ ($C_{12}H_{26}$)

Figure 1. Kerosene Standard Calibration Curve



APPLICATION INSTRUCTIONS FOR END-USERS:

Use product directly as is from container. DO NOT DILUTE. Masking or painters tape can be applied along one edge of the area that is to be decontaminated to aid creating a peeled edge to grip for peeling the dried film. Apply DeconGel using a paint brush, a trowel, a handheld sprayer, or an industrial grade sprayer (use DeconGel 1120 or 1121 for spray application).

The thickness of the gel and the number of coats is dictated by the surface to be decontaminated. Coating thickness required for good peel characteristics varies with substrate and generally increases with substrate porosity. It is recommended that first time customers test DeconGel on a small sample area to confirm the required film thickness and dry time for their specific application. If the film is difficult to peel, please apply an additional coat. A razor blade is useful to start the peel. Lay the blade nearly flat and fillet the edge of the film to create a tab that can be pulled. For surfaces that the gel adheres to well, such as concrete, 12” – 24” strips can be cut in the film resulting in less force being required to peel the film.

- Let DeconGel dry for 24 hours

Dry time will vary depending on humidity, temperature, air flow and thickness of the DeconGel. This can take from as little time as an hour for thin coats in a dry environment with plenty of airflow, to overnight or longer if thicker coats are applied in humid environments. Dry times exceeding 24 hours may sometimes be required for good peel performance on bare concrete, wood and other highly porous substrates and substrates with deep cracks or grooves. However, 18-24 hrs is often sufficient dry time on good quality concrete. It is recommended that users test a small area to determine drying time prior to applying DeconGel for an entire job. Supplemental heat or air circulation will accelerate DeconGel’s drying time for any job.

- Peel DeconGel off the surface by starting from one of the edges

When dry, the product locks the contaminants into a polymer matrix. The film containing the encapsulated contamination can then be peeled. DeconGel peels from most non-porous and porous hard surfaces if the dried film is thick enough. If the film is difficult to peel, add another coat, let dry, and peel. In most cases the DeconGel will come off in a single sheet but for odd shaped surfaces you may be required to score DeconGel in order to be able to peel it off.

- Dispose of the dried DeconGel in accordance with the local, state and Federal disposal regulations of the contaminant/substance you are removing. DeconGel itself has no special disposal restrictions.

For more information on application techniques visit our website: www.decongel.com

6.39. End-User Report for Iodine Compounds (Potassium Iodide)

TITLE: Surface Decontamination of Iodine Compounds (Potassium Iodide (KI)) by DeconGel 1101

AUTHOR: Andreas Mylonakis PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel 1101 on stainless steel, carbon steel, floor tile, and concrete surfaces contaminated with potassium iodide (KI) using ICP-OES (Inductively Coupled Plasma-Optical Emission Spectroscopy) following Environmental Protection Agency (EPA) SW-846 Methods 3005A (sampling) and 6010C (analysis).

HAZARDOUS MATERIALS RELEVANCE: Iodine and its compounds are used in medicine, photography, and the dye industry. Iodides are soluble in water and are concentrated in seawater. Iodine is an oxidizing irritant and allergen and is poisonous if taken orally in large amounts. Potassium iodide was chosen as a representative iodine compound for evaluating DeconGel's efficacy; DeconGel is expected to have similar efficacy towards the wide range of iodine compounds.

HIGHLIGHTS:

- Excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with iodine compounds resulting in encapsulation of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 98.6% (on floor tile) to 98.7% (on concrete) to 98.8% (on carbon steel) to 99.5% (on stainless steel) as determined by residual swipe analysis.

- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for the determination of inorganic compounds in aqueous samples. When necessary, experimental methods were customized to afford complete dissolution of inorganic contaminants, and to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Table 1 shows the decontamination efficacies of DeconGel 1101 on stainless steel, carbon steel, floor tile and concrete surfaces contaminated with potassium iodide as determined by residual swipe testing.

Table 1. Decontamination efficacy of DeconGel 1101 on KI contaminated stainless steel, carbon steel, floor tile, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	531.3 ± 2.5
	Residual	8.9 ± 4.1
	Decon. Efficacy (%)	**99.5 ± 2.9**
Carbon Steel	Control	521.8 ± 11.6
	Residual	6.2 ± 4.8
	Decon. Efficacy (%)	**98.8 ± 6.8**
Floor Tile	Control	293.3 ± 19.8
	Residual	4.14 ± 3.30
	Decon. Efficacy (%)	**98.6 ± 1.1**
Concrete	Control	514.4 ± 11.5
	Residual	6.7 ± 2.8
	Decon. Efficacy (%)	**98.7 ± 3.2**

21880x dilution factor for samples and controls

NOTES:

- Evaluation of the decontamination efficacy of DeconGel on KI on waxed floor tile was conducted to simulate an iodine/iodide spill as evidenced in a medical/clinical setting, such that the formed KI deposit is intended to simulate a radioactive and/or pharmaceutical form of iodine/iodide. DeconGel effectively decontaminated several forms of iodide contamination (from loose residual solids to dense deposits) on multiple types of surfaces (from non-porous, inert metals to waxy, chemically active plastics, to porous substrates).

- ASTM method E1728-03, a standardized swipe testing method used for sampling of inorganic contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes (pre-wetted with DI H_2O) were utilized in this swipe testing method.

- Standardized EPA SW-846 Sampling Method 3005A "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy" was followed as a guideline to prepare all samples and controls.

- To ensure accurate determination of DeconGel decontamination efficacy, calibration standards of the analyte of interest were prepared using either a sufficiently pure analyte or an appropriate ICP-MS Standard (Ricca Chemical Company; Arlington, TX); the respective standards were diluted to a known concentration (ppm) using the same digestion method as the one used for samples and controls. Instrument blank controls were DI H_2O ($\geq$17 M-Ohm).

- ICP-OES instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of inorganic compounds. Standardized EPA SW-846 Analytical Method 6010C "Inductively Coupled Plasma-Atomic Emission Spectrometry" was followed as a guideline to prepare all samples and controls.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(KI (ppm) of Swipe Control) – (KI (ppm) of Residual Swipe)/KI (ppm) of Swipe Control] x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.05 g KI was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons; for waxed floor tile (surface area: 221.9 cm^2), 1.0 mL aqueous KI (50 mg/mL) was homogeneously pipetted onto the coupon surface and let to stand for one week until all water had evaporated and a KI deposit had formed. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24-48 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 100 mL aqueous acidic solution (15% HCl, 15% HNO_3, 70% deionized (DI) H_2O) for 24 h. When deemed necessary, samples were heated to 90 °C for 8-24 h to afford complete dissolution of inorganics using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC). Samples were allowed to cool to room temperature and were then analyzed via ICP-OES (see below).

Controls Methods

For Swipe Control samples, 0.05 g KI was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons: for waxed floor tile (surface area: 221.9 cm^2), 1.0 mL aqueous KI (50 mg/mL) was homogeneously pipetted onto the coupon surface and let to stand for one week until all water had evaporated and a KI deposit had formed. Coupon surfaces were swipe tested (ASTM method 1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 100 mL aqueous acidic solution (15% HCl, 15% HNO_3, 70% DI H_2O) for 24 h and analyzed via ICP-OES (see below).

Reagents and Standards

Reagent grade potassium iodide (KI) (CAS# 7681-11-0, Fisher Scientific; Fair Lawn, NJ) was used as received.

A 10.0 ppm calibration standard was prepared using reagent grade granular KI and freshly prepared aqueous acidic solution (15% HCl, 15% HNO_3, 70% DI H_2O). DI H_2O was used as the blank sample.

Analytical Instrumentation

A Thermo ICP-OES instrument model Radial iCap 6300 was used to determine KI concentration (ppm) of all samples and controls using a freshly prepared 10.0 ppm calibration standard.
Analyte (iodine) analyzed at 183.0 nm

Pump Speed: 0.5 mL/min

APPLICATION INSTRUCTIONS FOR END-USERS:

Use product directly as is from container. DO NOT DILUTE. Masking or painters tape can be applied along one edge of the area that is to be decontaminated to aid creating a peeled edge to grip for peeling the dried film. Apply DeconGel using a paint brush, a trowel, a handheld sprayer, or an industrial grade sprayer (use DeconGel 1120 or 1121 for spray application).

The thickness of the gel and the number of coats is dictated by the surface to be decontaminated. Coating thickness required for good peel characteristics varies with substrate and generally increases with substrate porosity. It is recommended that first time customers test DeconGel on a small sample area to confirm the required film thickness and dry time for their specific application. If the film is difficult to peel, please apply an additional coat. A razor blade is useful to start the peel. Lay the blade nearly flat and fillet the edge of the film to create a tab that can be pulled. For surfaces that the gel adheres to well, such as concrete, 12" – 24" strips can be cut in the film resulting in less force being required to peel the film.

- Let DeconGel dry for 24 hours

Dry time will vary depending on humidity, temperature, air flow and thickness of the DeconGel. This can take from as little time as an hour for thin coats in a dry environment with plenty of airflow, to overnight or longer if thicker coats are applied in humid environments. Dry times exceeding 24 hours may sometimes be required for good peel performance on bare concrete, wood and other highly porous substrates and substrates with deep cracks or grooves. However, 18-24 hrs is often sufficient dry time on good quality concrete. It is recommended that users test a small area to determine drying time prior to applying DeconGel for an entire job. Supplemental heat or air circulation will accelerate DeconGel's drying time for any job.

- Peel DeconGel off the surface by starting from one of the edges

When dry, the product locks the contaminants into a polymer matrix. The film containing the encapsulated contamination can then be peeled. DeconGel peels from most non-porous and porous hard surfaces if the dried film is thick enough. If the film is difficult to peel, add another coat, let dry, and peel. In most cases the DeconGel will come off in a single sheet but for odd shaped surfaces you may be required to score DeconGel in order to be able to peel it off.

- Dispose of the dried DeconGel in accordance with the local, state and Federal disposal regulations of the contaminant/substance you are removing. DeconGel itself has no special disposal restrictions.

For more information on application techniques visit our website: www.decongel.com

6.40. End-User Report for Industrial Coolants (Ethylene Glycol)

TITLE: Surface Decontamination of Industrial Coolants (Ethylene Glycol) by DeconGel 1101

ANALYST: Geoff Nadolski PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel formulation 1101 on stainless steel, aluminum, and concrete surfaces contaminated with Industrial Coolants (ethylene glycol (EG)) using GC/MS (Gas Chromatography/Mass Spectrometry) following Environmental Protection Agency (EPA) SW-846 Methods 3500C (Sampling) and 8270C (Analysis).

HAZARDOUS MATERIALS RELEVANCE: EG is viscous odorless liquid used widely as a coolant/heat transfer agent. EG is also used as a desiccant, and as a polymer precursor, and is moderately orally toxic to humans. Ingestion of large amounts of EG can be fatal if untreated. EG was chosen as a representative industrial coolant; DeconGel is expected to have similar efficacy towards the wide range of industrial coolants.

SUMMARY RESULTS:

- Excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with industrial coolants (EG) resulting in encapsulation of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 93.6% (on concrete) to 95.5% (on stainless steel) to 95.5% (on aluminum) for EG determined by residual swipe analysis.
- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of organic compounds dissolved in a suitable solvent able to completely solvate aniline as well as DeconGel components. When necessary, experimental methods were customized to afford complete dissolution of organic contaminants. Additionally, analytical methods and associated equipment (GC column, GC temperature gradient program, MS sample ionization parameters) were appropriately utilized to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 shows the decontamination efficacies of DeconGel 1101 on stainless steel, aluminum, and concrete surfaces contaminated with Industrial Coolants (EG) as determined by residual swipe testing.

Table 1. Decontamination efficacy of DeconGel 1101 on Industrial Coolant Ethylene Glycol contaminated stainless steel, aluminum, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	203.43 ± 0.29
	Residual	9.08 ± 0.01
	Decon. Efficacy (%)	**95.5 ± 0.01**
Aluminum	Control	203.61 ± 1.03
	Residual	9.08 ± 0.02
	Decon. Efficacy (%)	**95.5 ± 0.01**
Concrete	Control	155.31 ± 0.74
	Residual	9.97 ± 0.03
	Decon. Efficacy (%)	**93.6 ± 0.01**

1422x dilution factor for samples and controls

NOTES:

- ASTM method E1728-03, a standardized swipe testing method used for sampling of contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. Air-dried GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes were utilized in this swipe testing method.

- Standardized EPA SW-846 Sampling Method 3500C "Organic Extraction and Sample Preparation" was followed as a guideline to prepare all samples and controls. All samples, controls, and standards were prepared using the same solvent and appropriate experimental conditions to ensure accurate and GC/MS instrumental analysis.

- GC/MS instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of organic compounds. Standardized EPA SW-846 Analytical Method 8270C "SEMIVOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)" was followed as a guideline to prepare all samples and controls.

- To ensure accurate determination of DeconGel decontamination efficacy, a standard curve of the analyte of interest was prepared using sufficiently pure analyte; the respective standards were diluted to a known concentration (ppm) using the same solvent as used for samples and controls.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 25 uL of contaminant was evenly applied via brushing on 1) aluminum (commercial grade, surface area: 56.3 cm^2), 2) stainless steel (commercial grade, surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe and gel samples were suspended in 50 mL 60% methanol in water for 24 h. Samples were then analyzed via GC/MS (see below).

Controls Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied via brushing on 1) aluminum (commercial grade, surface area: 56.3 cm^2), 2) stainless steel (commercial grade, surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 50 mL 60% methanol in water for 24 h and analyzed via GC/MS (see below).

Reagents and Standards

Ethylene glycol (EG) (ethane-1,2-diol) (CAS# 107-21-1, Fisher Scientific; Fair Lawn, NJ) was used as received.

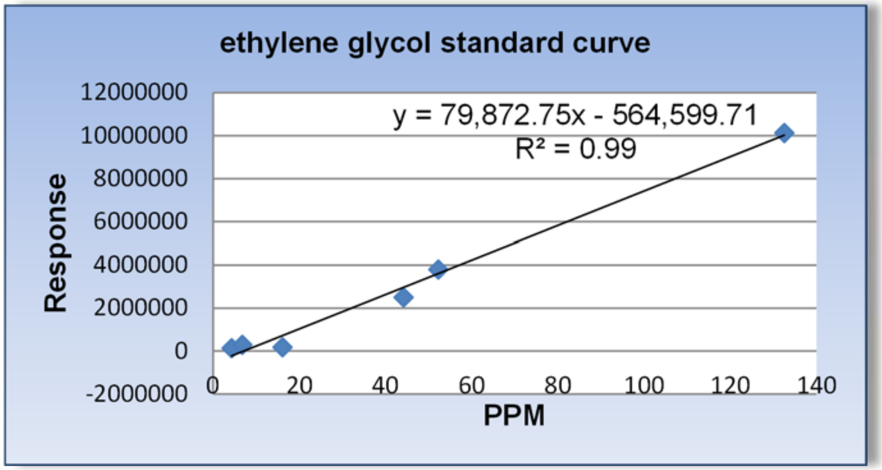
Analytical Instrumentation

A Thermo DSQII GC/MS with autosampler in positive mode was used to determine EG concentration (ppm, wt/wt) of all samples and controls, using a Restek (Bellefonte, PA) Rtx-5 capillary column (30 m x 0.25 mm, 0.5 um).

A 7-point standard curve derived from three independently prepared stock solutions was prepared using DMSO as the working solvent. The calibration curve exhibits a curve fitting as approximated by the coefficient of determination of linear regression R^2, where $R^2 = 0.99$ (see Figure 1). GC method: start at 45 ºC, hold for 1 min, ramp at 30 ºC/min to 320 ºC, hold 10 min.

EG GC/MS data: 2.8 min; $M^+ = 62$.

Figure 1. **EG Standard Calibration Curve**



APPLICATION INSTRUCTIONS FOR END-USERS:

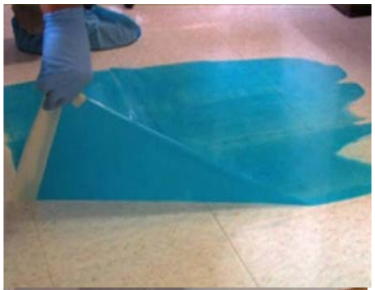
Use product directly as is from container. DO NOT DILUTE. Masking or painters tape can be applied along one edge of the area that is to be decontaminated to aid creating a peeled edge to grip for peeling the dried film. Apply DeconGel using a paint brush, a trowel, a handheld sprayer, or an industrial grade sprayer (use DeconGel 1120 or 1121 for spray application).

The thickness of the gel and the number of coats is dictated by the surface to be decontaminated. Coating thickness required for good peel characteristics varies with substrate and generally increases with substrate porosity. It is recommended that first time customers test DeconGel on a small sample area to confirm the required film thickness and dry time for their specific application. If the film is difficult to peel, please apply an additional coat. A razor blade is useful to start the peel. Lay the blade nearly flat and fillet the edge of the film to create a tab that can be pulled. For surfaces that the gel adheres to well, such as concrete, 12" – 24" strips can be cut in the film resulting in less force being required to peel the film.

- Let DeconGel dry for 24 hours

Dry time will vary depending on humidity, temperature, air flow and thickness of the DeconGel. This can take from as little time as an hour for thin coats in a dry environment with plenty of airflow, to overnight or longer if thicker coats are applied in humid environments. Dry times exceeding 24 hours may sometimes be required for good peel performance on bare concrete, wood and other highly porous substrates and substrates with deep cracks or grooves. However, 18-24 hrs is often sufficient dry time on good quality concrete. It is recommended that users test a small area to determine drying time prior to applying DeconGel for an entire job. Supplemental heat or air circulation will accelerate DeconGel's drying time for any job.

- Peel DeconGel off the surface by starting from one of the edges



When dry, the product locks the contaminants into a polymer matrix. The film containing the encapsulated contamination can then be peeled. DeconGel peels from most non-porous and porous hard surfaces if the dried film is thick enough. If the film is difficult to peel, add another coat, let dry, and peel. In most cases the DeconGel will come off in a single sheet but for odd shaped surfaces you may be required to score DeconGel in order to be able to peel it off.

- Dispose of the dried DeconGel in accordance with the local, state and Federal disposal regulations of the contaminant/substance you are removing. DeconGel itself has no special disposal restrictions.

For more information on application techniques visit our website:
www.decongel.com

6.41. End-User Report for Industrial Solvents (*m*-Cresol)

TITLE: Surface Decontamination of Industrial Solvents (*m*-Cresol) by DeconGel 1101

ANALYST: Geoff Nadolski PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel formulation 1101 on stainless steel, aluminum, and concrete surfaces contaminated with Industrial Solvents (*m*-Cresol) using LC/MS (Liquid Chromatography/Mass Spectrometry) following Environmental Protection Agency (EPA) SW-846 Methods 3500C (Sampling) and 8321B (Analysis).

HAZARDOUS MATERIALS RELEVANCE: Meta-Cresol is a methylated aromatic phenolic liquid used as a disinfectant, deodorizer, precursor to insecticides, and as an industrial solvent. Cresol is a phenolic irritant that can cause a severe burning of sensitive tissues, and if ingested or absorbed at high levels can damage the kidneys, liver, brain, and lungs. Cresol was chosen as a representative industrial solvent for evaluating DeconGel's efficacy; DeconGel is expected to have similar efficacy towards the wide range of industrial solvents.

SUMMARY RESULTS:

- Excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with industrial solvents (*m*-cresol) resulting in encapsulation of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 99.4% (on concrete) to 99.8% (on aluminum) to 99.8% (on stainless steel) for *m*-Cresol determined by residual swipe analysis.

- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of organic compounds dissolved in a suitable solvent able to completely solvate nicotine as well as DeconGel components. When necessary, experimental methods were customized to afford complete dissolution of organic contaminants. Additionally, analytical methods and associated equipment (LC column, LC gradient program, MS sample ionization parameters) were appropriately utilized to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 shows the decontamination efficacies of DeconGel 1101 on stainless steel, aluminum, and concrete surfaces contaminated with Industrial Solvents (*m*-cresol) as determined by residual swipe testing.

Table 1. Decontamination efficacy of DeconGel 1101 on Cresol Industrial Solvent contaminated stainless steel, aluminum, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	524.38 ± 1.98
	Residual	1.23 ± 0.01
	Decon. Efficacy (%)	**99.8 ± 0.0**
Aluminum	Control	523.94 ± 1.16
	Residual	1.24 ± 0.003
	Decon. Efficacy (%)	**99.8 ± 0.0**
Concrete	Control	410.48 ± 2.37
	Residual	2.40 ± 0.03
	Decon. Efficacy (%)	**99.4 ± 0.0**

1375x dilution factor for samples and controls

NOTES:

- ASTM method E1728-03, a standardized swipe testing method used for sampling of contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. Air-dried GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes were utilized in this swipe testing method.

- Standardized EPA SW-846 Sampling Method 3500C "Organic Extraction and Sample Preparation" was followed as a guideline to prepare all samples and controls. All samples, controls, and standards were prepared using the same solvent and appropriate experimental conditions to ensure accurate and LC/MS instrumental analysis.

- LC/MS instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of organic compounds. Standardized EPA SW-846 Analytical Method 8321B "Solvent-Extractable Nonvolatile Compounds by High-Performance Liquid Chromatography/Thermospray/Mass Spectrometry (HPLC/TS/MS) or Ultraviolet (UV) Detection" was followed as a guideline to prepare all samples and controls.

- To ensure accurate determination of DeconGel decontamination efficacy, a standard curve of the analyte of interest was prepared using sufficiently pure analyte; the respective standards were diluted to a known concentration (ppm) using the same solvent as used for samples and controls.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 40 uL of contaminant was evenly applied via brushing on 1) stainless steel (commercial grade, surface area: 56.3 cm^2), 2) aluminum (commercial grade, surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe and gel samples were suspended in 50 mL DMSO for 24 h. Samples were then analyzed via LC/MS (see below).

Controls Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied via brushing on 1) stainless steel (surface area: 56.3 cm^2), 2) aluminum (surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 50 mL DMSO for 24 h and analyzed via LC/MS (see below).

Reagents and Standards

Meta-Cresol (3-methylphenol) (CAS# 108-39-4, Fisher Scientific; Fair Lawn, NJ) was used as received.

Analytical Instrumentation

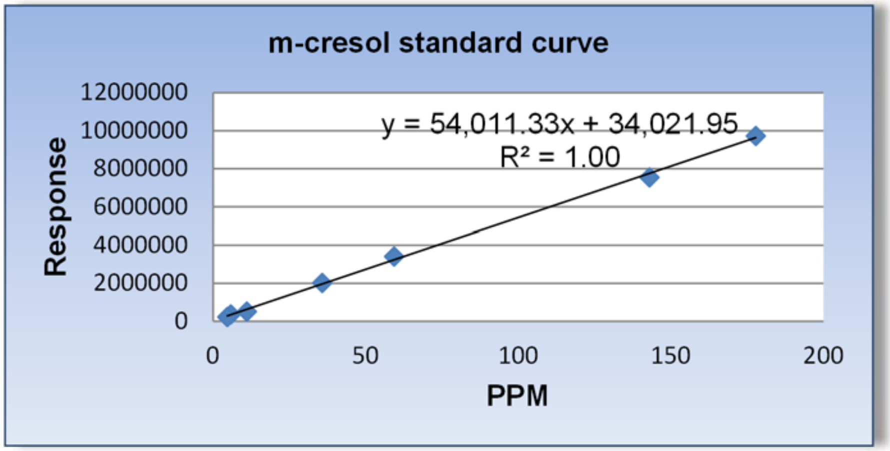
A Thermo LCQ LC/MS with autosampler in positive mode was used to determine nicotine concentration (ppm, wt/wt) of all samples and controls, using a Grace Davison (Deerfield, IL) C18-Select analytical column (150 x 4.6 mm, 5 um).

A 7-point standard curve derived from three independently prepared stock solutions was prepared using DMSO as the working solvent.

LC method using A= water (0.1% formic acid), B=acetonitrile (0.1% formic acid); start at 95%A to 3 min at 70%A, to 6 min at 50%A, to 12 min at 30%A, to 15 min at 90%B, to 16 min at 100%B, hold until 22 min at 100% B, to 23 min at 95%A, hold until 24 min.

Meta-Cresol LC/MS data: 9.8 min; lambda max = 261, 272, 283 nm; M^+ = 108.

Figure 1. Cresol Standard Calibration Curve



APPLICATION INSTRUCTIONS FOR END-USERS:

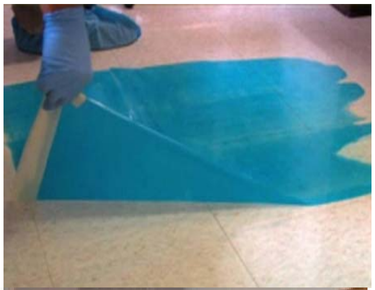
Use product directly as is from container. DO NOT DILUTE. Masking or painters tape can be applied along one edge of the area that is to be decontaminated to aid creating a peeled edge to grip for peeling the dried film. Apply DeconGel using a paint brush, a trowel, a handheld sprayer, or an industrial grade sprayer (use DeconGel 1120 or 1121 for spray application).

The thickness of the gel and the number of coats is dictated by the surface to be decontaminated. Coating thickness required for good peel characteristics varies with substrate and generally increases with substrate porosity. It is recommended that first time customers test DeconGel on a small sample area to confirm the required film thickness and dry time for their specific application. If the film is difficult to peel, please apply an additional coat. A razor blade is useful to start the peel. Lay the blade nearly flat and fillet the edge of the film to create a tab that can be pulled. For surfaces that the gel adheres to well, such as concrete, 12" – 24" strips can be cut in the film resulting in less force being required to peel the film.

- Let DeconGel dry for 24 hours

Dry time will vary depending on humidity, temperature, air flow and thickness of the DeconGel. This can take from as little time as an hour for thin coats in a dry environment with plenty of airflow, to overnight or longer if thicker coats are applied in humid environments. Dry times exceeding 24 hours may sometimes be required for good peel performance on bare concrete, wood and other highly porous substrates and substrates with deep cracks or grooves. However, 18-24 hrs is often sufficient dry time on good quality concrete. It is recommended that users test a small area to determine drying time prior to applying DeconGel for an entire job. Supplemental heat or air circulation will accelerate DeconGel's drying time for any job.

- Peel DeconGel off the surface by starting from one of the edges



When dry, the product locks the contaminants into a polymer matrix. The film containing the encapsulated contamination can then be peeled. DeconGel peels from most non-porous and porous hard surfaces if the dried film is thick enough. If the film is difficult to peel, add another coat, let dry, and peel. In most cases the DeconGel will come off in a single sheet but for odd shaped surfaces you may be required to score DeconGel in order to be able to peel it off.

- Dispose of the dried DeconGel in accordance with the local, state and Federal disposal regulations of the contaminant/substance you are removing. DeconGel itself has no special disposal restrictions.

For more information on application techniques visit our website: www.decongel.com

6.42. End-User Report for Iron Compounds (Iron Chloride)

TITLE: Surface Decontamination of Iron Compounds (Iron (II) Chloride ($FeCl_2$)) by DeconGel 1101

AUTHOR: Andreas Mylonakis PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel 1101 on stainless steel, carbon steel, aluminum and concrete surfaces contaminated with Iron (II) Chloride ($FeCl_2$) using ICP-OES (Inductively Coupled Plasma-Optical Emission Spectroscopy) following Environmental Protection Agency (EPA) SW-846 Methods 3005A (sampling) and 6010C (analysis).

HAZARDOUS MATERIALS RELEVANCE: While iron itself is non-hazardous, iron dust can be considered as an inhalation hazard. Compounds of iron are often corrosive and harmful; one such compound is iron (II) chloride ($FeCl_2$) or ferrous chloride which is often found in the waste water treatment arena and in laboratory settings as a reducing agent. Ferrous chloride was chosen as a representative iron compound for evaluating DeconGel's efficacy; DeconGel is expected to have similar efficacy towards the wide range of iron compounds.

HIGHLIGHTS:

- Excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with iron chloride resulting in the encapsulation of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 98.8% (on concrete) to 99.5% (on carbon steel) to 99.5% (on stainless steel) as determined by residual swipe testing.

- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of inorganics in aqueous samples. When necessary, experimental methods were customized to afford complete dissolution of inorganic contaminants, and to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Table 1 shows the decontamination efficacies of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with ferrous chloride as determined by residual swipe testing.

Table 1. Decontamination efficacies of DeconGel 1101 against ferrous chloride ($FeCl_2$) contaminated stainless steel, carbon steel, and concrete surfaces as determined by residual swipe testing method.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	574.5 + 19.8
	Residual	2.60 + 0.51
	Decon. Efficacy (%)	**99.5 + 5.1**
Carbon Steel	Control	582.6 + 19.9
	Residual	2.66 + 1.1
	Decon. Efficacy (%)	**99.5 + 6.3**
Concrete	Control	536.2 + 20.4
	Residual	6.20 + 3.4
	Decon. Efficacy (%)	**98.8 + 6.5**

21880x dilution factor for samples and controls

NOTES:

- ASTM method E1728-03, a standardized swipe testing method used for inorganic contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes (pre-wetted with DI H_2O) were utilized in this swipe testing method.

- Standardized EPA SW-846 Sampling Method 3005A "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy" was followed as a guideline to prepare all samples and controls. When necessary, digestion methods were customized by increasing hydrochloric and nitric acid concentrations from 20% to 30% wt, and/or by heating samples to higher temperatures using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC) for the complete digestion/dissolution of the inorganic contaminants. All samples, controls, and standards were prepared using the same dissolution solution and experimental conditions to ensure both correct instrument calibration and accurate analytical results.

- ICP-OES instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of inorganic compounds. Standardized EPA SW-846 Analytical Method 6010C "Inductively Coupled Plasma-Atomic Emission Spectrometry" was followed as a guideline to prepare all samples and controls.

- To ensure accurate determination of DeconGel decontamination efficacy, calibration standards of the analyte of interest were prepared using either a sufficiently pure analyte or an appropriate ICP-MS Standard (Ricca Chemical Company; Arlington, TX); the respective standards were diluted to a known concentration (ppm) using the same digestion method as the one used for samples and controls. Instrument blank controls were DI H_2O ($\geq$17 M-Ohm).

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.05 g contaminant was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), and 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24-48 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipes were suspended in 100 mL aqueous acidic solution (15% HCl, 15% HNO_3, 70% deionized (DI) H_2O) for 24 h. When deemed necessary, samples were heated to 94 ^oC for 4-14 h to effectively complete dissolution of inorganics using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC). Samples were allowed to cool to room temperature and were then analyzed via ICP-OES (see below).

Control Method

For Swipe Controls, 0.05 g contaminant was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 100 mL aqueous acidic solution (15% HCl, 15% HNO_3, 70% DI H_2O) for 24 h and analyzed via ICP-OES (see below).

Reagents and Standards

Reagent grade ferrous chloride, Iron (II) Chloride, $FeCl_2$, (CAS# 13478-10-9, Fisher Scientific; Fair Lawn, NJ) was used as received.

A 1000 ppm calibration standard was prepared using reagent grade ferrous chloride and freshly prepared aqueous acidic solution (15% HCl, 15% HNO_3, 70% DI H_2O). DI H_2O was used as the blank sample.

Analytical Instrumentation

A Thermo ICP-OES instrument model radial iCap 6300 was used to determine $FeCl_2$ concentration (ppm) of all samples and controls using a freshly prepared 1000 ppm calibration standard.

Analyte (iron) analyzed at 259.9 nm

Pump Speed: 0.5 mL/min

APPLICATION INSTRUCTIONS FOR END-USERS:

Use product directly as is from container. DO NOT DILUTE. Masking or painters tape can be applied along one edge of the area that is to be decontaminated to aid creating a peeled edge to grip for peeling the dried film. Apply DeconGel using a paint brush, a trowel, a handheld sprayer, or an industrial grade sprayer (use DeconGel 1120 or 1121 for spray application).

The thickness of the gel and the number of coats is dictated by the surface to be decontaminated. Coating thickness required for good peel characteristics varies with substrate and generally increases with substrate porosity. It is recommended that first time customers test DeconGel on a small sample area to

confirm the required film thickness and dry time for their specific application. If the film is difficult to peel, please apply an additional coat. A razor blade is useful to start the peel. Lay the blade nearly flat and fillet the edge of the film to create a tab that can be pulled. For surfaces that the gel adheres to well, such as concrete, 12” – 24” strips can be cut in the film resulting in less force being required to peel the film.

➢ Let DeconGel dry for 24 hours

Dry time will vary depending on humidity, temperature, air flow and thickness of the DeconGel. This can take from as little time as an hour for thin coats in a dry environment with plenty of airflow, to overnight or longer if thicker coats are applied in humid environments. Dry times exceeding 24 hours may sometimes be required for good peel performance on bare concrete, wood and other highly porous substrates and substrates with deep cracks or grooves. However, 18-24 hrs is often sufficient dry time on good quality concrete. It is recommended that users test a small area to determine drying time prior to applying DeconGel for an entire job. Supplemental heat or air circulation will accelerate DeconGel’s drying time for any job.

➢ Peel DeconGel off the surface by starting from one of the edges

When dry, the product locks the contaminants into a polymer matrix. The film containing the encapsulated contamination can then be peeled. DeconGel peels from most non-porous and porous hard surfaces if the dried film is thick enough. If the film is difficult to peel, add another coat, let dry, and peel. In most cases the DeconGel will come off in a single sheet but for odd shaped surfaces you may be required to score DeconGel in order to be able to peel it off.

- Dispose of the dried DeconGel in accordance with the local, state and Federal disposal regulations of the contaminant/substance you are removing. DeconGel itself has no special disposal restrictions.

For more information on application techniques visit our website: www.decongel.com

6.43. End-User Report for non-elemental Mercury Compounds (Mercury (II) Chloride, mercury (II) Oxide)

TITLE: Surface Decontamination of Mercury Compounds (Mercury (II) Chloride ($HgCl_2$) and Mercury Oxide (HgO)) by DeconGel 1101

AUTHOR: Andreas Mylonakis PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel formulation 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with mercury(II) chloride ($HgCl_2$) and mercury(II) oxide (HgO) using ICP-OES (Inductively Coupled Plasma-Optical Emission Spectroscopy) following Environmental Protection Agency (EPA) SW-846 Methods 3005A (sampling) and 6010C (analysis).

HAZARDOUS MATERIALS RELEVANCE: Mercury compounds (mercury (II) chloride ($HgCl_2$) and mercury (II) oxide (HgO)) are commonly used for a variety of industrial applications. Mercury oxide is often decomposed to produce elemental mercury and is used in the production of mercuric batteries. Mercury (II) chloride is a well known reagent in analytical and organic chemistry and is also used as a depolarizer in batteries. Both compounds are highly toxic if ingested or inhaled. Mercury (II) chloride and mercury (II) oxide were chosen as representative mercury compounds for evaluating DeconGel's efficacy; DeconGel is expected to have similar efficacy towards the wide range of mercury compounds.

HIGHLIGHTS:

- Acceptable to excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with mercury compounds resulting in encapsulation of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from greater than 95.3% (on concrete) to greater than 57.7% (carbon steel) to greater than 64.6% (on stainless steel) as determined by residual swipe analysis

- Due to the corrosive nature of mercury(II) chloride, DeconGel surface decontamination was not found to be exceptional on steel (carbon and stainless steel) surfaces due to mercury chloride's ability to react with these surfaces, forming a fixed residue that was not able to be completely removed by DeconGel. Nevertheless, DeconGel showed acceptable decontamination efficacy of loose mercury chloride contamination from such surfaces.

- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of inorganic compounds in aqueous samples. When necessary, experimental methods were customized to afford complete dissolution of inorganic contaminants, and to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 and 2 show the decontamination efficacies of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with mercury compounds as determined by residual swipe testing.

Table 1. Decontamination efficacies of DeconGel 1101 against mercury(II) chloride ($HgCl_2$) contaminated stainless steel, carbon steel, and concrete surfaces as determined by residual swipe testing method.

Swipe Testing* (ppm)		**Formulation**
		DeconGel 1101
Stainless Steel*	Control	568.9 ± 32.6
	Residual	31.9 ± 38.2
	Decon. Efficacy (%)	**64.6 ± 13.8**
Carbon Steel*	Control	584.9 ± 32.6
	Residual	97.2 ± 75.3
	Decon. Efficacy (%)	**57.7 ± 8.1**
Concrete	Control	525.0 ± 21.3
	Residual	24.6 ± 12.0
	Decon. Efficacy (%)	**95.3 ± 4.8**

21880x dilution factor for samples and controls

* Mercury chloride is corrosive and reacted with all the surfaces evaluated, resulting in a fixed residue on the contaminated surface that could not be completely removed by DeconGel.

Table 2. Decontamination efficacies of DeconGel 1101 against mercury(II) oxide (HgO) contaminated stainless steel, carbon steel, and concrete surfaces as determined by residual swipe testing method.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	517.5 ± 33.4
	Residual residual	0.47 ± 0.19
	Decon. Efficacy (%)	**99.9 ± 11.5**
Carbon Steel	Control	511.4 ± 22.6
	Residual	0.67 ± 0.27
	Decon. Efficacy (%)	**99.9 ± 7.4**
Concrete	Control	490.6 ± 32.3
	Residual	1.21 ± 0.91
	Decon. Efficacy (%)	**99.8 ± 5.4**

21880x dilution factor for samples and controls

NOTES:

- ASTM method E1728-03, a standardized swipe testing method used for sampling of inorganic contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes (pre-wetted with DI H_2O) were utilized in this swipe testing method.

- Standardized EPA SW-846 Sampling Method 3005A "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy" was followed as a guideline to prepare all samples and associated controls. When necessary, digestion methods were customized by increasing hydrochloric and nitric acid concentrations from 25% to 35% wt, and/or by heating samples to higher temperatures using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC) to afford the complete digestion of the inorganic contaminants. All samples, controls, and standards were prepared using the same dissolution solution and experimental conditions to ensure both correct instrument calibration and accurate analytical results.

- ICP-OES instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of inorganic compounds. Standardized EPA SW-846 Analytical Method 6010C "Inductively Coupled Plasma-Atomic Emission Spectrometry" was followed as a guideline to prepare all samples and controls.

- Mercury chloride underwent a chemical reaction with stainless steel and carbon steel surfaces (Figure 1) which prevented some contamination from being sampled (swipe testing) and encapsulated into dry DeconGel. Nevertheless DeconGel showed acceptable decontamination efficacy against loose mercury chloride contamination.

- To ensure accurate determination of DeconGel decontamination efficacy, calibration standards of the analyte of interest were prepared using either a sufficiently pure analyte or an appropriate ICP-MS Standard (Ricca Chemical Company; Arlington, TX); the respective standards were diluted to a known concentration (ppm) using the same digestion method as the one used for samples and controls. Instrument blank controls were DI H_2O ($\geq$17 M-Ohm).

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.05 g contaminant was evenly applied on 1) stainless steel (commercial grade, surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24-48 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipes were suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% deionized (DI) H_2O) for 24 h and were then analyzed via ICP-OES (see below).

Control Method

For Swipe Controls, 0.05 g of contaminant was evenly applied on 1) stainless steel (commercial grade, surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O) for 24 h and analyzed via ICP-OES (see below).

Reagents and Standards

Reagent grade Mercury(II) Chloride, $HgCl_2$, (CAS# 7487-94-7, Fisher Scientific; Fair Lawn, NJ) was used as received.

Reagent grade Mercury(II) Oxide, HgO, (CAS # 21508-53-2, Fisher Scientific; Fair Lawn, NJ), was used as received.

1000 ppm calibration standards were prepared using reagent grade HgO and $HgCl_2$ in freshly prepared aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O). DI H_2O ($\geq$ 17 M-Ohm) was used as the blank sample.

Analytical Instrumentation

A Thermo ICP-OES instrument model radial iCap 6300 was used to determine mercury compound concentration (ppm) of all samples and controls using a freshly prepared 1000 ppm calibration standard.

Analyte (mercury) analyzed at 184.9 nm

Pump Speed: 0.5 mL/min

APPLICATION INSTRUCTIONS FOR END-USERS:

Use product directly as is from container. DO NOT DILUTE. Masking or painters tape can be applied along one edge of the area that is to be decontaminated to aid creating a peeled edge to grip for peeling the dried film. Apply DeconGel using a paint brush, a trowel, a handheld sprayer, or an industrial grade sprayer (use DeconGel 1120 or 1121 for spray application).

The thickness of the gel and the number of coats is dictated by the surface to be decontaminated. Coating thickness required for good peel characteristics varies with substrate and generally increases with substrate porosity. It is recommended that first time customers test DeconGel on a small sample area to confirm the required film thickness and dry time for their specific application. If the film is difficult to peel, please apply an additional coat. A razor blade is useful to start the peel. Lay the blade nearly flat and fillet the edge of the film to create a tab that can be pulled. For surfaces that the gel adheres to well, such as concrete, 12" – 24" strips can be cut in the film resulting in less force being required to peel the film.

- Let DeconGel dry for 24 hours

Dry time will vary depending on humidity, temperature, air flow and thickness of the DeconGel. This can take from as little time as an hour for thin coats in a dry environment with plenty of airflow, to overnight or longer if thicker coats are applied in humid environments. Dry times exceeding 24 hours may sometimes be required for good peel performance on bare concrete, wood and other highly porous substrates and substrates with deep cracks or grooves. However, 18-24 hrs is often sufficient dry time on good quality concrete. It is recommended that users test a small area to determine drying time prior to applying DeconGel for an entire job. Supplemental heat or air circulation will accelerate DeconGel's drying time for any job.

- Peel DeconGel off the surface by starting from one of the edges

When dry, the product locks the contaminants into a polymer matrix. The film containing the encapsulated contamination can then be peeled. DeconGel peels from most non-porous and porous hard surfaces if the dried film is thick enough. If the film is difficult to peel, add another coat, let dry, and peel. In most cases the DeconGel will come off in a single sheet but for odd shaped surfaces you may be required to score DeconGel in order to be able to peel it off.

- Dispose of the dried DeconGel in accordance with the local, state and Federal disposal regulations of the contaminant/substance you are removing. DeconGel itself has no special disposal restrictions.

For more information on application techniques visit our website: www.decongel.com

6.44. End-User Report for Nickel Compounds (Nickel Nitrate)

TITLE: Surface Decontamination of Nickel Compounds (Nickel Nitrate ($Ni(NO_3)_2$)) by DeconGel 1101

AUTHOR: Andreas Mylonakis PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with nickel nitrate ($Ni(NO_3)_2$) using ICP-OES (Inductively Coupled Plasma-Optical Emission Spectroscopy) following Environmental Protection Agency (EPA) SW-846 Methods 3005A (sampling) and 6010C (analysis).

HAZARDOUS MATERIALS RELEVANCE: Nickel is corrosion-resistant and is used in alloys and platings, and in the manufacturing of rechargeable batteries, magnets, and coins .Nickel and its compounds can cause contact allergy and are believed to be carcinogenic. Nickel nitrate was chosen as a representative nickel compound for evaluating DeconGel's efficacy; DeconGel is expected to have similar efficacy towards the wide range of nickel compounds.

HIGHLIGHTS:

- Excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with nickel compounds facilitating encapsulation/emulsification of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 98.3% (on concrete) to 99.3% (on carbon steel) to 99.9% (on stainless steel) as determined by residual swipe analysis

- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of inorganic compounds in aqueous samples. When necessary, experimental methods were customized to facilitate complete dissolution of inorganic contaminants, and to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Table 1 shows the decontamination efficacies of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with nickel nitrate as determined by residual swipe testing.

Table 1. Decontamination efficacy of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with $Ni(NO_3)_2$ as determined by the residue swipe testing method.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	565.7 ± 7.3
	Residual	0.67 ± 0.22
	Decon. Efficacy (%)	**99.9 ± 7.3**
Carbon Steel	Control	574.2 ± 37.4
	Residual	3.99 ± 0.63
	Decon. Efficacy (%)	**99.3 ± 8.5**
Concrete	Control	533.7 ± 17.9
	Residual	9.14 ± 2.72
	Decon. Efficacy (%)	**98.3 ± 4.6**

2280x dilution factor for samples and controls

NOTES:

- ASTM method E1728-03 (a standardized swipe testing method used for the sampling of inorganic contaminants) was the integral method used to accurately evaluate DeconGel's decontamination efficacy. GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes (pre-wetted with DI H_2O) were utilized in this swipe testing method.

- Standardized EPA SW-846 Sampling Method 3005A "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy" was followed as a guideline to prepare all samples and controls. Because potassium ferricyanide reacts violently with acid releasing poisonous cyanide gas, all samples were digested solely in DI H2O (≥ 17 M-Ohm). To reduce the viscosity of samples, a 1:100 dilution in water took place on all samples and controls after ample soaking and vigorous shaking to incorporate any contaminant contained within swipes and to ensure full dissolution of the gel. All samples, controls, and standards were prepared using the same dissolution solution and experimental conditions to ensure both correct instrument calibration and accurate analytical results.

- To ensure accurate determination of DeconGel decontamination efficacy, calibration standards of the analyte of interest were prepared using either a sufficiently pure analyte or an appropriate ICP-MS Standard (Ricca Chemical Company; Arlington, TX); the respective standards were diluted to a known concentration (ppm) using the same digestion method as that used for samples and controls. Instrument blank controls were DI H_2O ($\geq$17 M-Ohm).

- ICP-OES instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of inorganic compounds. Standardized EPA SW-846 Analytical Method 6010C "Inductively Coupled Plasma-Atomic Emission Spectrometry" was followed as a guideline to prepare all samples and controls.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.05 g of contaminant was evenly applied on 1) stainless steel (commercial grade, surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24-48 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% deionized (DI) H_2O) for 24 hrs and were then analyzed via ICP-OES (see below).

Controls Methods

For Swipe Control samples, 0.05 g of $Ni(NO_3)_2$ was evenly applied on 1) stainless steel (commercial grade, surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 100 mL aqueous acidic solution (15% HCl, 15% HNO_3, 70% DI H_2O) for 24 h and analyzed via ICP-OES (see below).

Reagents and Standards

Reagent Grade Nickel Nitrate, $Ni(NO_3)_2$, (CAS# 13478-00-7, Fisher Scientific; Fair Lawn, NJ), was used as received.

A 1000 ppm calibration standard was prepared using reagent grade powdered $Ni(NO_3)_2$ and freshly prepared aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O). DI H_2O ($\geq$ 17 M-Ohm) was used as the blank sample.

Analytical Instrumentation

A Thermo ICP-OES instrument model radial iCap 6300 was used to determine $Ni(NO_3)_2$ concentration (ppm) of all samples and controls using a freshly prepared 1000 ppm calibration standard.

Analyte (nickel) analyzed at 221.6 nm

Pump Speed: 0.5 mL/min

APPLICATION INSTRUCTIONS FOR END-USERS:

Use product directly as is from container. DO NOT DILUTE. Masking or painters tape can be applied along one edge of the area that is to be decontaminated to aid creating a peeled edge to grip for peeling the dried film. Apply DeconGel using a paint brush, a trowel, a handheld sprayer, or an industrial grade sprayer (use DeconGel 1120 or 1121 for spray application).

The thickness of the gel and the number of coats is dictated by the surface to be decontaminated. Coating thickness required for good peel characteristics varies with substrate and generally increases with substrate porosity. It is recommended that first time customers test DeconGel on a small sample area to confirm the required film thickness and dry time for their specific application. If the film is difficult to peel, please apply an additional coat. A razor blade is useful to start the peel. Lay the blade nearly flat and fillet the edge of the film to create a tab that can be pulled. For surfaces that the gel adheres to well, such as concrete, 12" – 24" strips can be cut in the film resulting in less force being required to peel the film.

- Let DeconGel dry for 24 hours

Dry time will vary depending on humidity, temperature, air flow and thickness of the DeconGel. This can take from as little time as an hour for thin coats in a dry environment with plenty of airflow, to overnight or longer if thicker coats are applied in humid environments. Dry times exceeding 24 hours may sometimes be required for good peel performance on bare concrete, wood and other highly porous substrates and substrates with deep cracks or grooves. However, 18-24 hrs is often sufficient dry time on good quality concrete. It is recommended that users test a small area to determine drying time prior to applying DeconGel for an entire job. Supplemental heat or air circulation will accelerate DeconGel's drying time for any job.

- Peel DeconGel off the surface by starting from one of the edges

When dry, the product locks the contaminants into a polymer matrix. The film containing the encapsulated contamination can then be peeled. DeconGel peels from most non-porous and porous hard surfaces if the dried film is thick enough. If the film is difficult to peel, add another coat, let dry, and peel. In most cases the DeconGel will come off in a single sheet but for odd shaped

surfaces you may be required to score DeconGel in order to be able to peel it off.

- Dispose of the dried DeconGel in accordance with the local, state and Federal disposal regulations of the contaminant/substance you are removing. DeconGel itself has no special disposal restrictions.

For more information on application techniques visit our website: www.decongel.com

6.45. End-User Report for Organic Acids (Benzoic Acid)

TITLE: Surface Decontamination of Organic Acids (Benzoic Acid) by DeconGel 1101

ANALYST: Geoff Nadolski PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel formulation 1101 on stainless steel, aluminum, and concrete surfaces contaminated with Organic Acids (benzoic acid (BA)) using LC/MS (Liquid Chromatography/Mass Spectrometry) following Environmental Protection Agency (EPA) SW-846 Methods 3500C (Sampling) and 8321B (Analysis).

HAZARDOUS MATERIALS RELEVANCE: BA is an aromatic carboxylic acid used as a food preservative and as industrial chemical feedstock used to prepare flavors, insect repellents, and plasticizers. As with other organic acids, benzoic acid is acidic, an irritant, and can be corrosive to sensitive tissues including mucous membranes and the gastrointestinal tract. Benzoic acid was chosen as a representative organic acid; DeconGel is expected to have similar efficacy towards the wide range of organic acids.

SUMMARY RESULTS:

- Excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with organic acids (benzoic acid) resulting in encapsulation of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 98.4% (on concrete) to 99.7% (on stainless steel) to 99.8% (on aluminum) for BA determined by residual swipe analysis.

- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of organic compounds dissolved in a suitable solvent able to completely solvate BA as well as DeconGel components. When deemed necessary, experimental methods were customized to afford complete dissolution of organic contaminants. Additionally, analytical methods and associated equipment (LC column, LC gradient program, MS sample ionization parameters) were appropriately developed to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 shows the decontamination efficacies of DeconGel 1101 on stainless steel, aluminum, and concrete surfaces contaminated with Organic Acids (BA) as determined by residual swipe testing.

Table 1. Decontamination efficacy of DeconGel 1101 on BA Organic Acid contaminated stainless steel, aluminum, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	426.77 ± 1.85
	Residual	1.17 ± 0.06
	Decon. Efficacy (%)	**99.7 ± 0.01**
Aluminum	Control	427.01 ± 1.61
	Residual	1.07 ± 0.04
	Decon. Efficacy (%)	**99.8 ± 0.0**
Concrete	Control	350.63 ± 4.19
	Residual	5.65 ± 0.40
	Decon. Efficacy (%)	**98.4 ± 0.10**

1571x dilution factor for samples and controls

NOTES:

- ASTM method E1728-03, a standardized swipe testing method used for sampling of contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. Air-dried GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes (pre-wetted with DMSO immediately prior to performing swipe analysis) were utilized in this swipe testing method.
- Standardized EPA SW-846 Sampling Method 3500C "Organic Extraction and Sample Preparation" was followed as a guideline to prepare all samples and controls. All samples, controls, and standards were prepared using the same solvent and appropriate experimental conditions to ensure accurate and LC/MS instrumental analysis.
- LC/MS instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of organic compounds. Standardized EPA SW-846 Analytical Method 8321B "Solvent-Extractable Nonvolatile Compounds by High-Performance Liquid Chromatography/Thermospray/Mass Spectrometry (HPLC/TS/MS) or Ultraviolet (UV) Detection" was followed as a guideline to prepare all samples and controls.

- To ensure accurate determination of DeconGel decontamination efficacy, a standard curve of the analyte of interest was prepared using sufficiently pure analyte; the respective standards were diluted to a known concentration (ppm) using the same solvent as used for samples and controls.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.035 g of contaminant was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) aluminum (commercial grade, surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC) wetted with DMSO (2 mL) immediately prior to swipe analysis. Swipe and gel samples were suspended in 50 mL DMSO for 24 h. Samples were then analyzed via LC/MS (see below).

Controls Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) aluminum (commercial grade, surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC) wetted with DMSO (2 mL) immediately prior to swipe analysis. Swipe samples were suspended in 50 mL DMSO for 24 h and analyzed via LC/MS (see below).

Reagents and Standards

Benzoic acid (BA) (CAS# 65-85-0, Fisher Scientific; Fair Lawn, NJ) was used as received.

Analytical Instrumentation

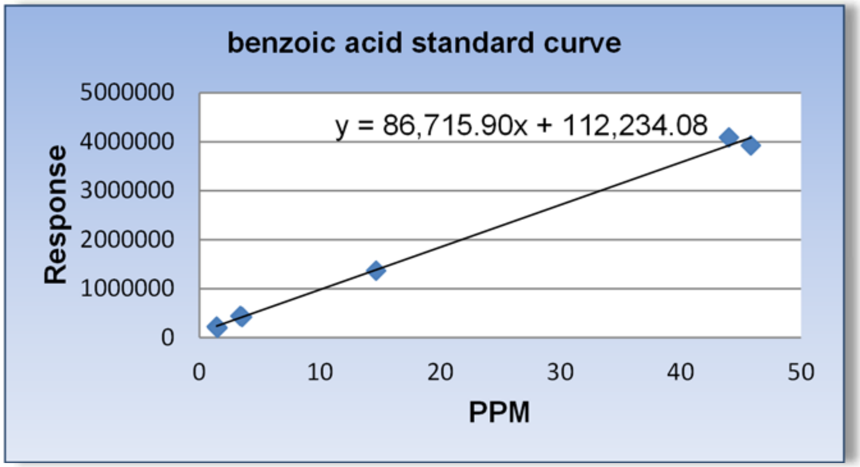
A Thermo LCQ LC/MS with autosampler in positive mode was used to determine BA concentration (ppm, wt/wt) of all samples and controls, using a Grace Davison (Deerfield, IL) C18-Select analytical column (150 x 4.6 mm, 5 um).

A 8-point standard curve derived from three independently prepared stock solutions was prepared using DMSO as the working solvent.

LC method using A= water (0.1% formic acid), B=acetonitrile (0.1% formic acid); start at 95%A to 3 min at 70%A, to 6 min at 50%A, to 12 min at 30%A, to 15 min at 90%B, to 16 min at 100%B, hold until 22 min at 100% B, to 23 min at 95%A, hold until 24 min.

BA LC/MS data: 9.7 min; lambda max = 245, 272 nm; M^+ = 122.

Figure 1. BA Standard Calibration Curve



APPLICATION INSTRUCTIONS FOR END-USERS:

Use product directly as is from container. DO NOT DILUTE. Masking or painters tape can be applied along one edge of the area that is to be decontaminated to aid creating a peeled edge to grip for peeling the dried film. Apply DeconGel using a paint brush, a trowel, a handheld sprayer, or an industrial grade sprayer (use DeconGel 1120 or 1121 for spray application).

The thickness of the gel and the number of coats is dictated by the surface to be decontaminated. Coating thickness required for good peel characteristics varies with substrate and generally increases with substrate porosity. It is recommended that first time customers test DeconGel on a small sample area to confirm the required film thickness and dry time for their specific application. If the film is difficult to peel, please apply an additional coat. A razor blade is useful to start the peel. Lay the blade nearly flat and fillet the edge of the film to create a tab that can be pulled. For surfaces that the gel adheres to well, such as concrete, 12" – 24" strips can be cut in the film resulting in less force being required to peel the film.

- Let DeconGel dry for 24 hours

Dry time will vary depending on humidity, temperature, air flow and thickness of the DeconGel. This can take from as little time as an hour for thin coats in a dry environment with plenty of airflow, to overnight or longer if thicker coats are applied in humid environments. Dry times exceeding 24 hours may sometimes be required for good peel performance on bare concrete, wood and other highly porous substrates and substrates with deep cracks or grooves. However, 18-24 hrs is often sufficient dry time on good quality concrete. It is recommended that users test a small area to determine drying time prior to applying DeconGel for an entire job. Supplemental heat or air circulation will accelerate DeconGel's drying time for any job.

- Peel DeconGel off the surface by starting from one of the edges

When dry, the product locks the contaminants into a polymer matrix. The film containing the encapsulated contamination can then be peeled. DeconGel peels from most non-porous and porous hard surfaces if the dried film is thick enough. If the film is difficult to peel, add another coat, let dry, and peel. In most cases the DeconGel will come off in a single sheet but for odd shaped surfaces you may be required to score DeconGel in order to be able to peel it off.

- Dispose of the dried DeconGel in accordance with the local, state and Federal disposal regulations of the contaminant/substance you are removing. DeconGel itself has no special disposal restrictions.

For more information on application techniques visit our website: www.decongel.com

6.46. End-User Report for Organic Bases (Triethylamine)

TITLE: Surface Decontamination of Organic Bases (Triethylamine) by DeconGel 1101

ANALYST: Geoff Nadolski PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel formulation 1101 on stainless steel, aluminum, and concrete surfaces contaminated with Organic Bases (Triethylamine (TEA)) using GC/MS (Gas Chromatography/Mass Spectrometry) following Environmental Protection Agency (EPA) SW-846 Methods 3500C (Sampling) and 8270C (Analysis).

HAZARDOUS MATERIALS RELEVANCE: Tertiary amine bases such as TEA are used in the chemical industry as acid scavengers and are used to prepare quaternary ammonium compounds for the textile/dye industries. TEA is volatile and irritating to mucous membranes and skin and possesses an offensive fishy odor. TEA was chosen as a representative organic base compound; DeconGel is expected to have similar efficacy towards the full range of organic bases (amines).

SUMMARY RESULTS:

- Excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with organic bases (TEA) resulting in encapsulation of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 99.7% (on concrete) to 99.9% (on stainless steel) to 99.9% (on aluminum) for TEA as determined by residual swipe analysis.

- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of organic compounds dissolved in a suitable solvent able to completely solvate triethylamine as well as DeconGel components. When deemed necessary, experimental methods were customized to afford complete dissolution of organic contaminants. Additionally, analytical methods and associated equipment (GC column, GC temperature gradient program, MS sample ionization parameters) were appropriately utilized to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 shows the decontamination efficacies of DeconGel 1101 on stainless steel, aluminum, and concrete surfaces contaminated with Organic Bases (TEA) as determined by residual swipe testing.

Table 1. Decontamination efficacy of DeconGel 1101 on Triethylamine Organic Base contaminated stainless steel, aluminum, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	295.48 ± 1.07
	Residual	0.30 ± 0.01
	Decon. Efficacy (%)	**99.9 ± 0.01**
Aluminum	Control	295.47 ± 0.80
	Residual	0.28 ± 0.004
	Decon. Efficacy (%)	**99.9 ± 0.01**
Concrete	Control	186.42 ± 0.84
	Residual	0.65 ± 0.01
	Decon. Efficacy (%)	**99.7 ± 0.07**

608x dilution factor for samples and controls

NOTES:

- ASTM method E1728-03, a standardized swipe testing method used for sampling of contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. Air-dried GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes were utilized in this swipe testing method.

- Standardized EPA SW-846 Sampling Method 3500C "Organic Extraction and Sample Preparation" was followed as a guideline to prepare all samples and controls. All samples, controls, and standards were prepared using the same solvent and appropriate experimental conditions to ensure accurate and GC/MS instrumental analysis.

- GC/MS instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of organic compounds. Standardized EPA SW-846 Analytical Method 8270C "SEMIVOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)" was followed as a guideline to prepare all samples and controls.

- To ensure accurate determination of DeconGel decontamination efficacy, a standard curve of the analyte of interest was prepared using sufficiently pure analyte; the respective standards were diluted to a known concentration (ppm) using the same solvent as used for samples and controls.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 90 uL of contaminant was evenly applied via brushing on 1) aluminum (commercial grade, surface area: 56.3 cm^2), 2) stainless steel (commercial grade, surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe and gel samples were suspended in 50 mL 60% methanol in water for 24 h. Samples were then analyzed via GC/MS (see below).

Controls Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied via brushing on 1) aluminum (surface area: 56.3 cm^2), 2) stainless steel (surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 50 mL 60% methanol in water for 24 h and analyzed via GC/MS (see below).

Reagents and Standards

Triethylamine (TEA) (CAS# 121-44-8, Fisher Scientific; Fair Lawn, NJ) was used as received.

Analytical Instrumentation

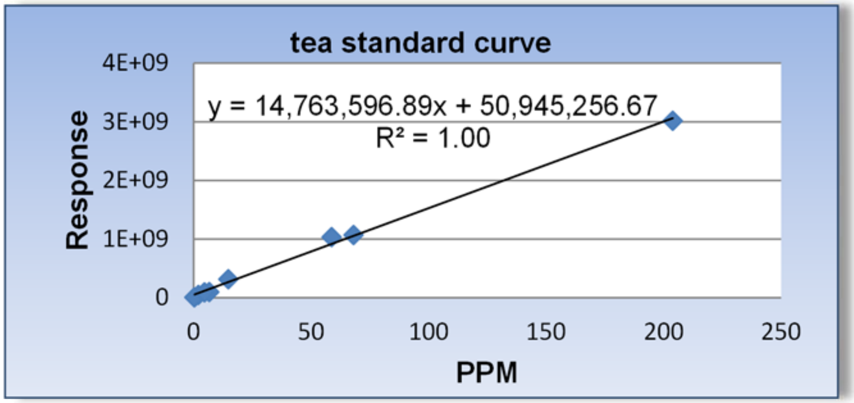
A Thermo DSQII GC/MS with autosampler in positive mode was used to determine glutaraldehyde concentration (ppm, wt/wt) of all samples and controls, using a Restek (Bellefonte, PA) Rtx-5 capillary column (30 m x 0.25 mm, 0.5 um).

A 7-point standard curve derived from three independently prepared stock solutions was prepared using DMSO as the working solvent. The calibration curve exhibits a curve fitting as approximated by the coefficient of determination of linear regression R^2, where $R^2 = 1.0$ (see Figure 1).

GC method: start at 45 °C, hold for 7 min, ramp at 30 °C/min to 320 °C, hold 10 min.

Triethylamine GC/MS data: 3.5 min; $M^+ = 101$.

Figure 1. TEA Standard Calibration Curve



APPLICATION INSTRUCTIONS FOR END-USERS:

Use product directly as is from container. DO NOT DILUTE. Masking or painters tape can be applied along one edge of the area that is to be decontaminated to aid creating a peeled edge to grip for peeling the dried film. Apply DeconGel using a paint brush, a trowel, a handheld sprayer, or an industrial grade sprayer (use DeconGel 1120 or 1121 for spray application).

The thickness of the gel and the number of coats is dictated by the surface to be decontaminated. Coating thickness required for good peel characteristics varies with substrate and generally increases with substrate porosity. It is recommended that first time customers test DeconGel on a small sample area to confirm the required film thickness and dry time for their specific application. If the film is difficult to peel, please apply an additional coat. A razor blade is useful to start the peel. Lay the blade nearly flat and fillet the edge of the film to create a tab that can be pulled. For surfaces that the gel adheres to well, such as concrete, 12” – 24” strips can be cut in the film resulting in less force being required to peel the film.

- Let DeconGel dry for 24 hours

Dry time will vary depending on humidity, temperature, air flow and thickness of the DeconGel. This can take from as little time as an hour for thin coats in a dry environment with plenty of airflow, to overnight or longer if thicker coats are applied in humid environments. Dry times exceeding 24 hours may sometimes be required for good peel performance on bare concrete, wood and other highly porous substrates and substrates with deep cracks or grooves. However, 18-24 hrs is often sufficient dry time on good quality concrete. It is recommended that users test a small area to determine drying time prior to applying DeconGel for an entire job. Supplemental heat or air circulation will accelerate DeconGel’s drying time for any job.

- Peel DeconGel off the surface by starting from one of the edges

When dry, the product locks the contaminants into a polymer matrix. The film containing the encapsulated contamination can then be peeled. DeconGel peels from most non-porous and porous hard surfaces if the dried film is thick enough. If the film is difficult to peel, add another coat, let dry, and peel. In most cases the DeconGel will come off in a single sheet but for odd shaped surfaces you may be required to score DeconGel in order to be able to peel it off.

- Dispose of the dried DeconGel in accordance with the local, state and Federal disposal regulations of the contaminant/substance you are removing. DeconGel itself has no special disposal restrictions.

For more information on application techniques visit our website: www.decongel.com

6.47. End-User Report for Pesticides (DDT)

TITLE: Surface Decontamination of Pesticides (DDT) by DeconGel 1101

ANALYST: Geoff Nadolski PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel formulation 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with Pesticides (dichlorodiphenyltrichloroethane (DDT)) using LC/MS (Liquid Chromatography/Mass Spectrometry) following Environmental Protection Agency (EPA) SW-846 Methods 3500C (Sampling) and 8321B (Analysis).

HAZARDOUS MATERIALS RELEVANCE: Pesticides can be dangerous to consumers and workers during manufacture, transport, or during and after use. DDT is a chlorinated aromatic pesticide that is a restricted persistent organic pollutant. DDT is a reproductive toxicant to birds, possesses endocrine disrupting activity in animals, and is considered moderately hazardous to humans by the World Health Organization (WHO). DDT was chosen as a representative pesticide; DeconGel is expected to have similar efficacy towards the wide range of pesticides.

SUMMARY RESULTS:

- Excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with pesticides (DDT) resulting in encapsulation of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 96.1% (on concrete) to 98.7% (on carbon steel) to 98.7% (on stainless steel) for DDT determined by residual swipe analysis.

- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of organic compounds dissolved in a suitable solvent able to completely solvate DDT as well as DeconGel components. When necessary, experimental methods were customized to afford complete dissolution of organic contaminants. Additionally, analytical methods and associated equipment (LC column, LC gradient program, MS sample ionization parameters) were appropriately utilized to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 shows the decontamination efficacies of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with Pesticides (DDT) as determined by residual swipe testing.

Table 1. Decontamination efficacy of DeconGel 1101 on DDT Pesticide contaminated stainless steel, carbon steel, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	188.49 ± 0.94
	Residual	2.48 ± 0.04
	Decon. Efficacy (%)	**98.7 ± 0.0**
Carbon Steel	Control	190.20 ± 1.73
	Residual	2.51 ± 0.04
	Decon. Efficacy (%)	**98.7 ± 0.0**
Concrete	Control	168.32 ± 0.15
	Residual	6.61 ± 0.15
	Decon. Efficacy (%)	**96.1 ± 0.13**

2200x dilution factor for samples and controls

NOTES:

- ASTM method E1728-03, a standardized swipe testing method used for sampling of contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. Air-dried GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes (pre-wetted with DMSO immediately prior to performing swipe analysis) were utilized in this swipe testing method.

- Standardized EPA SW-846 Sampling Method 3500C "Organic Extraction and Sample Preparation" was followed as a guideline to prepare all samples and controls. All samples, controls, and standards were prepared using the same solvent and appropriate experimental conditions to ensure accurate and LC/MS instrumental analysis.

- LC/MS instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of organic compounds. Standardized EPA SW-846 Analytical Method 8321B "Solvent-Extractable Nonvolatile Compounds by High-Performance Liquid Chromatography/Thermospray/Mass Spectrometry (HPLC/TS/MS) or Ultraviolet (UV) Detection" was followed as a guideline to prepare all samples and controls.

- To ensure accurate determination of DeconGel decontamination efficacy, a standard curve of the analyte of interest was prepared using sufficiently pure analyte; the respective standards were diluted to a known concentration (ppm) using the same solvent as used for samples and controls.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.025 g of contaminant was evenly applied on 1) stainless steel (commercial grade, surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC) wetted with DMSO (2 mL) immediately prior to swipe analysis. Swipe and gel samples were suspended in 50 mL DMSO for 24 h. Samples were then analyzed via LC/MS (see below).

Controls Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC) wetted with DMSO (2 mL) immediately prior to swipe analysis. Swipe samples were suspended in 50 mL DMSO for 24 h and analyzed via LC/MS (see below).

Reagents and Standards

DDT (1,1,1-trichloro-2,2-di(4-chlorophenyl)ethane) (CAS# 50-29-3, Fisher Scientific; Fair Lawn, NJ) was used as received.

Analytical Instrumentation

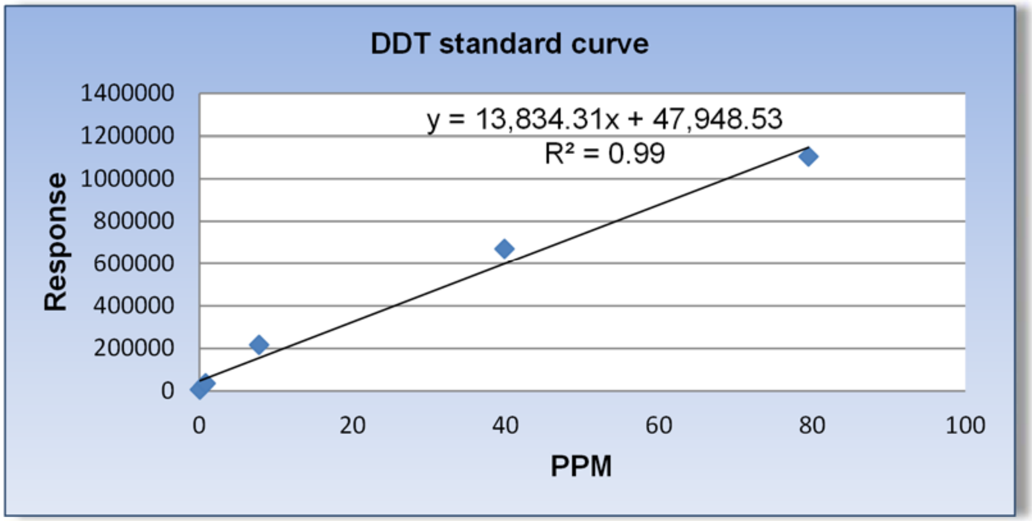
A Thermo LCQ LC/MS with autosampler in positive mode was used to determine DDT concentration (ppm, wt/wt) of all samples and controls, using a Grace Davison (Deerfield, IL) C18-Select analytical column (150 x 4.6 mm, 5 um).

A 6-point standard curve derived from three independently prepared stock solutions was prepared using DMSO as the working solvent. The calibration curve exhibits a curve fitting as approximated by the coefficient of determination of linear regression R^2, where $R^2 = 0.99$ (see Figure 1).

LC method using A= water (0.1% formic acid), B=acetonitrile (0.1% formic acid); start at 90%A to 2 min at 70%A, to 3 min at 50%A, to 5 min at 30%A, to 6 min at 100%B, hold until 16 min at 90% A hold until 17 min.

DDT LC/MS data: 9.9 min; lambda max = 244, 266 nm; $M^+ = 354$.

Figure 1. DDT Standard Calibration Curve



APPLICATION INSTRUCTIONS FOR END-USERS:

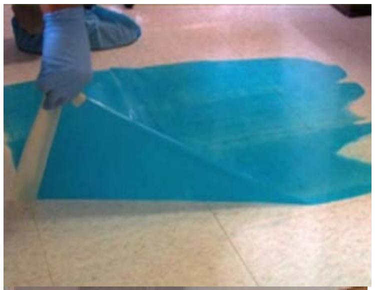
Use product directly as is from container. DO NOT DILUTE. Masking or painters tape can be applied along one edge of the area that is to be decontaminated to aid creating a peeled edge to grip for peeling the dried film. Apply DeconGel using a paint brush, a trowel, a handheld sprayer, or an industrial grade sprayer (use DeconGel 1120 or 1121 for spray application).

The thickness of the gel and the number of coats is dictated by the surface to be decontaminated. Coating thickness required for good peel characteristics varies with substrate and generally increases with substrate porosity. It is recommended that first time customers test DeconGel on a small sample area to confirm the required film thickness and dry time for their specific application. If the film is difficult to peel, please apply an additional coat. A razor blade is useful to start the peel. Lay the blade nearly flat and fillet the edge of the film to create a tab that can be pulled. For surfaces that the gel adheres to well, such as concrete, 12" – 24" strips can be cut in the film resulting in less force being required to peel the film.

- Let DeconGel dry for 24 hours

Dry time will vary depending on humidity, temperature, air flow and thickness of the DeconGel. This can take from as little time as an hour for thin coats in a dry environment with plenty of airflow, to overnight or longer if thicker coats are applied in humid environments. Dry times exceeding 24 hours may sometimes be required for good peel performance on bare concrete, wood and other highly porous substrates and substrates with deep cracks or grooves. However, 18-24 hrs is often sufficient dry time on good quality concrete. It is recommended that users test a small area to determine drying time prior to applying DeconGel for an entire job. Supplemental heat or air circulation will accelerate DeconGel's drying time for any job.

- Peel DeconGel off the surface by starting from one of the edges



When dry, the product locks the contaminants into a polymer matrix. The film containing the encapsulated contamination can then be peeled. DeconGel peels from most non-porous and porous hard surfaces if the dried film is thick enough. If the film is difficult to peel, add another coat, let dry, and peel. In most cases the DeconGel will come off in a single sheet but for odd shaped surfaces you may be required to score DeconGel in order to be able to peel it off.

- Dispose of the dried DeconGel in accordance with the local, state and Federal disposal regulations of the contaminant/substance you are removing. DeconGel itself has no special disposal restrictions.

For more information on application techniques visit our website: www.decongel.com

6.48. End-User Report for Phenolic Compounds (Catechol)

TITLE: Surface Decontamination of Phenolic Compounds (Catechol) by DeconGel 1101

ANALYST: Geoff Nadolski PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel formulation 1101 on aluminum, carbon steel, and concrete surfaces contaminated with Phenolic Compounds (Catechol) using LC/MS (Liquid Chromatography/Mass Spectrometry) following Environmental Protection Agency (EPA) SW-846 Methods 3500C (Sampling) and 8321B (Analysis).

HAZARDOUS MATERIALS RELEVANCE: Catechol is an aromatic phenolic compound used as a precursor to produce pesticides, perfumes, and pharmaceuticals. Catechol shares similar chemical reactivity with phenol – an acidic aromatic irritant and corrosive capable of producing tissue-damaging phenoxyl radicals. Catechol was chosen as a representative phenolic compound for evaluating DeconGel's efficacy; DeconGel is expected to have similar efficacy towards the wide range of phenolic compounds.

SUMMARY RESULTS:

- Excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with phenolic compounds (catechol) resulting in encapsulation of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 97.3% (on concrete) to 99.2% (on carbon steel) to 99.2% (on aluminum) for Catechol determined by residual swipe analysis.

- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of organic compounds dissolved in a suitable solvent able to completely solvate catechol as well as DeconGel components. When necessary, experimental methods were customized to afford complete dissolution of organic contaminants. Additionally, analytical methods and associated equipment (LC column, LC gradient program, MS sample ionization parameters) were appropriately utilized to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Table 1 shows the decontamination efficacies of DeconGel 1101 on aluminum, carbon steel, and concrete surfaces contaminated with Phenolic Compounds (catechol) as determined by residual swipe testing.

Table 1. Decontamination efficacy of DeconGel 1101 on Catechol Phenolic Compound contaminated aluminum, carbon steel, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Aluminum	Control	481.68 ± 1.51
	Residual	4.02 ± 0.40
	Decon. Efficacy (%)	**99.2 ± 0.01**
Carbon Steel	Control	480.42 ± 2.68
	Residual	3.82 ± 0.34
	Decon. Efficacy (%)	**99.2 ± 0.12**
Concrete	Control	433.26 ± 4.02
	Residual	11.90 ± 0.20
	Decon. Efficacy (%)	**97.3 ± 0.21**

1571x dilution factor for samples and controls

NOTES:

- ASTM method E1728-03, a standardized swipe testing method used for sampling of contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. Air-dried GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes (pre-wetted with DMSO immediately prior to performing swipe analysis) were utilized in this swipe testing method.

- Standardized EPA SW-846 Sampling Method 3500C "Organic Extraction and Sample Preparation" was followed as a guideline to prepare all samples and controls. All samples, controls, and standards were prepared using the same solvent and appropriate experimental conditions to ensure accurate and LC/MS instrumental analysis.

- LC/MS instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of organic compounds. Standardized EPA SW-846 Analytical Method 8321B "Solvent-Extractable Nonvolatile Compounds by High-Performance Liquid Chromatography/Thermospray/Mass Spectrometry (HPLC/TS/MS) or

Ultraviolet (UV) Detection" was followed as a guideline to prepare all samples and controls.

- To ensure accurate determination of DeconGel decontamination efficacy, a standard curve of the analyte of interest was prepared using sufficiently pure analyte; the respective standards were diluted to a known concentration (ppm) using the same solvent as used for samples and controls.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.035 g of contaminant was evenly applied on 1) aluminum (commercial grade, surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC) wetted with DMSO (2 mL) immediately prior to swipe analysis. Swipe and gel samples were suspended in 50 mL DMSO for 24 h. Samples were then analyzed via LC/MS (see below).

Controls Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied on 1) stainless steel (commercial grade, surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC) wetted with DMSO (2 mL) immediately prior to swipe analysis. Swipe samples were suspended in 50 mL DMSO for 24 h and analyzed via LC/MS (see below).

Reagents and Standards

Catechol (benzene-1,2-diol) (CAS# 120-80-9, Fisher Scientific; Fair Lawn, NJ) was used as received.

Analytical Instrumentation

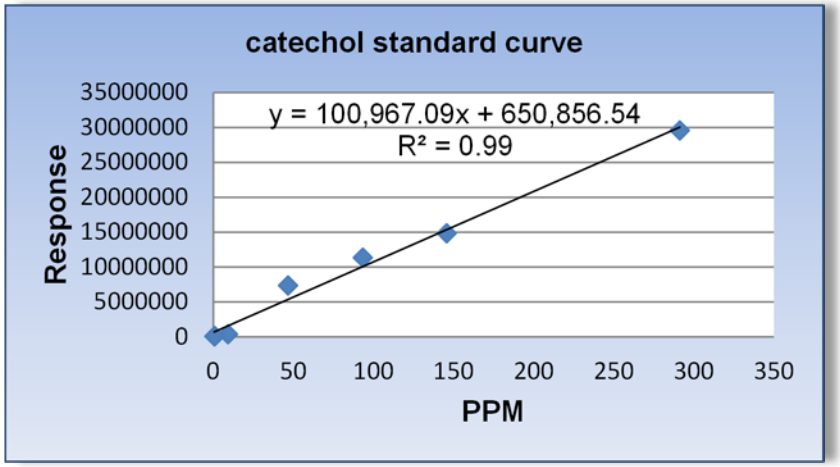
A Thermo LCQ LC/MS with autosampler in positive mode was used to determine catechol concentration (ppm, wt/wt) of all samples and controls, using a Grace Davison (Deerfield, IL) C18-Select analytical column (150 x 4.6 mm, 5 um).

A 7-point standard curve derived from three independently prepared stock solutions was prepared using DMSO as the working solvent. The calibration curve exhibits a curve fitting as approximated by the coefficient of determination of linear regression R^2, where $R^2 = 0.99$ (see Figure 1).

LC method using A= water (0.1% formic acid), B=acetonitrile (0.1% formic acid); start at 95%A to 3 min at 70%A, to 6 min at 50%A, to 12 min at 30%A, to 15 min at 90%B, to 16 min at 100%B, hold until 22 min at 100% B, to 23 min at 95%A, hold until 24 min.

Catechol LC/MS data: 6.9 min; lambda max = 235, 262 nm; $M^+ = 110$.

Figure 1. Catechol Standard Calibration Curve



APPLICATION INSTRUCTIONS FOR END-USERS:

Use product directly as is from container. DO NOT DILUTE. Masking or painters tape can be applied along one edge of the area that is to be decontaminated to aid creating a peeled edge to grip for peeling the dried film. Apply DeconGel using a paint brush, a trowel, a handheld sprayer, or an industrial grade sprayer (use DeconGel 1120 or 1121 for spray application).

The thickness of the gel and the number of coats is dictated by the surface to be decontaminated. Coating thickness required for good peel characteristics varies with substrate and generally increases with substrate porosity. It is recommended that first time customers test DeconGel on a small sample area to confirm the required film thickness and dry time for their specific application. If the film is difficult to peel, please apply an additional coat. A razor blade is useful to start the peel. Lay the blade nearly flat and fillet the edge of the film to create a tab that can be pulled. For surfaces that the gel adheres to well, such as concrete, 12" – 24" strips can be cut in the film resulting in less force being required to peel the film.

- Let DeconGel dry for 24 hours

Dry time will vary depending on humidity, temperature, air flow and thickness of the DeconGel. This can take from as little time as an hour for thin coats in a dry environment with plenty of airflow, to overnight or longer if thicker coats are applied in humid environments. Dry times exceeding 24 hours may sometimes be required for good peel performance on bare concrete, wood and other highly porous substrates and substrates with deep cracks or grooves. However, 18-24 hrs is often sufficient dry time on good quality concrete. It is recommended that users test a small area to determine drying time prior to applying DeconGel for an entire job. Supplemental heat or air circulation will accelerate DeconGel's drying time for any job.

- Peel DeconGel off the surface by starting from one of the edges

When dry, the product locks the contaminants into a polymer matrix. The film containing the encapsulated contamination can then be peeled. DeconGel peels from most non-porous and porous hard surfaces if the dried film is thick enough. If the film is difficult to peel, add another coat, let dry, and peel. In most cases the DeconGel will come off in a single sheet but for odd shaped surfaces you may be required to score DeconGel in order to be able to peel it off.

- Dispose of the dried DeconGel in accordance with the local, state and Federal disposal regulations of the contaminant/substance you are removing. DeconGel itself has no special disposal restrictions.

For more information on application techniques visit our website:
www.decongel.com

6.49. End-User Report for Selenium Compounds (Selenium elemental)

TITLE: Surface Decontamination of Selenium Compounds (Selenium powder) by DeconGel 1101.

AUTHOR: Andreas Mylonakis PhD, Senior Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with Selenium powder using ICP-OES (Inductively Coupled Plasma-Optical Emission Spectroscopy) following Environmental Protection Agency (EPA) SW-846 Methods 3005A (sampling) and 6010C (analysis).

HAZARDOUS MATERIALS RELEVANCE: Selenium is used in industrial applications including steel alloying and rubber compounding. It is also used to produce printers and copier drums. Selenium is hazardous by contact, ingestion, and inhalation as defined by OSHA.[11] Selenium powder was chosen as a representative selenium compound for evaluating DeconGel's efficacy; DeconGel is expected to have similar efficacy towards the wide range of selenium compounds.

HIGHLIGHTS:

- Excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with selenium resulting in encapsulation of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 96.2% (on concrete) to 99.8% (on carbon steel) to 99.2% (on stainless steel) as determined by residual swipe testing.
- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of inorganic compounds in aqueous samples. When necessary, experimental methods were customized to afford complete dissolution of inorganic contaminants, and to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Table 1 shows the decontamination efficacies of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with selenium as determined by residual swipe testing.

Table 1. Decontamination efficacy of DeconGel 1101 on Se contaminated stainless steel, carbon steel, and concrete surfaces as determined by residual swipe testing.

---

[11] Occupational Safety & Health Administration (OSHA); http://www.osha.gov/ (2010)

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	447.8 ± 6.8
	Residual	3.64 ± 4.72
	Decon. Efficacy (%)	**99.2 ± 2.8**
Carbon Steel	Control	455.8 ± 32.1
	Residual	0.88 ± 0.85
	Decon. Efficacy (%)	**99.8 ± 7.1**
Concrete	Control	412.3 ± 20.9
	Residual	15.9 ± 6.2
	Decon. Efficacy (%)	**96.2 ± 7.4**

21880x dilution factor for samples and controls

NOTES:

- ASTM method E1728-03 (a standardized swipe testing method used for sampling of inorganic contaminants) was the integral method used to accurately evaluate DeconGel's decontamination efficacy. GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes (pre-wetted with DI H_2O) were utilized in this swipe testing method.
- Direct chemical analysis of the dried peeled DeconGel samples was also utilized to provide an improved understanding of DeconGel's decontamination efficacy.
- Standardized EPA SW-846 Sampling Method 3005A "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy" was followed as a guideline to prepare all samples and controls. When deemed necessary, digestion methods were customized by increasing hydrochloric and nitric acid concentrations from 25% to 35% wt, and/or by heating samples to higher temperatures using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC) to result in complete digestion of the inorganic contaminants. All samples, controls, and standards were prepared using the same dissolution solution and experimental conditions to ensure both correct instrument calibration and accurate analytical results.
- ICP-OES instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of inorganic compounds. Standardized EPA SW-846 Analytical Method 6010C "Inductively Coupled Plasma-Atomic Emission Spectrometry" was followed as a guideline to prepare all samples and controls.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(contaminant (ppm) of Swipe Control) – (contaminant (ppm) of Residual Swipe)/contaminant (ppm) of Swipe Control] x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.05 g of contaminant was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), and 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24-48 hours. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe were suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% deionized (DI) H_2O) for 24 hours. When necessary, samples were heated to 94°C for 4-14 hours to facilitate complete dissolution of inorganics using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC). Samples were allowed to cool to room temperature and were then analyzed via ICP-OES (see below).

Control Method

For Swipe Control samples, 0.05 g of contaminant was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), and 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O) for 24 hours and analyzed via ICP-OES (see below).

Reagents and Standards

Selenium, Se, 200 mesh, (CAS# 7782-49-2, Fisher Scientific; Fair Lawn, NJ) was used as received.

A 10.0 ppm calibration standard was prepared using reagent grade powdered Se and freshly prepared aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O). DI H_2O was used as the blank sample.

Analytical Instrumentation

A Thermo ICP-OES instrument model radial iCap 6300 was used to determine Se concentration (ppm) of all samples and controls using a freshly prepared 10.0 ppm calibration standard.

Analyte (selenium) analyzed at 196.0 nm

Pump Speed: 0.5 mL/min

APPLICATION INSTRUCTIONS FOR END-USERS:

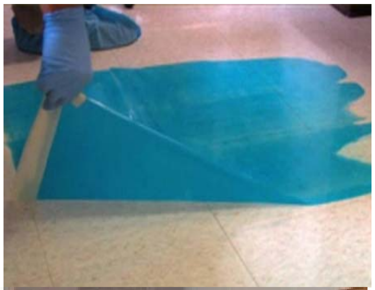
Use product directly as is from container. DO NOT DILUTE. Masking or painters tape can be applied along one edge of the area that is to be decontaminated to aid creating a peeled edge to grip for peeling the dried film. Apply DeconGel using a paint brush, a trowel, a handheld sprayer, or an industrial grade sprayer (use DeconGel 1120 or 1121 for spray application).

The thickness of the gel and the number of coats is dictated by the surface to be decontaminated. Coating thickness required for good peel characteristics varies with substrate and generally increases with substrate porosity. It is recommended that first time customers test DeconGel on a small sample area to confirm the required film thickness and dry time for their specific application. If the film is difficult to peel, please apply an additional coat. A razor blade is useful to start the peel. Lay the blade nearly flat and fillet the edge of the film to create a tab that can be pulled. For surfaces that the gel adheres to well, such as concrete, 12" – 24" strips can be cut in the film resulting in less force being required to peel the film.

- Let DeconGel dry for 24 hours

Dry time will vary depending on humidity, temperature, air flow and thickness of the DeconGel. This can take from as little time as an hour for thin coats in a dry environment with plenty of airflow, to overnight or longer if thicker coats are applied in humid environments. Dry times exceeding 24 hours may sometimes be required for good peel performance on bare concrete, wood and other highly porous substrates and substrates with deep cracks or grooves. However, 18-24 hrs is often sufficient dry time on good quality concrete. It is recommended that users test a small area to determine drying time prior to applying DeconGel for an entire job. Supplemental heat or air circulation will accelerate DeconGel's drying time for any job.

- ➢ Peel DeconGel off the surface by starting from one of the edges



When dry, the product locks the contaminants into a polymer matrix. The film containing the encapsulated contamination can then be peeled. DeconGel peels from most non-porous and porous hard surfaces if the dried film is thick enough. If the film is difficult to peel, add another coat, let dry, and peel. In most cases the DeconGel will come off in a single sheet but for odd shaped surfaces you may be required to score DeconGel in order to be able to peel it off.

- ➢ Dispose of the dried DeconGel in accordance with the local, state and Federal disposal regulations of the contaminant/substance you are removing. DeconGel itself has no special disposal restrictions.

For more information on application techniques visit our website:
www.decongel.com

6.50. End-User Report for Zinc Compounds (Zinc elemental, Zinc Oxide, Zinc Acetate)

TITLE: Surface Decontamination of Zinc Compounds (Elemental Zinc Powder (Zn), Zinc Oxide (ZnO), and Zinc Acetate ($Zn(O_2CCH_3)_2$) by DeconGel 1101.

AUTHOR: Andreas Mylonakis PhD, Senior Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with zinc compounds (elemental zinc powder (Zn), zinc oxide (ZnO), and zinc acetate ($Zn(O_2CCH_3)_2$)) using ICP-OES (Inductively Coupled Plasma-Optical Emission Spectroscopy) following Environmental Protection Agency (EPA) SW-846 Methods 3005A (sampling) and 6010C (analysis).

HAZARDOUS MATERIALS RELEVANCE: Zinc compounds are common industrial compounds and are used as anti-corrosive agents and in the semi conductors and batteries Industries. Zinc compounds are irritants if inhaled and may cause flu-like symptoms known as "metal fume fever". Zinc powder, zinc oxide, and zinc acetate were chosen as representative zinc compound for evaluating DeconGel's efficacy; DeconGel is expected to have similar efficacy towards the wide range of zinc compounds.

HIGHLIGHTS:

- Acceptable to excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with zinc compounds resulting in encapsulation of contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from greater than 86.5% (on concrete) to greater than 66.9% (on carbon steel) to greater than 77.3% (on stainless steel) as determined by residual swipe analysis

- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of inorganic compounds in aqueous samples. When necessary, experimental methods were customized to afford complete dissolution of inorganic contaminants, and to ensure accurate decontamination efficacy determination of DeconGel.

- Due to the corrosive nature of zinc powder, DeconGel surface decontamination was not found to be exceptional on steel (carbon and stainless steel) surfaces due to zinc powder's ability to react with these surfaces, forming a fixed residue that was not able to be completely removed by DeconGel. Nevertheless, DeconGel showed acceptable decontamination efficacy of loose zinc powder contamination from such surfaces.

RESULTS: Tables 1 through 3 show the decontamination efficacies of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with elemental zinc powder (Zn), zinc oxide (ZnO), and zinc acetate ($Zn(O_2CCH_3)_2$) as determined by the residual swipe testing method.

Table 1. Decontamination efficacy of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with zinc powder (Zn) as determined by residual swipe testing.

Swipe Testing (ppm)		**Formulation**
		DeconGel 1101
Stainless Steel*	Control	330.6 ± 14.4
	Residual	74.9 ± 17.7
	Decon. Efficacy (%)	**77.3 ± 6.4**
Carbon Steel*	Control	327.2 ± 7.0
	Residual	108.4 ± 8.0
	Decon. Efficacy (%)	**66.9 ± 2.9**
Concrete	Control	292.1 ± 16.1
	Residual	39.3 ± 13.8
	Decon. Efficacy (%)	**86.5 ± 7.7**

21880x dilution factor for samples and controls

* Zinc powder is corrosive and reacted with all the surfaces evaluated, resulting in a fixed residue on the contaminated surface that could not be completely removed by DeconGel.

Table 2. Decontamination efficacy of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with zinc oxide (ZnO) as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	553.4 ± 33.2
	Residual	0.086 ± 0.075
	Decon. Efficacy (%)	**100.0 ± 0.2**
Carbon Steel	Control	578.8 ± 14.3
	Residual	0.204 ± 0.104
	Decon. Efficacy (%)	**100.0 ± 0.4**
Concrete	Control	543.2 ± 22.4
	Residual	8.52 ± 7.23
	Decon. Efficacy (%)	**98.4 ± 0.2**

21880x dilution factor for samples and controls

Table 3. Decontamination efficacy of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with zinc acetate ($Zn(O_2CCH_3)_2$) as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	468.9 ± 25.3
	Residual	8.58 ± 1.38
	Decon. Efficacy (%)	**98.2 ± 6.0**
Carbon Steel	Control	474.4 ± 17.5
	Residual	7.71 ± 0.14
	Decon. Efficacy (%)	**98.4 ± 3.9**
Concrete	Control	477.1 ± 19.6
	Residual	11.4 ± 5.8
	Decon. Efficacy (%)	**97.6 ± 4.4**

21880x dilution factor for samples and controls

NOTES:

- ASTM method E1728-03 (a standardized swipe testing method used for the sampling of inorganic contaminants) was the integral method used to accurately evaluate DeconGel's decontamination efficacy. GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes (pre-wetted with DI H_2O) were utilized in this swipe testing method.

- Standardized EPA SW-846 Sampling Method 3005A "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy" was followed as a guideline to prepare all samples and controls. Because potassium ferricyanide reacts violently with acid releasing poisonous cyanide gas, all samples were digested solely in DI H2O ($\geq$ 17 M-Ohm). To reduce the viscosity of samples, a 1:100 dilution in water took place on all samples and controls after ample soaking and vigorous shaking to incorporate any contaminant contained within swipes and to ensure full dissolution of the gel. All samples, controls, and standards were prepared using the same dissolution solution and experimental conditions to ensure both correct instrument calibration and accurate analytical results.

- To ensure accurate determination of DeconGel decontamination efficacy, calibration standards of the analyte of interest were prepared using either a sufficiently pure analyte or an appropriate ICP-MS Standard (Ricca Chemical Company; Arlington, TX); the respective standards were diluted to a known concentration (ppm) using the same digestion method as that used for samples and controls. Instrument blank controls were DI H_2O ($\geq$17 M-Ohm).

- ICP-OES instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of inorganic compounds. Standardized EPA SW-846 Analytical Method 6010C "Inductively Coupled Plasma-Atomic Emission Spectrometry" was followed as a guideline to prepare all samples and controls.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.05 g of contaminant was evenly applied on 1) stainless steel (commercial grade, surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24-48 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% deionized (DI) H_2O) for 24 h and were then analyzed via ICP-OES (see below).

Controls Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O) for 24 h and analyzed via ICP-OES (see below).

Reagents and Standards

Zinc metal, Zn, finest powder grade (CAS# 7440-66-6, Fisher Scientific; Fair Lawn, NJ) was used as received.

Zinc oxide, ZnO (CAS# 1314-13-2, Fisher Scientific; Fair Lawn, NJ) was used as received.
Zinc acetate, $Zn(O_2CCH_3)_2$ (CAS# 5970-45-6, Fisher Scientific; Fair Lawn, NJ) was used as received.

A 1000 ppm calibration standard of each contaminant was prepared using the zinc and zinc compounds in freshly prepared aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O). DI H_2O ($\geq$ 17 M-Ohm) was used as the blank sample.

Analytical Instrumentation

A Thermo ICP-OES instrument model radial iCap 6300 was used to determine zinc concentration (ppm) of all samples and controls using a freshly prepared 1000 ppm calibration standard.

Analyte (zinc) analyzed at 213.8 nm

Pump Speed: 0.5 mL/min

APPLICATION INSTRUCTIONS FOR END-USERS:

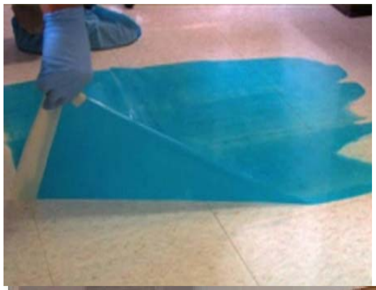
Use product directly as is from container. DO NOT DILUTE. Masking or painters tape can be applied along one edge of the area that is to be decontaminated to aid creating a peeled edge to grip for peeling the dried film. Apply DeconGel using a paint brush, a trowel, a handheld sprayer, or an industrial grade sprayer (use DeconGel 1120 or 1121 for spray application).

The thickness of the gel and the number of coats is dictated by the surface to be decontaminated. Coating thickness required for good peel characteristics varies with substrate and generally increases with substrate porosity. It is recommended that first time customers test DeconGel on a small sample area to confirm the required film thickness and dry time for their specific application. If the film is difficult to peel, please apply an additional coat. A razor blade is useful to start the peel. Lay the blade nearly flat and fillet the edge of the film to create a tab that can be pulled. For surfaces that the gel adheres to well, such as concrete, 12" – 24" strips can be cut in the film resulting in less force being required to peel the film.

➢ Let DeconGel dry for 24 hours

Dry time will vary depending on humidity, temperature, air flow and thickness of the DeconGel. This can take from as little time as an hour for thin coats in a dry environment with plenty of airflow, to overnight or longer if thicker coats are applied in humid environments. Dry times exceeding 24 hours may sometimes be required for good peel performance on bare concrete, wood and other highly porous substrates and substrates with deep cracks or grooves. However, 18-24 hrs is often sufficient dry time on good quality concrete. It is recommended that users test a small area to determine drying time prior to applying DeconGel for an entire job. Supplemental heat or air circulation will accelerate DeconGel's drying time for any job.

➢ Peel DeconGel off the surface by starting from one of the edges



When dry, the product locks the contaminants into a polymer matrix. The film containing the encapsulated contamination can then be peeled. DeconGel peels from most non-porous and porous hard surfaces if the dried film is thick enough. If the film is difficult to peel, add another coat, let dry, and peel. In most cases the DeconGel will come off in a single sheet but for odd shaped surfaces you may be required to score DeconGel in order to be able to peel it off.

- Dispose of the dried DeconGel in accordance with the local, state and Federal disposal regulations of the contaminant/substance you are removing. DeconGel itself has no special disposal restrictions.

For more information on application techniques visit our website: www.decongel.com

Appendix B

7. Individual Technical and End User Reports Navy Priority Contaminant/Substrate Combinations

7.1. Technical Report for Asbestos

TITLE: Surface Decontamination of Asbestos by DeconGel 1101

AUTHOR: Andreas Mylonakis PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Determine the surface decontamination efficacy of DeconGel 1101 on linoleum tile, painted drywall and concrete surfaces contaminated with Asbestos fibers (Chrysotile fibers) using Transmission Electron Microscopy (TEM; ASTM standard test method D6480-05) and Polarized Light Microscopy (PLM; EPA approved method for analysis of asbestos from bulk samples).

HAZARDOUS MATERIALS RELEVANCE: Asbestos is a set of six naturally occurring silicate minerals exploited commercially for their desirable physical properties. They all have in common their long and thin fibrous crystals. The inhalation of asbestos fibers can cause serious illnesses, including malignant lung cancer, mesothelioma, and asbestosis.

HIGHLIGHTS:

- Excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with Asbestos. Decontamination efficacies (wt% based on the residual asbestos fibers on the surface of interest) were 99.9+% from painted drywall, linoleum tile and concrete surfaces.
- Both semi-qualitative (tape lift adhesion sampling method followed by EPA approved PLM analysis) and semi-quantitative (ASTM standard test swipe sampling method followed by TEM analysis) methods have been utilized in these evaluations.
- Application of asbestos contamination on the respective substrate and sampling tests were performed in CBI Polymers labs; Analysis of the samples were performed at independent labs (EMSL Analytical, Centennial, CO, and Bureau Veritas North America, Inc., Kennesaw, GA)

SUMMARY RESULTS:

Tables 1 and 2 show the decontamination efficacies of DeconGel 1101 against asbestos on painted drywall and linoleum tile as determined by the tape lift adhesion test method and EPA approved PLM analysis method and on linoleum tile and concrete as determined by ASTM 6480-05 swipe sampling test method and TEM analysis respectively.

Table 1. Decontamination efficacies of DeconGel 1101 against Asbestos contaminated painted drywall and linoleum tile as determined by the tape lift adhesion sampling and PLM analysis method.

Tape Lift Sampling Testing (10 cm^2 sample area)		Formulation
		DeconGel 1101
Linoleum tile	Before Decon	Trace amounts of chrysotile Asbestos*
	After Decon	None Detected**
	Decon. Efficacy (%)	**100%**
Painted drywall	Before Decon	Trace amounts of chrysotile Asbestos*
	After Decon	None Detected**
	Decon. Efficacy (%)	**100%**

Surface area sampled: 10 cm^2

* Samples for which asbestos is detected under the Polarized Light Microscope at <1%are reported as trace.

** "None Detected" indicates that no asbestos fibers were observed under the Polarized Light Microscope

Table 2. Decontamination efficacies of DeconGel 1101 against Asbestos contaminated linoleum tile and concrete surfaces as determined by ASTM pre-wetted wipe sampling and TEM analysis method.

Swipe Testing Method (100 cm^2 sample area) CONCENTRATION (structures/cm^2)		Formulation
		DeconGel 1101
Linoleum tile*	Before Decon	117000000
	After Decon	44900
	Decon. Efficacy (%)	**99.97%**
Concrete	Before Decon	2670000
	After Decon	None Detected (<1970)
	Decon. Efficacy (%)	**100%**

Surface area sampled: 100 cm^2

* High loading of asbestos fibers was used for these evaluations.

NOTES:

- Chrysotile, which is the most prevalent type of asbestos, was used in these evaluation studies. Chrysotile is a group of fibrous minerals of the serpentine group that have the nominal composition $Mg_3Si_2O_5(OH)_4$ and have the crystal structure of either clinochrysotile, orthochrysotile, or parachrysotile. Most natural chrysotile deviates little from this nominal composition. Chrysotile may be partially dehydrated or magnesium-leached both in nature and in building materials. In some varieties of chrysotile, minor substitution of silicon by Al^{3+} may occur.
- A method that mimics a scaled-down yet real-world setting where decontamination of facilities contaminated with asbestos takes place after the wetting of the substrate to be decontaminated was followed. During these

evaluations the procedure followed includes contamination of the substrate of interest with a solution containing asbestos fibers (chrysotile) dispersed in water followed by partial evaporation of the excess water and the application of DeconGel 1101 on top of the wet asbestos contamination.

- ASTM method D 6480-05, is a standardized procedure used to sample and analyze asbestos fibers where pre-wetted wipes are utilized to sample asbestos from surfaces. This method provides an estimate of the concentration of asbestos reported as the number of asbestos structures per unit area of sampled surface.

- Pre-wetted wipers (Model: TX1084 QuanSat with Vectra Quantex from ITW Texwipe, NJ) wetted with 70% isopropanol and 30% deionized water (sealed-border at the edges) were used for the swipe sample tests.

- Tape lift sampling method is a semi-qualitative quick and reliable sampling test method for analysis and monitoring of asbestos contamination. Analysis of the amount of asbestos that has been sampled with this type of sampling method is performed with polarized light microscopy which is an EPA approved method for analysis of asbestos structures in bulk samples (EPA/600/R-93/116).

- Clear (transparent) 3M Scotch tape type was utilized for the tape lift adhesion sampling method. Before and after decon tape lift samples were sent to an independent lab for analysis of the number of asbestos structures per unit area by polarized light microscopy (PLM).

CALCULATIONS:

Decontamination Efficacy (Swipe Testing Method) =

[(Concentration (structures/cm^2) of Swipe Control) – (Concentration (structures/cm^2) of Residual Swipe)/ Concentration (structures/cm^2) of Swipe Control] x 100%

MATERIALS AND METHODS:

Asbestos solution

Asbestos fibers (Chrysotile, SPI Supplies / Structure Probe, Inc, West Chester, PA) were dispersed in Deionized Water (DI, ≥17MOhm) utilizing a mixer. 0.0971 g of Asbestos fibers were dispersed in 89.3 g of DI water (0.1 wt%).

Application of Asbestos solution on the substrates

Dispersed asbestos fibers in water were applied on top of the surface of interest on a predetermined spot (10cm^2 surface area for the tape lift sample tests;

$100cm^2$ surface area for the swipe sample tests) and were let to dry up to semi-wet (dampened state) before DeconGel 1101 was applied.

Polarized Light Microscopy (PLM, Analytical Method for Asbestos in Bulk Samples)

Use of EPA/600/R-93/116 satisfies applicable requirements of the USEPA's "Interim Method for the Determination of Asbestos in Bulk Insulation Sample", EPA-600/M4-82-020, December 1982, published as Appendix E to Subpart E of 40CFR763. Bulk samples analyzed by New York State methods follow stratified point counting methods (198.1) or Method 198.6 for PLM non-friable organically bound materials (NYSDOH Lab Code 11645). Percentages are visual estimations of asbestos >10:1 aspect ratio. The reliable limit of quantification of the method is 1%, although asbestos may be qualitatively detected at concentrations less than 1%. Samples for which asbestos is detected at <1% are reported as trace, "<1%". "None Detected" indicates that no asbestos fibers were observed.

SAMPLE AND ANALYSIS METHODS:

Tape lift adhesion sampling followed by PLM analysis (semi qualitative test)

1g of asbestos solution (0.1wt% of asbestos in DI H_2O) was applied on top of the linoleum tiles ($10cm^2$ surface area sampled). 1g of asbestos solution (0.1wt% of asbestos in DI H_2O) was applied on top of painted drywall panels ($10cm^2$ surface area for the swipe sample tests). Linoleum tile panels and painted drywall were sampled before and after decon with Scotch tape. Scotch tape samples were put into tightly sealed pre-labeled plastic containers (free of dust) and sent to Bureau Veritas North America, Inc. Labs (Kennesaw, GA) for analysis by PLM according to an EPA approved method for analysis of asbestos in bulk samples (EPA/600/R-93/116 satisfies applicable requirements of the USEPA's "Interim Method for the Determination of Asbestos in Bulk Insulation Sample", EPA-600/M4-82-020, December 1982, published as Appendix E to Subpart E of 40CFR763).

Swipe sampling with prewetted wipes (ASTM standard method) followed by TEM analysis (semi quantitative test)

5.52g of asbestos solution (0.1wt% of asbestos in DI H_2O) was applied on top of the concrete panels ($100cm^2$ surface area for the swipe sample tests). 10g of asbestos solution (0.1wt% of asbestos in DI H_2O) was applied on top of the linoleum tile panels ($100cm^2$ surface area for the swipe sample tests). Concrete and linoleum tile panels were sampled before and after decon with a pre-wetted wipe (containing 70% Isopropanol/30% Water) according to ASTM 6480-05. Sample wipes were put into tightly sealed pre-labeled plastic containers (free of dust) and sent to EMSL labs for analysis by TEM according to the ASTM standard test method 6480-05 “Standard Test Method for Wipe Sampling of Surfaces, Indirect Preparation, and Analysis for Asbestos Structure Number Concentration by Transmission Electron Microscopy”.

Analytical Instrumentation

Polarized Light Microscopy (PLM, work performed at Bureau Veritas North America, Inc., Kennesaw, GA 30144) was used as the method of analysis of the tape lift adhesion samples (EPA approved method for analysis of asbestos in bulk samples).

Transmission Electron Microscopy (TEM, EMSL Analytical, Centennial, CO) was used as the method of analysis of the swipe samples (ASTM standard test method for sampling and analysis of asbestos contamination on a surface).

7.2. Technical Report for Chromium Compounds

TITLE: Surface Decontamination of Chromium Compound (Potassium Chromate (K_2CrO_4) by DeconGel 1101

ANALYSTS: Geoff Nadolski PhD, Senior Scientist, and Andreas Mylonakis PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel formulation 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with Chromium (K_2CrO_4) using ICP-OES (Inductively Coupled Plasma-Optical Emission Spectroscopy) following Environmental Protection Agency (EPA) SW-846 Methods 3005A (sampling) and 6010C (analysis).

HAZARDOUS MATERIALS RELEVANCE: Potassium chromate is a strong oxidizing agent and is used as a chemical indicator for chloride ion content. Potassium chromate is very toxic and may be fatal if swallowed, and can cause cancer on inhalation. Potassium chromate was chosen as a representative chromium compound for evaluating DeconGel's efficacy; DeconGel is expected to have similar efficacy towards the wide range of chromium compounds.

SUMMARY RESULTS:

- As seen in Table 1, excellent to acceptable surface decontamination was achieved by applying DeconGel 1101 onto contaminated surfaces, resulting in encapsulation of Chromium contaminant by DeconGel's active components. Decontamination efficacies of poured DeconGel 1101 ranged from 91.9% (on concrete) to 99.2% (on carbon steel) to 99.6% (on stainless steel) as determined by residual swipe analysis.
- In Table 2, excellent to acceptable surface decontamination was achieved by applying DeconGel 1101 onto contaminated surfaces, resulting in encapsulation of Chromium contaminant by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 67.4% (on concrete) to 97.7% (on stainless steel) to 98.9% (on carbon steel) as determined by direct DeconGel analysis.
- Potassium chromate was shown to react with concete surfaces, forming a fixed residue that was not able to be completely removed by DeconGel™. Nevertheless, DeconGel showed acceptable decontamination efficacy of loose potassium chromate contamination from such surfaces.

- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of inorganic compounds in aqueous samples. When deemed necessary, experimental methods were customized to afford complete dissolution of inorganic contaminants, and to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 and 2 show the decontamination efficacies of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with Potassium Chromate (K_2CrO_4) as determined by residual swipe testing and direct DeconGel analysis.

Table 1. Decontamination efficacies of DeconGel 1101 on Potassium Chromate contaminated stainless steel, carbon steel, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	140.5 ± 7.9
	Residual	0.58 ± 0.16
	Decon. Efficacy (%)	**99.6 ± 5.9**
Carbon Steel	Control	144.8 ± 10.4
	Residual	1.23 ± 0.24
	Decon. Efficacy (%)	**99.2 ± 9.6**
Concrete*	Control	144.0 ± 21.3
	Residual	11.7 ± 1.3
	Decon. Efficacy (%)	**91.9 ± 3.5**

2000x dilution factor for samples and controls

* Potassium chromate reacted with concrete surface, resulting in a fixed residue on the contaminated surface that could not be completely removed by DeconGel.

Table 2. Decontamination efficacies of DeconGel 1101 on Potassium Chromate contaminated stainless steel, carbon steel and concrete surfaces as determined by direct DeconGel analysis.

Direct DeconGel Testing (ppm)		**Formulation**
		DeconGel 1101
Stainless Steel	Control	157.5 ± 15.2
	Encapsulated in Gel (non-brushed)	153.9 ± 12.3
	Decon. Efficacy (non-brushed) (%)*	**97.7 ± 11.8**
Carbon Steel	Control	160.9 ± 4.5
	Encapsulated in Gel (non-brushed)	159.1 ± 5.5
	Decon. Efficacy (non-brushed) (%)	**98.9 ± 9.6**
Concrete*	Control	159.6 ± 15.6
	Encapsulated in Gel (non-brushed)	107.6 ± 17.9
	Decon. Efficacy (non-brushed) (%)	**67.4 ± 13.7**

2000x dilution factor for samples and controls

* Potassium chromate reacted with concrete surface, resulting in a fixed residue on the contaminated surface that could not be completely removed by DeconGel.

TECHNICAL OBSERVATIONS:

- Application of a homogenous, thin layer of Chromium contaminant on the respective substrate facilitated an optimized interaction between contaminant and DeconGel, and provided an accurate measure of DeconGel's decontamination efficacy in a scaled-down yet real-world setting. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel. Potassium chromate readily dissolves in aqueous acidic solutions used to prepare all samples and controls (15% HCl, 15% HNO_3, 70% DI H_2O).

- Potassium chromate undergoes a chemical reaction with concrete surface which prevents some contamination from being sampled (swipe testing) and encapsulated into dry DeconGel (direct gel testing). Nevertheless DeconGel showed acceptable decontamination efficacy against loose potassium chromate contamination.

- ASTM method E1728-03, a standardized swipe testing method used for sampling of inorganic contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes (pre-wetted with DI H_2O) were utilized in this swipe testing method.

- Direct chemical analysis of the dried peeled DeconGel samples was also utilized to provide an improved understanding of DeconGel's decontamination efficacy.

- Standardized EPA SW-846 Sampling Method 3005A “Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy” was followed as a guideline to prepare all samples and controls. When deemed necessary, digestion methods were customized by increasing hydrochloric and nitric acid concentrations from 15% to 25% wt, and/or by heating samples to higher temperatures using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC) to afford the complete digestion of the inorganic contaminants. All samples, controls, and standards were prepared using the same dissolution solution and experimental conditions to ensure both correct instrument calibration and accurate analytical results.

- ICP-OES instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of inorganic compounds. Standardized EPA SW-846 Analytical Method 6010C “Inductively Coupled Plasma-Atomic Emission Spectrometry” was followed as a guideline to prepare all samples and controls.

- To ensure accurate determination of DeconGel decontamination efficacy, calibration standards of the analyte of interest were prepared using either a sufficiently pure analyte or an appropriate ICP-MS Standard (Ricca Chemical Company; Arlington, TX); the respective standards were diluted to a known concentration (ppm) using the same digestion method as the one used for samples and controls. Instrument blank controls were DI H_2O ($\geq$17 M-Ω).

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

Decontamination Efficacy (DeconGel Testing) =

(Contaminant (ppm) of DeconGel Direct/Contaminant (ppm) of DeconGel Control) x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.050 g potassium chromate contaminant was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe and gel samples were suspended in 100 mL aqueous acidic solution (15% HCl, 15% HNO_3, 70% deionized (DI) H_2O) for 24 h. When deemed necessary, samples were heated to 94°C for 4-24 h to effectively complete dissolution of inorganics using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC). Samples were allowed to cool to room temperature and were then analyzed via ICP-OES (see below).

Controls Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied on 1) stainless steel (56.3 cm^2), 2) carbon steel (commercial grade, 100 cm^2), or 3) concrete (industrial grade, 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 100 mL aqueous acidic solution (15% HCl, 15% HNO_3, 70% DI H_2O) for 24 h and analyzed via ICP-OES (see below).

For DeconGel Control samples, the respective amount of contaminant and approximately 6.0 g dry DeconGel 1101 was suspended in 100 mL aqueous acidic solution (15% HCl, 15% HNO_3, 70% DI H_2O) for 24 h and analyzed via ICP-OES (see below).

Reagents and Standards

Reagent grade Potassium Chromate, K_2CrO_4, (CAS# 7789-00-6, Fisher Scientific; Fair Lawn, NJ) was used as received.

1000 ppm calibration standards were prepared using reagent grade K_2CrO_4 in freshly prepared aqueous acidic solution (15% HCl, 15% HNO_3, 70% DI H_2O). DI H_2O was used as the blank sample.

Analytical Instrumentation

A Thermo ICP-OES instrument model radial iCap 6300 was used to determine chromium compound concentration (ppm) of all samples and controls using a freshly prepared 1000 ppm calibration standard.

Analyte (chromium) analyzed at 283.5 nm

Pump Speed: 0.5 mL/min

7.3. Technical Report for Crude Oil

TITLE: Surface Decontamination of Crude Oil by DeconGel 1101 and 1102

ANALYSTS: Geoff Nadolski PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVES: Surface decontamination determination of DeconGel formulation 1101 and 1102 on aluminum, stainless steel, and concrete surfaces contaminated with Crude Oil (polyaromatic hydrocarbons fraction) using LC/MS (Liquid Chromatography/Mass Spectrometry) following Environmental Protection Agency (EPA) SW-846 Methods 3500C (Sampling), 8321B (Analysis), and 1654A (Analysis of PAH Content of Oil).

HAZARDOUS MATERIALS RELEVANCE: Crude oil is used throughout the world as an energy source, and is used to provide a broad range of valuable and useful materials. Crude oil and its decomposition products are environmentally persistent toxins, and when released into the environment require extensive remediation typically resulting in the generation of large quantities of toxic contamination. Crude oil is comprised of 0.2-7% polyaromatic hydrocarbons (PAHs), widespread organic pollutants known for their carcinogenic, mutagenic, and tetratogenic properties.

SUMMARY RESULTS:

- As seen in Table 1, excellent surface decontamination was achieved by applying DeconGel 1101 and 1102 both via brushing or pouring (non-brushed) onto contaminated surfaces, resulting in encapsulation of Crude oil contaminant by DeconGel's active components. Decontamination efficacies of poured DeconGel 1101 ranged from 85.9% (on concrete) to 93.9% (on aluminum) to 93.9% (on stainless steel), brushed DeconGel 1101 ranged from 98.4% (on concrete) to 99.0% (on stainless steel) to 99.1% (on aluminum), as determined by residual swipe analysis. Decontamination efficacies of poured DeconGel 1102 ranged from 86.6% (on concrete) to 91.3% (on stainless steel) to 94.1% (on aluminum), brushed DeconGel 1102 ranged from 98.9% (on concrete) to 99.5% (on stainless steel) to 99.6% (on aluminum), as determined by residual swipe analysis.

- In Table 2, excellent to acceptable surface decontamination was achieved by applying DeconGel 1101 onto contaminated surfaces, resulting in encapsulation of Crude oil contaminant by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 84.5% (on concrete) to 95.6% (on stainless steel) to 95.7% (on aluminum) as determined by direct DeconGel analysis. Decontamination efficacies of DeconGel 1102 ranged from 81.7% (on concrete) to 93.5% (on stainless steel) to 93.5% (on aluminum) as determined by direct DeconGel analysis.

- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of organics in polar solvent solvated samples. When necessary, the digestion methods were customized to result in the complete dissolution of the organic contaminants and to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 and 2 show the decontamination efficacies of DeconGel 1101 on stainless steel, aluminum, and concrete surfaces contaminated with Crude oil as determined by residual swipe testing and direct DeconGel analysis.

Table 1. Decontamination efficacies of DeconGel 1101 and 1102 on Crude Oil contaminated stainless steel, aluminum, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation	Formulation
		DeconGel 1101	**DeconGel 1102**
Stainless Steel	Control	213.63 ± 1.60	213.63 ± 1.60
	Residual (non-brushed)	12.88 ± 0.29	18.63 ± 0.33
	Residual (brushed)	2.04 ± 0.02	0.97 ± 0.03
	Decon. Efficacy (non-brushed) (%)	**93.9 ± 0.10**	**91.3 ± 0.16**
	Decon. Efficacy (brushed) (%)	**99.0 ± 0.10**	**99.5 ± 0.10**
Aluminum	Control	215.82 ± 1.07	215.82 ± 1.07
	Residual (non-brushed)	13.16 ± 0.66	19.10 ± 0.41
	Residual (brushed)	2.02 ± 0.01	0.93 ± 0.02
	Decon. Efficacy (non-brushed) (%)	**93.9 ± 0.10**	**94.1 ± 0.34**
	Decon. Efficacy (brushed) (%)	**99.1 ± 0.10**	**99.6 ± 0.10**
Concrete	Control	156.03 ± 1.93	156.03 ± 1.93
	Residual (non-brushed)	22.02 ± 0.26	20.88 ± 0.12
	Residual (brushed)	2.44 ± 0.10	1.79 ± 0.02
	Decon. Efficacy (non-brushed) (%)	**85.9 ± 0.18**	**86.6 ± 0.36**
	Decon. Efficacy (brushed) (%)	**98.4 ± 0.10**	**98.9 ± 0.10**

13133x dilution factor for samples and controls

Table 2. Decontamination efficacies of DeconGel 1101 and 1102 on Crude Oil contaminated stainless steel, aluminum, and concrete surfaces as determined by direct DeconGel analysis.

Direct DeconGel Testing (ppm)		Formulation	
		DeconGel 1101	**DeconGel 1102**
Stainless Steel	Control	220.43 ± 1.96	220.34 ± 1.60
	Encapsulated in Gel (non-brushed)	210.76 ± 0.42	205.95 ± 0.37
	Decon. Efficacy (non-brushed) (%)*	**95.6 ± 0.42**	**93.5 ± 0.33**
Aluminum	Control	220.43 ± 1.96	220.34 ± 1.60
	Encapsulated in Gel (non-brushed)	210.87 ± 0.30	206.06 ± 0.22
	Decon. Efficacy (non-brushed) (%)	**95.7 ± 0.24**	**93.5 ± 0.33**
Concrete	Control	220.43 ± 1.96	220.34 ± 1.60
	Encapsulated in Gel (non-brushed)	186.29 ± 0.55	180.04 ± 0.24
	Decon. Efficacy (non-brushed) (%)	**84.5 ± 0.66**	**81.7 ± 0.58**

13133x dilution factor for samples and controls

* Decontamination Efficacy by direct DeconGel testing was determined only for non-brushed films, such that brushing DeconGel onto contaminated surfaces results in a loss of some portion of contaminant on brushing tool.

TECHNICAL OBSERVATIONS:

- Application of a homogenous, thin layer of Crude oil contaminant on the respective substrate facilitated an optimized interaction between contaminant and DeconGel, and provided an accurate measure of DeconGel's decontamination efficacy in a scaled-down yet real-world setting. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel.

- ASTM method E1728-03, a standardized swipe testing method used for sampling of contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. Air-dried GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes wetted with methanol/DMSO (1:1) solvent were utilized in this swipe testing method.

- Direct chemical analysis of the dried peeled DeconGel samples was also utilized to provide an improved understanding of DeconGel's decontamination efficacy. Decontamination efficacy was determined only for non-brushed (poured) DeconGel films, since brushing DeconGel onto contaminated surfaces resulted in a loss of some portion of contaminant on the brushing tool used, and as such accurate decontamination efficacies for brushed DeconGel 1101 could not be determined. For brushed films, accurate decontamination efficacies were best determined utilizing swipe testing.

- Standardized EPA SW-846 Sampling Method 3500C "Organic Extraction and Sample Preparation" and EPA Method 1654A "PAH Content of Oil by

HPLC/UV) were followed as a guideline to prepare all samples and controls. All samples, controls, and standards were prepared using the same solvent and appropriate experimental conditions to ensure accurate and LC/MS instrumental analysis.

- LC/MS instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of organic compounds. Standardized EPA SW-846 Analytical Method 8321B "Solvent-Extractable Nonvolatile Compounds by High-Performance Liquid Chromatography/Thermospray/Mass Spectrometry (HPLC/TS/MS) or Ultraviolet (UV) Detection" was followed as a guideline to prepare all samples and controls.
- To ensure accurate determination of DeconGel decontamination efficacy, a standard curve of the analyte of interest was prepared using sufficiently pure analyte; the respective standards were diluted to a known concentration (ppm) using the same solvent as used for samples and controls.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

Decontamination Efficacy (DeconGel Testing) =

(Contaminant (ppm) of DeconGel Direct/Contaminant (ppm) of DeconGel Control) x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.10 g (1.0 mL of 5 g/50 mL hexane) Crude oil contaminant was evenly applied via brushing on 1) aluminum (surface area: 56.3 cm^2), 2) stainless steel (surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and then the hexane carrier solvent allowed to evaporate for 20 min. Approximately 6.0 g of DeconGel 1101 or 1102 was either poured or brushed (brushed first in a top-bottom, then in a left-right fashion) onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC) wetted with methanol:DMSO (1:1) solvent (2 mL). Swipe and dried film samples were suspended in 50 mL methanol:DMSO (1:1) for 24 h. Samples were then analyzed via LC/MS (see below).

Control Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied via brushing on 1) aluminum (surface area: 56.3 cm^2), 2) stainless steel (surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC) wetted with methanol:DMSO (1:1) solvent (2 mL). Swipe and dried film samples were suspended in 50 mL methanol:DMSO (1:1) for 24 h. Samples were then analyzed via LC/MS (see below).

For DeconGel Control samples, the respective amount of contaminant and approximately 6.0 g of dry DeconGel1101 or 1102 (pre-poured gel on the respective un-contaminated substrate and let to dry for 24 h) was suspended in 50 mL methanol:DMSO (1:1) for 24 h. Samples were then analyzed via LC/MS (see below).

Reagents and Standards

Crude oil (light, sweet Chevron crude oil sourced Oahu, Hawaii) was suspended in methanol/DMSO (1:1) solvent mixture, sonicated for 10 min, let to stand for 24 h, and then the yellow liquid decanted from insoluble solids used to generate standard curves.

Analytical Instrumentation

A Thermo LCQ LC/MS with autosampler in positive mode was used to determine PAHs (polyaromatic hydrocarbons) fraction concentration (ppm, wt/wt) of all samples and controls, using a Grace Davison (Deerfield, IL) C18-Select analytical column (150 x 4.6 mm, 5 um).

A 7-point standard curve derived from three independently prepared stock solutions (crude oil dissolved in methanol:DMSO (1:1) was prepared.

LC method using A= water (0.1% formic acid), B=acetonitrile (0.1% formic acid); start at 90%A to 2 min at 70%A, to 3 min at 50%A, to 5 min at 30%A, to 6 min at 100%B, hold until 16 min at 90% A hold until 17 min.

Crude oil (PAHs fraction) LC/MS data: 9.2 min; lambda max = 275 nm; no data acquired for MS.

7.4. Technical Report for Copper Compounds (Elemental Copper, Copper (I) Oxide)

TITLE: Surface Decontamination of Copper Compounds (Copper (elemental), Copper (I) Oxide) by DeconGel 1101

ANALYSTS: Geoff Nadolski PhD, Senior Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel 1101 on stainless steel, carbon steel, concrete, Lexan, and rubber surfaces contaminated with elemental copper and copper (I) oxide (Cu_2O) using ICP-OES (Inductively Coupled Plasma-Optical Emission Spectroscopy) following Environmental Protection Agency (EPA) SW-846 Methods 3005A (sampling) and 6010C (analysis).

HAZARDOUS MATERIALS RELEVANCE: Copper and its compounds such as copper (I) oxide are commonly used as components of semiconductors, pigments, fungicides, and as antifouling agents for marine paints. Due to its toxicity toward aquatic organisms, copper is considered an environmentally persistent pollutant. Elemental copper and copper (I) oxide were chosen as representative copper compounds for evaluating DeconGel's efficacy. DeconGel is expected to have similar efficacy towards a wide range of copper compounds.

SUMMARY RESULTS:

- As seen in Tables 1 and 3, excellent surface decontamination was achieved by applying DeconGel 1101 onto contaminated surfaces, resulting in encapsulation of Copper contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 on copper (elemental) ranged from 98.0% (on concrete) to 99.4% (on rubber) to 99.5% (on Lexan) to 99.5% (on carbon steel) to 99.9% (on stainless steel) as determined by residual swipe analysis; decontamination efficacies of DeconGel 1101 on copper (I) oxide ranged from 94.7% (on concrete) to 98.9% (on carbon steel) to 99.7% (on stainless steel) as determined by residual swipe analysis.

- In Tables 2 and 4, excellent to acceptable surface decontamination was achieved by applying DeconGel 1101 onto contaminated surfaces, resulting in encapsulation of Copper contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 on copper elemental ranged from 97.8% (on carbon steel) to 99.9% (on concrete) to 98.3% (on Lexan) to 99.1% (on rubber) to 99.9% (on stainless steel) as determined by direct DeconGel analysis; decontamination efficacies of DeconGel 1101 on copper

(I) oxide ranged from 94.5% (on stainless steel) to 100.5% (on carbon steel) to 74.7% (on concrete) as determined by direct DeconGel analysis.

- Copper (I) oxide was evidenced to react with concete surfaces, forming a fixed residue that was not able to be completely removed by DeconGel. Nevertheless, DeconGel showed acceptable decontamination efficacy of loose copper (I) oxide contamination from such surfaces.
- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of inorganic compounds in aqueous samples. When deemed necessary, experimental methods were customized to afford complete dissolution of inorganic contaminants, and to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1-4 show the decontamination efficacies of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with copper compound (Cu_2O) as determined by residual swipe testing and direct DeconGel analysis.

Table 1. Decontamination efficacies of DeconGel 1101 on Copper (elemental) contaminated stainless steel, carbon steel, concrete, rubber, and Lexan surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		**Formulation**
		DeconGel 1101
Stainless Steel	Control	396.3 ± 16.3
	Residual	0.56 ± 0.59
	Decon. Efficacy (%)	**99.9 ± 8.9**
Carbon Steel	Control	396.1 ± 10.1
	Residual	1.81 ± 0.81
	Decon. Efficacy (%)	**99.5 ± 7.0**
Concrete	Control	352.0 ± 8.2
	Residual	7.0 ± 4.0
	Decon. Efficacy (%)	**98.0 ± 2.9**
Rubber*	Control	61.0 ± 6.0
	Residual	0.34 ± 0.40
	Decon. Efficacy (%)	**99.4 ± 9.9**
Lexan*	Control	59.7 ± 1.2
	Residual	0.28 ± 0.34
	Decon. Efficacy (%)	**99.5 ± 4.9**

2000x dilution factor for samples and controls

*12000x dilution factor for samples and controls

Table 2. Decontamination efficacies of DeconGel 1101 on Copper (elemental) contaminated stainless steel, carbon steel, concrete, rubber, and Lexan surfaces as determined by direct DeconGel analysis.

Direct DeconGel Testing (ppm)		**Formulation**
		DeconGel 1101
Stainless Steel	Control	1477 ± 13
	Residual	1475 ± 91
	Decon. Efficacy (%)	**99.9 ± 6.2**
Carbon Steel	Control	1488 ± 2
	Residual	1455 ± 71.4
	Decon. Efficacy (%)	**97.8 ± 4.8**
Concrete	Control	1523 ± 17
	Residual	1496 ± 82
	Decon. Efficacy (%)	**98.2 ± 5.4**
Rubber*	Control	60.4 ± 5.4
	Residual	59.9 ± 2.0
	Decon. Efficacy (%)	**99.1 ± 9.7**
Lexan*	Control	67.1 ± 4.9
	Residual	65.9 ± 2.6
	Decon. Efficacy (%)	**98.3 ± 8.1**

2000x dilution factor for samples and controls
*12000x dilution factor for samples

Table 3. Decontamination efficacies of DeconGel 1101 on Copper (I) Oxide contaminated stainless steel, carbon steel, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		**Formulation**
		DeconGel 1101
Stainless Steel	Control	526.5 ± 41.9
	Residual	1.50 ± 0.40
	Decon. Efficacy (%)	**99.7 ± 8.7**
Carbon Steel	Control	499.7 ± 33.9
	Residual	5.52 ± 3.53
	Decon. Efficacy (%)	**98.9 ± 6.9**
Concrete*,[1]	Control	55.8 ± 1.4
	Residual	2.95 ± 1.67
	Decon. Efficacy (%)	**94.7 ± 3.9**

2000x dilution factor for samples and controls
[1] 12000x dilution factor for samples
* Copper (I) oxide reacted with concrete surface, resulting in a fixed residue on the contaminated surface that could not be completely removed by DeconGel.

Table 4. Decontamination efficacies of DeconGel 1101 on Copper (I) Oxide contaminated stainless steel, carbon steel, and concrete surfaces as determined by direct DeconGel analysis.

Direct DeconGel Testing (ppm)		**Formulation**		
		DeconGel 1101[a]	**DeconGel 1101[b]**	**DeconGel 1101[c]**
Stainless Steel	Control	1049 ± 12.0	455.0 ± 10.2	430.0 ± 5.9
	Residual	754.3 ± 45.5	387.4 ± 12.7	410.2 ± 17.6
	Decon. Efficacy (%)	**71.9 ± 4.2**	**85.2 ± 4.6**	**94.5 ± 4.1**
Carbon Steel	Control	1049 ± 12.0	476.0 ± 3.2	NC
	Residual	671.8 ± 88.4	478.2 ± 100.4	NC
	Decon. Efficacy (%)	**64.1 ± 8.4**	**100.5 ± 6.0**	**ND**
Concrete*	Control	1049 ± 12.0	728.5 ± 8.5	751.0 ± 2.0
	Residual	783.2 ± 190.4	180.0 ± 38.2	523.4 ± 195.1
	Decon. Efficacy (%)	**74.7 ± 18.1**	**24.7 ± 5.2**	**69.7 ± 26.0**

2000x dilution factor for samples and controls

* Copper (I) oxide reacted with concrete surface, resulting in a fixed residue on the contaminated surface that could not be completely removed by DeconGel.

NC: experiments not conducted

ND: not determined

a. 100 mg of contaminant was applied on the substrate.

b. 50 mg of contaminant was applied on the substrate, 1st application of DeconGel

c. 50 mg of contaminant was applied on the substrate, 2nd application of DeconGel

TECHNICAL OBSERVATIONS:

- Application of a homogenous, thin layer of Copper contaminant on the respective substrate facilitated an optimized interaction between contaminant and DeconGel, and provided an accurate measure of DeconGel's decontamination efficacy in a scaled-down yet real-world setting. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel. Copper (elemental) and copper (I) oxide readily dissolve in aqueous acidic solutions used to prepare all samples and controls (20% HCl, 15% HNO_3, 65% DI H_2O).

- Copper (I) oxide underwent a chemical reaction with the concrete coupon surface, which prevented some contamination from being sampled (swipe testing) and encapsulated into dry DeconGel (direct gel testing). Nevertheless DeconGel showed acceptable decontamination efficacy against loose copper (I) oxide contamination.

- ASTM method E1728-03, a standardized swipe testing method used for sampling of inorganic contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. GhostWipe™

(Environmental Express; Mt. Pleasant, SC) swipes (pre-wetted with DI H_2O) were utilized in this swipe testing method.

- Direct chemical analysis of the dried peeled DeconGel samples was also utilized to provide an improved understanding of DeconGel's decontamination efficacy.
- Standardized EPA SW-846 Sampling Method 3005A "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy" was followed as a guideline to prepare all samples and controls. When deemed necessary, digestion methods were customized by increasing hydrochloric and nitric acid concentrations from 15% to 25% wt, and/or by heating samples to higher temperatures using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC) to afford the complete digestion of the inorganic contaminants. All samples, controls, and standards were prepared using the same dissolution solution and experimental conditions to ensure both correct instrument calibration and accurate analytical results.
- ICP-OES instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of inorganic compounds. Standardized EPA SW-846 Analytical Method 6010C "Inductively Coupled Plasma-Atomic Emission Spectrometry" was followed as a guideline to prepare all samples and controls.
- To ensure accurate determinations of DeconGel decontamination efficacy, calibration standards of the analyte of interest were prepared using either a sufficiently pure analyte or an appropriate ICP-MS Standard (Ricca Chemical Company; Arlington, TX); the respective standards were diluted to a known concentration (ppm) using the same digestion method as the one used for samples and controls. Instrument blank controls were DI H_2O ($\geq$17 M-Ω).

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

Decontamination Efficacy (DeconGel Testing) =

(Contaminant (ppm) of DeconGel Direct/Contaminant (ppm) of DeconGel Control) x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.050 g copper contaminant was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) carbon steel (surface area: 100 cm^2), 3) concrete (industrial grade, surface area: 56.3 cm^2), 4) rubber (surface area: 56.3 cm^2), or 5) Lexan (surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe and gel samples were suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% deionized (DI) H_2O) for 24 h. When deemed necessary, samples were heated to 94°C for 4-24 h to effectively complete dissolution of inorganics using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC). Samples were allowed to cool to room temperature and were then analyzed via ICP-OES (see below).

Controls Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied on 1) stainless steel (56.3 cm^2), 2) carbon steel (commercial grade, 100 cm^2), or 3) concrete (industrial grade, 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O) for 24 h and analyzed via ICP-OES (see below).

For DeconGel Control samples, a respectable amount of contaminant and approximately 6.0 g dry DeconGel 1101 was suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O) for 24 h and analyzed via ICP-OES (see below).

Reagents and Standards

Reagent grade Copper (elemental), (CAS# 7440-50-8, Fisher Scientific; Fair Lawn, NJ) was used as received.

Reagent grade Copper (I) Oxide, Cu_2O, (CAS# 1317-39-1, Fisher Scientific; Fair Lawn, NJ) was used as received.

1000 ppm calibration standards were prepared using reagent grade copper (elemental) and Cu_2O in freshly prepared aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O). DI H_2O was used as the blank sample.

Analytical Instrumentation

A Thermo ICP-OES instrument model radial iCap 6300 was used to determine copper compound concentration (ppm) of all samples and controls using a freshly prepared 1000 ppm calibration standard.

Analyte (copper) analyzed at 327.4 nm

Pump Speed: 0.5 mL/min

7.5. Technical Report for CWA Sulfur Mustard-Simulant (CEES)

TITLE: Surface Decontamination of the Sulfur Mustard Simulant 2-Chloroethyl Ethyl Sulfide (CEES) by DeconGel 1101

ANALYST: Geoff Nadolski PhD, Senior Polymer Scientist, CBI Polymers, Inc.

SUPERVISOR: Garry J. Edgington PhD, Chief Scientist, CBI Polymers, Inc.

OBJECTIVE: Evaluate the decontamination efficacy of DeconGel 1101 on coupon types:

1) Cadmium-plated Steel,
2) Carbon Steel,
3) Chemical Agent Resistant Coating (CARC),
4) Silicone Rubber, and
5) Black Rubber surfaces contaminated with the Sulfur Mustard simulant CEES.

- Experimentation and Reporting conducted following JPEO-CBD Source Document:
  - 2007 Chemical Decontamination Performance Evaluation Testing,
    - Section 6: Panel Contact Test to Determine Contact Hazard,
    - Section 6-E: Panel (Coupon) Extraction Method to Determine Remaining Agent;
- Sensitive (low ppb-level) GC/MS (gas chromatography/mass spectrometry) analytical methods developed.

HAZARDOUS MATERIALS RELEVANCE: Sulfur Mustard, bis-(2-chloroethyl) sulfide, is a powerful vesicant, cytotoxic agent regulated under the 1993 Chemical Weapons Convention (CWC). Sulfur mustard can be deployed by spraying, or more typically distributed via artillery shells and bombs. For experimental testing purposes, the half-mustard 2-chloroethyl ethyl sulfide

(CEES) is used as an effective yet less toxic simulant for sulfur mustard chemical warfare agents.

SUMMARY RESULTS:

- Because none of the highly volatile contaminant CEES was detected for either samples on the non-porous steel surfaces tested, or Positive Control 1, an adequate measure of surface decontamination could not be calculated for Cd-plated and Carbon Steel coupons.

- On porous and/or chemically active coupon surfaces such as CARC and Black and Silicone Rubbers, modest to excellent Surface Decontamination of volatile CEES was realized; even on delicate or difficult-to-clean customized rubber, epoxy, or polyester surfaces, DeconGel is able to effectively remove hydrophobic contaminants through encapsulation/emulsification.

    - Reduction in starting challenge (Percent Efficacies of DeconGel 1101) ranged from 80.5% (on Silicone Rubber) to 97.0% (on Black Rubber) to 99.9% (on CARC) as determined by solvent extraction.

    - DeconGel 1101 Decontamination Efficacies ranged from 28.6% (on CARC) to 54.6% (on Silicone Rubber) to 93.2% (on Black Rubber) as determined by solvent extraction.

- DeconGel 1101 can serve as a partial chemical (vapor) barrier. The vapor barrier effect demonstrates the reduction in the rate of release of agent to the surrounding atmosphere as well as indicating a prolonged residence-time inside the gel layer. An extended residence-time in the gel is predicted to enhance the efficacy of neutralizing agents that are proposed to be employed in future versions of the gel. This reduction in rate of release of agents is proposed to be especially valuable in enhancing neutralization efficacies of CWAs with high reactivity and volatility.

  To support the hypothesis that DeconGel 1101 can serve as a partial chemical (vapor) barrier, additional experimentation ((+)-Control 2) was performed to demonstrate the Chemical Barrier Efficiency of DeconGel 1101.

    - On Black and Silicone Rubber surfaces, DeconGel 1101 Chemical Barrier Efficiencies ranged from 84.7% (on Silicone Rubber) to 85.8% (on Black Rubber) as determined by solvent extraction, indicating a significant chemical surface barrier capability for DeconGel 1101 when applied to porous, chemically active rubber/ epoxy/polyester surfaces able to absorb and entrain contaminants;

    - On non-porous, chemically inert Cd- and Carbon Steel surfaces, as well as the porous CARC coupon, due to CEES's significant volatility, contaminant applied to the steel coupons was not detectable after undergoing a 24-hour coupon-contaminant incubation as implemented for positive controls ((+)-Control 2). As such, samples and controls could not be compared, and therefore Chemical Barrier Efficiencies for Cd- and Carbon Steels and CARC surfaces could not be determined.

RESULTS: Tables 1 and 2 contain the Data Test Set, and Decontamination Efficacy, Reduction in Starting Challenge Percent Efficacy, and Chemical Barrier Efficiency, respectively, for the sulfur mustard simulant contaminant CEES decontamination study conducted using DeconGel 1101 as the decontaminant on a number of coupon surfaces including Cadmium-plated and Carbon Steels, CARC, Silicone Rubber and Black Rubbers by solvent extraction.

Table 1. DeconGel decontaminant Data Test Set for CEES decontamination study on multiple surfaces as determined by residue analysis after solvent extraction

Coupon Type		**RA$_M$** (ng)	**CD** (g/m^2)
Cd-plated Steel	(-)-Control[1]	ND[2]	ND
	Remaining Agent	ND	ND
	(+)-Control 1[3]	ND	ND
	(+)-Control 2[4]	ND	ND
	Dose Confirmation	2127253 ± 8925	N/A[5]
Carbon Steel	(-)-Control	ND	ND
	Remaining Agent	ND	ND
	(+)-Control 1	ND	ND
	(+)-Control 2	ND	ND
	Dose Confirmation	2127253 ± 8925	N/A
CARC	(-)-Control	ND	ND
	Remaining Agent	1776.4 ± 0.0	0.0009 ± 0.0
	(+)-Control 1	2490.05 ± 14.06	0.0013 ± 0.0001
	(+)-Control 2	ND	ND
	Dose Confirmation	2127253 ± 8925	N/A
Silicone Rubber	(-)-Control	ND	ND
	Remaining Agent	415762.6 ± 1529.8	0.211 ± 0.001
	(+)-Control 1	915092.6 ± 4302.8	0.466 ± 0.002
	(+)-Control 2	55177.7 ± 216.3	0.028 ± 0.0
	Dose Confirmation	2127253 ± 8925	N/A
Black Rubber	(-)-Control	ND	ND
	Remaining Agent	63515.08 ± 1144.5	0.038 ± 0.001
	(+)-Control 1	930026.6 ± 23048.3	0.553 ± 0.014
	(+)-Control 2	7849.5 ± 86.58	0.005 ± 0.0003
	Dose Confirmation	2127253 ± 8925	N/A

[1] (-)-Control: no contaminant applied, then decontaminant applied
[2] ND: not detected, See Test Specific section, lower limits of detection (LOD) for CEES using GC/MS ≤ 80 ppb
[3] (+)-Control 1: contaminant applied for 1 h, no decontaminant applied
[4] (+)-Control 2: contaminant applied for 24 h, no decontaminant applied
[5] N/A: Not Applicable
RA$_M$: Remaining Agent (mass); CD: Contamination Density

Table 2. Decontamination Efficacy, Percent Efficacy, and Chemical Barrier Efficiency evaluation for the sulfur mustard simulant CEES decontamination study on multiple surfaces as determined by residue analysis after solvent extraction

Coupon Type	Decontamination Efficacy[1] (%)	Percent Efficacy[2] (%)	Chemical Barrier Efficacy[3] (%)
Cd-plated Steel	ND[4]	ND	ND
Carbon Steel	ND	ND	ND
CARC	28.6 ± 0.0	99.9 ± 0.0	ND
Silicone Rubber	54.6 ± 0.20	80.5 ± 0.11	84.7 ± 0.10
Black Rubber	93.2 ± 0.12	97.0 ± 0.05	85.8 ± 0.27

[1] See Decontamination Efficacy equation (Eqn 1) in Experimental section
[2] See Percent Efficacy equation (Eqn 2) in Experimental section
[3] See Chemical Barrier Efficacy equations (Eqn 3) in Experimental section
[4] ND: not determined (no contaminant detected for samples and/or controls)

METHOD DEVELOPMENT OPTIMIZATIONS/OBSERVATIONS:

- Due to CEES's low boiling point/volatility, significant loss of contaminant CEES was noted when storing contaminated coupons in a chemical hood for the required 60 min contact hold time. Utilizing an inverted Pyrex dessicator greatly improved both volatile surface retention and contaminant-coupon interaction, as evidenced after the 60 min hold time as either a significant surface retention of wetted drops, or as a raised surface.

- In applying a small amount (2.0 uL) of neat CEES contaminant as the starting challenge, there was no need to brush the volatile liquid contaminant throughout coupon surfaces to ensure a thin layer of CEES was loaded onto coupon surfaces; using a minute amount of contaminant facilitated a favorable interaction between DeconGel and CEES without overloading the relatively small amount of applied DeconGel decontaminant (1.0-1.1 g).

- Acetonitrile solvent was used to dissolve CEES contaminant and to generate a standard calibration curve possessing a significant concentration range and favorable accuracy and precision. Acetonitrile was used as the extraction solvent due to its ability to readily solvate methyl benzoate, compatibility with GC/MS analysis, relatively low volatility, and low toxicity profile. In accordance with the JPEO guidelines, 20 mL of acetonitrile extraction solvent was effective in generating precise data as evidenced by the low standard deviation of the modestly concentrated Dose Confirmation Samples ((DCS); DCS avg= 2127253 ± 8925 ng)

- Sulfur Mustard and its simulant CEES are nearly chemically equivalent ethyl sulfides, and as such behave similarly in both environmental settings and biological system, qualifying CEES as a suitable sulfur mustard chemical warfare simulant.

- GC/MS instrumentation is a sensitive and accurate analytical tool routinely used for the qualitative and quantitative determination of small- to medium-sized, low- to medium-polarity organic compounds. A nine-point calibration curve utilizing three independent stock solutions was generated, exhibiting a calibrated range for the standard calibration curve of 0.080-150 ppm (wt/wt) = 1200-2358000 ng CEES. The calibration curve exhibits excellent fit as demonstrated by the coefficient of determination of linear regression R^2, where $R^2 = 1.0$ (Figure 2). The limits of detection (LOD) and quantitation (LOQ) normal MS scan mode, the MS method used for the present decontamination study, are ≤ 40 ppb and ≤ 80 ppb, respectively, however in using either SIM (single-ion mode) or CI (chemical ionization, using methane) MS capabilities, the lowest limits of detection/quantitation of CEES approach ≤ 1 ppb (with SIM), and with CI could potentially approach ≤ 1 ppt (parts per trillion) or lower.

- As seen in Table 2, for Decontamination Efficacy and reduction in starting challenge Percent Efficacy determinations, on the non-porous Steel surfaces tested, because none of the highly volatile contaminant CEES was detected for either samples or Positive Control 1, an adequate measure of surface decontamination could not be calculated for Cd-plated and Carbon Steel coupons.

- On porous and/or chemically active coupon surfaces such as CARC and Black and Silicone Rubbers, modest to excellent Surface Decontamination of volatile CEES was realized; even on delicate or difficult-to-clean customized rubber, epoxy, or polyester surfaces, DeconGel is able to effectively remove hydrophobic contaminants through encapsulation/emulsification.

- To gauge DeconGel 1101's capabilities as an effective chemical (vapor) barrier, additional experimentation ((+)-Control 2) and data compilation provided a measure of DeconGel 1101's chemical barrier capacity - termed Chemical Barrier Efficiency (see Table 2). On Black and Silicone Rubber surfaces, calculated DeconGel 1101 Chemical Barrier Efficiencies indicate a significant chemical surface barrier capability when applied to porous, chemically active rubber/ epoxy/polyester surfaces that are able to absorb and entrain contaminants. Also, when applied to non-porous, chemically inert Cd- and Carbon Steel surfaces, as well as the porous CARC coupon. Due to CEES's significant volatility, contaminant applied to the steel coupons was not detectable after undergoing a 24-hour coupon-contaminant incubation as implemented for positive controls ((+)-Control 2). As such, samples and controls could not be compared, and therefore Chemical Barrier Efficiencies for Cd- and Carbon Steels and CARC surfaces could not be determined.

EXPERIMENTAL SECTION:

Reagents:

- Reagent grade CEES (Sigma-Aldrich) was utilized as the starting challenge (2.0 uL) contaminant in the decontamination study.

- Proprietarily formulated DeconGel 1101 was used as the decontaminant such that 1.0-1.1 g wet DeconGel was applied to contaminated surfaces, then allowed to dry for 24 h, and finally peeled off the surface. Immediately following gel peeling, the decontaminated coupon was extracted with solvent as elaborated below.

- Acetonitrile was used as the extraction solvent due to its ability to readily solvate CEES, compatibility with GC/MS analysis, relatively low volatility, and low toxicity profile. In addition, generated analytical calibration curves using acetonitrile exhibited favorable accuracy and precision.

Equipment (see Figure 1):

- A manual 2.0 uL glass syringe (National Scientific; Rockwood, TN) with a volume dispensing range of 0.0 to 2.0 uL, 0.01 uL grading, was used to dispense CEES contaminant, such that two non-touching 1.0 uL drops were dispensed on coupon surfaces per test.

- Calibrated manual pipets (Fisher Scientific; Fair Lawn, NJ) with disposable tips were used to generate the CEES calibration curve used to analytically determine amounts of CEES.

- A plastic squeezable bottle with coned dispenser was supplied with DeconGel 1101 and used to dispense 1.0-1.1 g wet DeconGel upon contaminated surfaces.

- Extraction solvent acetonitrile (20 mL) was dispensed into a 100 mL polypropylene bottle with screwcap prior to introduction of contaminated coupons using a 50 mL graduated cylinder.

- Samples and controls were prepared without further dilution/manipulation and immediately analyzed via GC/MS upon sample preparation.

- Due to the low boiling point of CEES, the 60 min contaminant contact time with coupon surfaces was conducted under an inverted Pyrex dessicator in attempts to minimize evaporative loss of CEES contaminant.

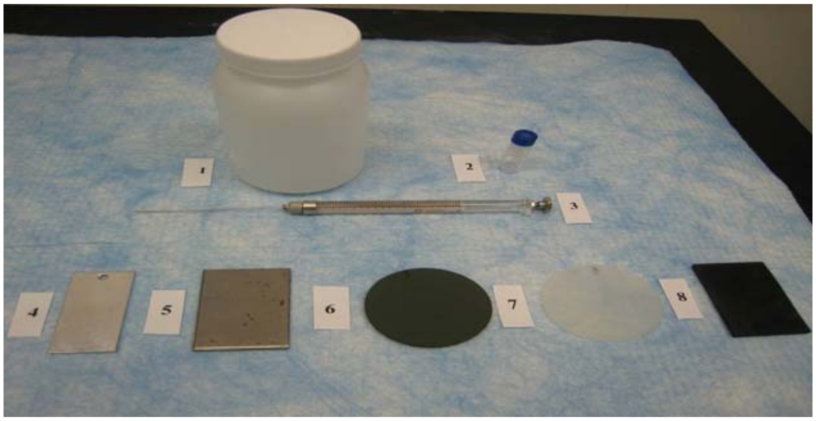
- Coupon area measurements in inches were performed using a standard ruler. Unit conversion and calculation was then conducted to achieve a coupon area in cm^2. Wet DeconGel was dispensed on coupons bordering the coupon edges and then the surface void filled in evenly as to maximize the spreading of DeconGel throughout the entire coupon surface area.

- A Thermo DSQ II capillary GC-double quadrupole MS with autosampler in positive mode was used to determine CEES concentration (ppm, wt/wt) of all samples and controls using a 9-point calibration curve utilizing three independent stock solutions.

Materials (see Figure 1):

- Coupons utilized in the decontamination study of CEES include Cadmium-plated Steel (12.75 cm^2 surface area), Carbon Steel (17.85 cm^2 surface area), Chemical Agent Resistant Coating (CARC) (19.63 cm^2 surface area), Black Rubber (16.83 cm^2 surface area), and Silicone Rubber (19.63 cm^2 surface area). All coupons were supplied by JPEO to Dr. Garry Edgington (CBI Polymers, Inc.; Honolulu, HI) and were conditioned on a laboratory countertop overnight.

Figure 1. Equipment and Materials utilized for the sulfur mustard simulant CEES decontamination study on multiple surfaces



<u>Legend</u>: **1.** 100 mL wide mouth extraction bottle with screw cap, **2.** GC vapor-tight sampling vial, **3.** 2 uL syringe for contaminant dispensing, **4.** Cd-plated Steel coupon, **5.** Carbon Steel coupon, **6.** CARC coupon, **7.** Silicone Rubber coupon, **8.** Black Rubber coupon.

Calculations:

- **Eqn 1.** Decontamination Efficacy

  = [(Contamination Density (CD) (g/m^2) of (+)-Control 1 – CD (g/m^2) of Remaining Agent) / CD (g/m^2) of (+)-Control 1] x 100%

- **Eqn 2.** Percent Efficacy

  = [(RAM (ng) of Dose Confirmation Mass – RAM (ng) of Remaining Agent Mass) / RAM (ng) of Dose Confirmation Mass] x 100%

- **Eqn 3.** Chemical Barrier Efficiency

  = [1 – (RAM (ng) of (+)-Control 2 / (RAM (ng) of Remaining Agent Mass – (RAM (ng) of (+)-Control 2))] x 100%

Detailed Test Summary:

- Experimental decontamination sampling and controls (positive and negative) were conducted on five different coupon types (as previously mentioned) in five replicates.

- Upon addition of 2.0 uL of CEES (experimental samples and positive controls) to coupon surfaces, contaminated coupons were placed under an inverted Pyrex dessicator for 60 min, and then either a) placed in extraction solvent via extraction bottle (for positive controls), or b) 1.0-1.1 g of wet DeconGel 1101 was poured evenly and thoroughly throughout the coupon surface area, DeconGel was allowed to dry for 24 h, the dried gel was peeled from the contaminated surface, and the contaminated coupon was immediately placed in an inert polypropylene bottle pre-filled with 20 mL extraction solvent as to completely submerse the coupon in extraction solvent. The extraction bottle was screw-capped, then swirled to facilitate contaminant extraction and let to stand for 60 min. Upon sample/control preparation, all experiments were immediately set-up for GC/MS analytical analysis in appropriate screw-cap GC vapor-tight vials (Fisher Scientific; Fair Lawn, NJ) (Figure 1).

- For negative controls, no contaminant was applied to coupon surfaces, and instead 1.0-1.1 g of wet DeconGel 1101 was poured evenly and thoroughly throughout the coupon surface area. DeconGel was allowed to dry for 24 h, the dried gel was peeled from coupon surfaces, and the coupon placed in an inert polypropylene bottle pre-filled with 20 mL extraction solvent as to completely submerse the coupon in extraction solvent. The extraction bottle was screw-capped, then swirled and let to stand for 60 min. Upon sample/control preparation, all experiments were immediately set-up for GC/MS analytical analysis.

- For positive controls, 2 uL of contaminant was applied to coupon surfaces and applied for either a) 1 h ((+)-Control 1), or b) 24 h ((+)-Control 2) coupon-contaminant incubation time, and then treated as described above for sample preparation. Positive Control 1 was implemented to provide an experimental control addressing the typical 60 min coupon-contaminant contact time; Positive Control 2 was implemented to provide an experimental control addressing the 24 h surface residence time employed for both contaminant and decontaminant, the timeframe needed to afford both adequate drying, and optimized surface decontamination by DeconGel 1101. After the appropriate incubation time, no decontaminant was applied, and contaminated coupons were immediately placed in an inert polypropylene bottle pre-filled with 20 mL extraction solvent. The extraction bottle was screw-capped, then swirled to facilitate contaminant extraction and let to stand for 60 min. Upon sample/control preparation, all experiments were immediately set-up for GC/MS analytical analysis.
- For dose confirmation samples, seven replicates were performed such that 2.0 uL CEES was charged via glass syringe into an inert polypropylene bottle pre-filled with 20 mL extraction solvent. The extraction bottle was screw-capped, then swirled to facilitate contaminant extraction and let to stand for 60 min. Upon sample preparation, all experiments were immediately set-up for GC/MS analytical analysis.

TEST SPECIFIC:

Precondition Coupons:

- Coupons were conditioned for 24 h on the laboratory countertop under normal ambient air-conditioned laboratory RT (25 ^{o}C) and humidity (40-60% humidity), where no extreme temperatures or humidity fluctuations were noted.

Contamination:

- CEES coupon contamination was performed (as previously mentioned) such that 2.0 uL total CEES neat liquid at RT (2x 1.0 uL non-touching drops) was manually added via glass syringe. Contaminated coupons were immediately placed under an inverted Pyrex dessicator and let to stand for 60 min.

- Contamination density (CD) of coupons were optimized to utilize the entire surface area of coupons by applying two non-touching 1.0 uL contaminant drops, and by applying DeconGel decontaminant on top and evenly over the whole coupon surface area. Coupons utilized in the decontamination study of CEES include Cadmium-plated Steel (12.75 cm^2 surface area), Carbon Steel (17.85 cm^2 surface area), Chemical Agent Resistant Coating (CARC) (19.63 cm^2 surface area), Black Rubber (16.83 cm^2 surface area), and Silicone Rubber (19.63 cm^2 surface area).

Dose Confirmation Sample Preparation:

- For dose confirmation samples, seven replicates were performed such that 2.0 uL CEES was charged via glass syringe into an inert polypropylene bottle pre-filled with 20 mL acetonitrile extraction solvent. The extraction bottle was screw-capped, then swirled to facilitate contaminant extraction and let to stand for 60 min. Upon sample preparation, all experiments were immediately set-up for GC/MS analytical analysis.

Post-Contamination Surface Contamination Observation:

- On non-porous inert coupon surfaces such as Cadmium-plated Steel and Carbon Steel, We observed that CEES contaminant drops initially beaded on the surface. However, after about 20 min, drops applied to Carbon Steel slowly spread out to cover roughly 2-3x the initial surface area, while contaminant drops applied to Cadmium-plated Steel stayed beaded throughout the 60 min contaminant-coupon contact hold time. After the 60 min hold time, CEES contaminant drops were evidenced on non-porous steel surfaces as wetted drops (Cd-plated Steel) or as wetted smears (Carbon Steel); after the 24 h hold time employed for (+)-Control 2, no CEES contaminant drops were evidenced on non-porous steel surfaces as either drops or smears.

- On porous and/or chemically active coupon surfaces such as CARC and Silicone Rubber, CEES contaminant drops were observed to rapidly spread out from the initial drop surface area over 10 min, becoming absorbed into coupon surfaces as evidenced by a wetted surface roughly 2-3x the initial surface area that persisted for the 60 min hold time; after the 24 h hold time employed for (+)-Control 2, no CEES contaminant drops were evidenced on these surfaces. When CEES drops were applied on top of Black Rubber, the contaminant did not roll or spread out, but instead absorbed within minutes into the coupon surface as evidenced by a dry surface bulging that persisted throughout the 24 h hold time employed for (+)-Control 2.

Aging:

- Coupons were conditioned for 24 h on the laboratory countertop under normal ambient air-conditioned laboratory room temperature (25 ºC) and humidity (40-60% humidity), where no extreme temperature or humidity fluctuations were noted.

Post-Aging Surface Contamination Observation:

- No significant difference in appearance, size, or color of conditioned/aged coupons were noted, indicating that no significant or unexpected swelling, shrinking, deformation, decomposition, or surface drying or wetting had occurred.

Pre-Rinse:

- No pre-rinsing step of coupons was performed. Coupons were delivered to CBI Polymers directly from JPEO in a residue-free, clean state and used as received.
- Coupons were conditioned for 24 h on the laboratory countertop under normal ambient air-conditioned laboratory RT (25 ºC) and humidity (40-60% humidity), where no extreme temperatures or humidity fluctuations were noted.

Decontamination:

- To decontaminate the contaminated coupons, after the 60 min contaminant-coupon surface hold time under an inverted Pyrex dessicator, 1.0-1.1 g of wet DeconGel 1101 at room temperature was poured evenly on top of the contaminated coupon throughout the whole surface area in an attempt to decontaminate all contaminant that might have spread throughout the coupon surface area.
- DeconGel was allowed to dry for 24 h, the dried gel was peeled from the contaminated surface, and the contaminated coupon was immediately placed in an inert polypropylene bottle pre-filled with 20 mL extraction solvent as to completely submerse the coupon in extraction solvent. The extraction bottle was screw-capped, then swirled to facilitate contaminant extraction and let to stand for 60 min. Upon sample preparation, all experiments were immediately set-up for GC/MS analytical analysis.

Post-Rinse:

- No post-rinsing step of DeconGel decontaminant was performed due to the chemical nature and intended utility of DeconGel; DeconGel is a viscous gel that upon surface contact first spreads out into a thin layer and then must air-dry for 12-24 h before it is peeled off of the contaminated surface. Rinsing either wet or dry DeconGel will result in partial to complete dissolution/emulsification of the decontaminant and most likely reduce its decontamination efficacy.

Drying:

- Upon addition of decontaminant to contaminated surfaces, DeconGel was allowed to dry for 24 h, the dried gel was peeled from the contaminated surface, and the contaminated coupon was immediately placed in an inert polypropylene bottle pre-filled with 20 mL extraction solvent as to completely submerse the coupon in the extraction solvent.

- Subsequent coupon post-washing or drying steps were not conducted or deemed necessary for this decontamination study.

Remaining Agent Measurement:

- Upon addition of 2.0 uL of CEES (experimental samples and positive controls) to coupon surfaces, contaminated coupons were placed under an inverted Pyrex dessicator for 60 min, and then either placed in extraction solvent as described above (for positive control) or 1.0-1.1 g of wet DeconGel 1101 was poured evenly throughout the whole coupon surface area. DeconGel was allowed to dry for 24 h, the dried gel was peeled from the contaminated surface, and the contaminated coupon was immediately placed in an inert polypropylene bottle pre-filled with 20 mL extraction solvent to completely submerse the coupon in extraction solvent. The extraction bottle was screw-capped, then swirled to facilitate contaminant extraction and let to stand for 60 min. Upon sample/control preparation, all experiments were immediately set-up for GC/MS analytical analysis in appropriate screw-cap GC vapor-tight vials (Fisher Scientific; Fair Lawn, NJ).

Chromatographic Analysis:

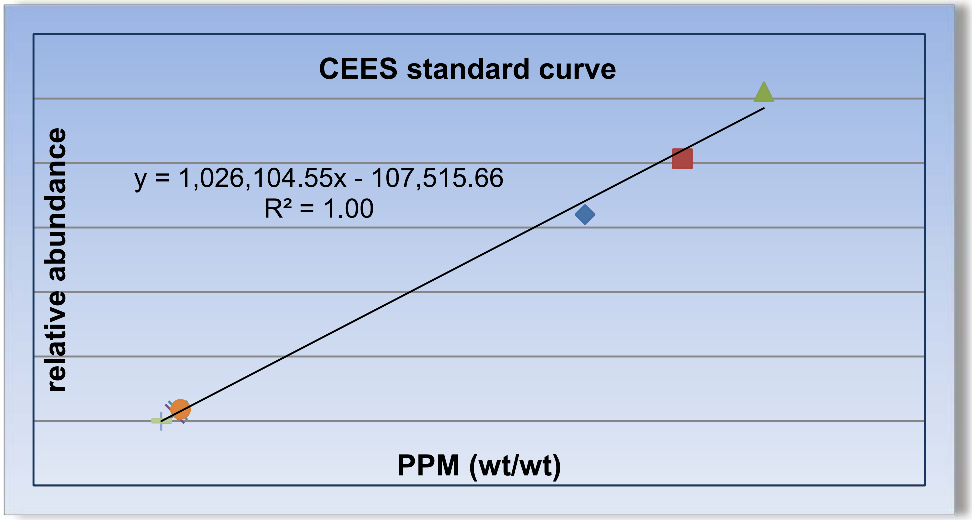
- A Thermo DSQ II capillary GC-double quadrupole MS with autosampler in positive mode was used to determine CEES concentration (ppm, wt/wt) of all samples and controls using a 9-point calibration curve utilizing three independent stock solutions.

- A Restek (Bellefonte, PA) Rtx-5 polysiloxane capillary column was employed using Temperature Program: Initial at 45 ºC, hold 1 min, Ramp at 30 ºC/min to 300 ºC, hold for 5 min.

- The calibrated range for the standard calibration curve is 0.080-150 ppm (wt/wt) = 1200-2358000 ng CEES.

- The limits of detection (LOD) and quantitation (LOQ) normal MS scan mode, the MS method used for the present decontamination study, are ≤ 40 ppb and ≤ 80 ppb, respectively, however in using either SIM (single-ion mode) or CI (chemical ionization using methane) MS capabilities the lowest limits of detection/quantitation of CEES approach ≤ 1 ppb (with SIM), and with CI could potentially approach ≤ 1 ppt (parts per trillion) or lower.

Figure 2. CEES Standard Calibration Curve



Reporting Statements/Summary:

- Tables 1 and 2 contain the Data Test Set, and Decontamination Efficacy, Reduction in Starting Challenge Percent Efficacy, and Chemical Barrier Efficacy, respectively, for the sulfur mustard simulant contaminant CEES decontamination study conducted using DeconGel 1101 as the decontaminant on a number of coupon surfaces including Cadmium-plated and Carbon Steels, CARC, and Silicone and Black Rubbers by solvent extraction.

- As seen in Table 2, for Decontamination Efficacy and reduction in starting challenge Percent Efficacy determinations it is noted: 1) On the non-porous Steel surfaces tested, because none of the highly volatile contaminant CEES was detected for either samples or Positive Control 1, an adequate measure of surface decontamination could not be calculated for Cd-plated and Carbon Steel coupons; and 2) On porous and/or chemically active coupon surfaces DeconGel 1101 decontamination efficacy and reduction in starting challenge Percent Efficacy ranged from 28.6 and 99.9% (on CARC) to 54.6 and 80.5% (on Silicone Rubber) to 93.2 and 97.0% (on Black Rubber) as determined by residue analysis after solvent extraction. Overall, the decontamination study using decontaminant DeconGel 1101 on CEES contaminant reveals DeconGel's ability to both 1) encapsulate/emulsify hydrophobic contaminants such as CEES and other chlorinated ethyl sulfide/sulfur mustards, and 2) act as a chemical barrier against volatile agents such as CEES or other more toxic volatiles/substances that might pose a significant contact risk or possess prolonged environmental persistence.

- For porous and/or chemically active coupon surfaces such as CARC and Rubbers, modest to excellent surface decontamination of volatile CEES was realized; interestingly, CEES was noted to appreciably absorb into such coupon surfaces, which seemed to sequester/dissolve CEES within the coupon surfaces, hindering sufficient decontaminant-contaminant interaction and limiting DeconGel 1101's abilities to encapsulate/emulsify contaminant CEES as reflected by low Decontamination and Percent Efficacies (Table 2). On the other hand, CEES' limiting volatility introduced difficulties in accurately determining Surface Decontamination for all coupon types tested; rapid surface evaporation of CEES when applied to coupon surfaces could result in falsely inflated Decontamination Efficacies and Percent Efficacies.
- To gauge DeconGel 1101's capabilities as an effective chemical (vapor) barrier, additional experimentation ((+)-Control 2) and data compilation provided a measure of DeconGel 1101's chemical barrier capacity - termed Chemical Barrier Efficiency (see Table 2). On Black and Silicone Rubber surfaces, calculated DeconGel 1101 Chemical Barrier Efficiencies indicate a significant chemical surface barrier capability when applied to porous, chemically active rubber/ epoxy/polyester surfaces able to absorb and entrain contaminants; and 2) On non-porous, chemically inert Cd- and Carbon Steel surfaces, as well as the porous CARC coupon, due to CEES's significant volatility, contaminant applied to the steel coupons was not detectable after undergoing a 24-hour coupon-contaminant incubation as implemented for positive controls ((+)-Control 2). As such, samples and controls could not be compared, and therefore Chemical Barrier Efficiencies for Cd- and Carbon Steels and CARC surfaces could not be determined.

- The 60 min contact time between CEES contaminant and all coupons tested was deemed necessary, however adoption of a shorter contact time holds promise to allow the required interaction between contaminant and coupon surface while minimizing loss of volatile contaminant CEES to evaporation. Utilizing an inverted Pyrex dessicator to store contaminated coupons immediately after contamination and during the required 60 min contact time aided in retaining significant amounts of contaminant on/within the coupon surfaces.

- Since live agents may not exhibit the same bioactivity, volatility, or absorption characteristics as CEES, live agent testing is recommended to accurately measure DeconGel effectiveness on test surfaces.

7.6. Technical Report for CWA Sulfur Mustard-Simulant (CEPS)

TITLE: Surface Decontamination of the Sulfur Mustard Simulant 2-Chloroethyl Phenyl Sulfide (CEPS) by DeconGel 1101

ANALYST: Geoff Nadolski PhD, Senior Polymer Scientist, CBI Polymers, Inc.

SUPERVISOR: Garry J. Edgington PhD, Chief Scientist, CBI Polymers, Inc.

OBJECTIVE: Evaluate the decontamination efficacy of DeconGel 1101 on coupon types:

a. Cadmium-plated Steel,

b. Carbon Steel,

c. Chemical Agent Resistant Coating (CARC),

d. Silicone Rubber, and

e. Black Rubber.

- Experimentation and Reporting conducted following JPEO-CBD Source Document:
  - 2007 Chemical Decontamination Performance Evaluation Testing,
  - Section 6: Panel Contact Test to Determine Contact Hazard,
  - Section 6-E: Panel (Coupon) Extraction Method to Determine Remaining Agent;
- Sensitive (low ppb-level) GC/MS (gas chromatography/mass spectrometry) analytical methods developed.

HAZARDOUS MATERIALS RELEVANCE: Sulfur Mustard, bis-(2-chloroethyl) sulfide, is a powerful vesicant, cytotoxic agent regulated under the 1993 Chemical Weapons Convention (CWC). Sulfur mustard can be deployed by spraying, or more typically distributed via artillery shells and bombs. For experimental testing purposes, the half-mustard 2-chloroethyl phenyl sulfide (CEPS) is commonly used as an effective yet less toxic simulant for sulfur mustard chemical warfare agents.

SUMMARY RESULTS:

- On the non-porous steel surfaces tested, excellent surface decontamination was achieved by applying DeconGel 1101 onto contaminated surfaces facilitating encapsulation/emulsification of contaminants by DeconGel's active components.
    - DeconGel 1101 reduction in starting challenge Percent Efficacies, a measure comparing samples to the Dose Confirmation Control, ranged from 96.3% (on Cd-plated steel) to 97.2% (on Carbon steel) as determined by solvent extraction;
    - DeconGel 1101 Decontamination Efficacies, a measure comparing samples to the Positive Control (adjusted accordingly regarding particular coupon surface area), ranged from 96.3% (on Cd-plated steel) to 97.2% (on carbon steel) as determined by solvent extraction.
- On porous and/or chemically active coupon surfaces such as CARC and Black and Silicone Rubbers, modest Surface Decontamination of CEPS was realized; even on delicate or difficult-to-clean customized rubber, epoxy, or polyester surfaces, DeconGel is able to effectively remove hydrophobic contaminants through encapsulation/emulsification.
    - Reduction in starting challenge Percent Efficacies of DeconGel 1101 ranged from 51.2% (on CARC) to 51.8% (on Silicone Rubber) to 76.9% (on Black Rubber) as determined by solvent extraction.
    - DeconGel 1101 Decontamination Efficacies ranged from 51.2% (on CARC) to 51.9% (on Silicone Rubber) to 76.9% (on Black Rubber) as determined by solvent extraction.
- Due to its viscous, slow-drying, and emulisifying/encapsulating properties, DeconGel 1101 can serve as a partial chemical (vapor) barrier. This chemical barrier effect can serve to reduce the release of contaminant to the surrounding environment, as well as promote a prolonged residence-time inside the gel layer. An extended residence-time in the gel is predicted to enhance the efficacy of neutralizing agents that are proposed to be employed in future versions of the gel. This reduction in rate of release of agents is proposed to be especially valuable in enhancing neutralization efficacies of CWAs with high reactivity such as sulfur mustards and phosphono-esters.

RESULTS: Tables 1 and 2 contain the Data Test Set, and Decontamination Efficacy and Reduction in Starting Challenge Percent Efficacy, respectively, for the sulfur mustard simulant contaminant CEPS decontamination study conducted using DeconGel 1101 as the decontaminant on a number of coupon surfaces including Cadmium-plated and Carbon Steels, CARC, Silicone Rubber and Black Rubbers by solvent extraction.

Table 1. DeconGel decontaminant Data Test Set for CEPS decontamination study on multiple surfaces as determined by residue analysis after solvent extraction

Coupon Type		RA_M (ng)	CD (g/m^2)
Cd-plated Steel	(-)-Control[1]	ND[2]	ND
	Remaining Agent	85048.3 ± 826.0	0.067 ± 0.0007
	(+)-Control 1[3]	2338224 ± 7223.0	1.83 ± 0.0057
	Dose Confirmation	2334869 ± 8674	N/A[4]
Carbon Steel	(-)-Control	ND	ND
	Remaining Agent	64382.8 ± 751.3	0.036 ± 0.0003
	(+)-Control 1	2335394 ± 9478.8	1.31 ± 0.0055
	Dose Confirmation	2334869 ± 8674	N/A
CARC	(-)-Control	ND	ND
	Remaining Agent	1138814 ± 5408.6	0.580 ± 0.0028
	(+)-Control 1	2335112 ± 7818.6	1.19 ± 0.0041
	Dose Confirmation	2334869 ± 8674	N/A
Silicone Rubber	(-)-Control	ND	ND
	Remaining Agent	1125300 ± 3701.8	0.573 ± 0.0019
	(+)-Control 1	2337627 ± 8288.8	1.19 ± 0.0042
	Dose Confirmation	2334869 ± 8674	N/A
Black Rubber	(-)-Control	ND	ND
	Remaining Agent	539647.6 ± 1167.7	0.321 ± 0.0007
	(+)-Control 1	2337564 ± 8147.2	1.39 ± 0.0048
	Dose Confirmation	2334869 ± 8674	N/A

[1] (-)-Control: no contaminant applied, and then decontaminant applied
[2] ND: not detected, See Test Specific section, lower limits of detection (LOD) for CEPS using GC/MS ≤ 50 ppb
[3] (+)-Control 1: contaminant applied for 1 h, no decontaminant applied
[4] N/A: Not Applicable
RA_M: Remaining Agent (mass); CD: Contamination Density

Table 2. Decontamination Efficacy and Percent Efficacy evaluation for the sulfur mustard simulant CEPS decontamination study on multiple surfaces as determined by residue analysis after solvent extraction

Coupon Type	Decontamination Efficacy[1] (%)	Percent Efficacy[2] (%)
Cd-plated Steel	96.3 ± 0.1	96.3 ± 0.1
Carbon Steel	97.2 ± 0.1	97.2 ± 0.1
CARC	51.2 ± 0.51	51.2 ± 0.47
Silicone Rubber	51.9 ± 0.32	51.8 ± 0.25
Black Rubber	76.9 ± 0.11	76.9 ± 0.1

[1] See Decontamination Efficacy equation (Eqn 1) in Experimental section
[2] See Percent Efficacy equation (Eqn 2) in Experimental section

METHOD DEVELOPMENT OPTIMIZATIONS/OBSERVATIONS:

- In applying a small amount (2.0 uL) of neat CEPS contaminant as the starting challenge, there was no need to brush the contaminant throughout coupon surfaces to ensure a thin layer of CEPS was loaded onto coupon surfaces; using a minute amount of contaminant facilitated a favorable interaction between DeconGel and CEPS without overloading the relatively small amount of applied DeconGel decontaminant (1.0-1.1 g).

- Acetonitrile solvent was used to dissolve CEPS contaminant and to generate a standard calibration curve possessing a significant concentration range and favorable accuracy and precision. Acetonitrile was used as the extraction solvent due to its ability to readily solvate CEPS, compatibility with GC/MS analysis, relatively low volatility, and low toxicity profile. In accordance with the JPEO guidelines, 20 mL of acetonitrile extraction solvent was effective in generating precise data as evidenced by the low standard deviation of the modestly concentrated Dose Confirmation Samples ((DCS); DCS avg= 2224869 ± 8674 ng)

- Sulfur Mustard and its simulant CEPS are nearly chemically equivalent ethyl sulfides, and as such behave similarly in both environmental settings and biological system, qualifying CEPS as a suitable sulfur mustard chemical warfare simulant.

- GC/MS instrumentation is a sensitive and accurate analytical tool routinely used for the qualitative and quantitative determination of small- to medium-sized, low- to medium-polarity organic compounds. A nine-point calibration curve utilizing three independent stock solutions was generated, exhibiting a calibrated range for the standard calibration curve of 0.050-200 ppm (wt/wt) = 786-3144000 ng CEPS. The calibration curve exhibits excellent fit as demonstrated by the coefficient of determination of linear regression R^2, where $R^2 = 1.0$ (Figure 2). The limits of detection (LOD) and quantitation (LOQ) normal MS scan mode, the MS method used for the present decontamination study, are ≤ 40 ppb and ≤ 51 ppb, respectively, however in using either SIM (single-ion mode) or CI (chemical ionization, using methane) MS capabilities, the lowest limits of detection/quantitation of CEPS approach ≤ 1 ppb (with SIM), and with CI could potentially approach ≤ 1 ppt (parts per trillion) or lower.

- As seen in Table 2, for Decontamination Efficacy and reduction in starting challenge Percent Efficacy determinations, on non-porous inert coupon Steel surfaces excellent surface decontamination was achieved by applying DeconGel 1101 onto contaminated surfaces facilitating encapsulation/emulsification of contaminants by DeconGel's active components.

- On porous and/or chemically active coupon surfaces such as CARC and Rubbers, modest surface decontamination of CEPS is noted; specifically, when reviewing the Percent Efficacy achieved in comparing remaining contaminant agent versus contaminant confirmation dosage, modest (for CARC and Silicone Rubber) to good (for Black Rubber) surface decontamination of CEPS was achieved. DeconGel is able to effectively encapsulate/emulsify hydrophobic contaminants such as CEPS or other chlorinated organo-sulfur/ethyl sulfides from such porous, delicate, or difficult-to-clean customized rubber, epoxy, or polyester surfaces.

EXPERIMENTAL SECTION:

Reagents:

- Reagent grade CEPS (Sigma-Aldrich, USA) was utilized as the starting challenge (2.0 uL) contaminant in the decontamination study.
- Proprietarily formulated DeconGel 1101 was used as the decontaminant such that 1.0-1.1 g wet DeconGel was applied to contaminated surfaces, then allowed to dry for 24 h, and finally peeled off the surface. Immediately following gel peeling, the decontaminated coupon was extracted with solvent as elaborated below.
- Acetonitrile was used as the extraction solvent due to its ability to readily solvate CEPS, compatibility with GC/MS analysis, relatively low volatility, and low toxicity profile. In addition, generated analytical calibration curves using acetonitrile exhibited favorable accuracy and precision.

Equipment (see Figure 1):

- A manual 2.0 uL glass syringe (National Scientific; Rockwood, TN) with a volume dispensing range of 0.0 to 2.0 uL, 0.01 uL grading, was used to dispense CEPS contaminant, such that two non-touching 1.0 uL drops were dispensed on coupon surfaces per test.
- Calibrated manual pipets (Fisher Scientific; Fair Lawn, NJ) with disposable tips were used to generate the CEPS calibration curve used to analytically determine amounts of CEPS.
- A plastic squeezable bottle with coned dispenser was supplied with DeconGel 1101 and used to dispense 1.0-1.1 g wet DeconGel upon contaminated surfaces.
- Extraction solvent acetonitrile (20 mL) was dispensed into a 100 mL polypropylene bottle with screwcap prior to introduction of contaminated coupons using a 50 mL graduated cylinder.
- Samples and controls were prepared without further dilution/manipulation and immediately analyzed via GC/MS upon sample preparation.
- A coupon area measurement in inches was performed using a standard ruler. Unit conversion and calculation was then conducted to achieve a coupon area in cm^2. Wet DeconGel was dispensed on coupons

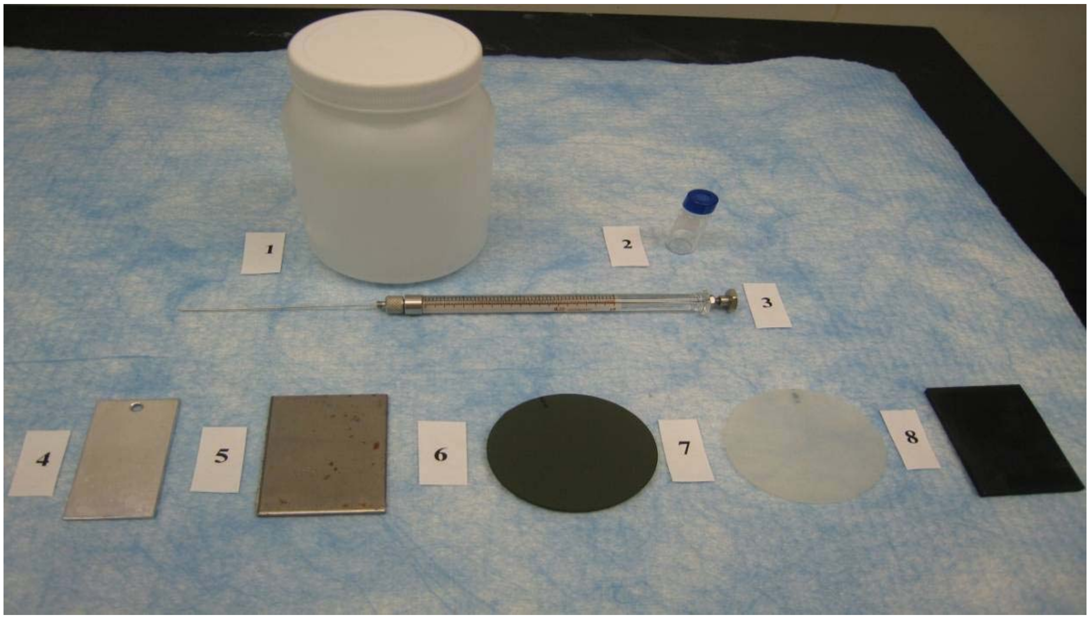
bordering the coupon edges and then the surface void filled in evenly as to maximize the spreading of DeconGel throughout the entire coupon surface area.

- A Thermo DSQ II capillary GC-double quadrupole MS with autosampler in positive mode was used to determine CEPS concentration (ppm, wt/wt) of all samples and controls using a 9-point calibration curve utilizing three independent stock solutions.

Materials (see Figure 1):

- Coupons utilized in the decontamination study of CEPS include Cadmium-plated Steel (12.75 cm^2 surface area), Carbon Steel (17.85 cm^2 surface area), Chemical Agent Resistant Coating (CARC) (19.63 cm^2 surface area), Black Rubber (16.83 cm^2 surface area), and Silicone Rubber (19.63 cm^2 surface area). All coupons were supplied by JPEO to Dr. Garry Edgington (CBI Polymers, LLC; Honolulu, HI) and were conditioned at RT on a laboratory countertop overnight.

Figure 1. Equipment and Materials utilized for the sulfur mustard simulant CEPS decontamination study on multiple surfaces



Legend: **1.** 100 mL wide mouth extraction bottle with screw cap, **2.** GC vapor-tight sampling vial, **3.** 2 uL syringe for contaminant dispensing, **4.** Cd-plated Steel coupon, **5.** Carbon Steel coupon, **6.** CARC coupon, **7.** Silicone Rubber coupon, **8.** Black Rubber coupon.

Calculations:

- **Eqn 1.** Decontamination Efficacy

= [(Contamination Density (CD) (g/m^2) of (+)-Control 1 – CD (g/m^2) of Remaining Agent) / CD (g/m^2) of (+)-Control 1] x 100%

- **Eqn 2.** Percent Efficacy
  = [(RA_M (ng) of Dose Confirmation Mass – RA_M (ng) of Remaining Agent Mass) / RA_M (ng) of Dose Confirmation Mass] x 100%

Detailed Test Summary:

- Experimental decontamination sampling and controls (positive and negative) were conducted on five different coupon types (as previously mentioned) in five replicates.

- Upon addition of 2.0 uL of CEPS (experimental samples and positive control) to coupon surfaces, contaminated coupons were placed under an inverted Pyrex dessicator for 60 min, and then either a) placed in extraction solvent via extraction bottle (for positive controls), or b) 1.0-1.1 g of wet DeconGel 1101 was poured evenly and thoroughly throughout the coupon surface area, DeconGel was allowed to dry for 24 h, the dried gel was peeled from the contaminated surface, and the contaminated coupon was immediately placed in an inert polypropylene bottle pre-filled with 20 mL extraction solvent as to completely submerse the coupon in extraction solvent. The extraction bottle was screw-capped, then swirled to facilitate contaminant extraction and let to stand for 60 min. Upon sample/control preparation, all experiments were immediately set-up for GC/MS analytical analysis in appropriate screw-cap GC vapor-tight vials (Fisher Scientific; Fair Lawn, NJ) (Figure 1).

- For negative controls, no contaminant was applied to coupon surfaces, and instead 1.0-1.1 g of wet DeconGel 1101 was poured evenly and thoroughly throughout the coupon surface area. DeconGel was allowed to dry for 24 h, the dried gel was peeled from coupon surfaces, and the coupon placed in an inert polypropylene bottle pre-filled with 20 mL extraction solvent as to completely submerse the coupon in extraction solvent. The extraction bottle was screw-capped, then swirled and let to stand for 60 min. Upon sample/control preparation, all experiments were immediately set-up for GC/MS analytical analysis.

- For positive controls, 2 uL of contaminant was applied to coupon surfaces and applied for 1 h ((+)-Control 1) coupon-contaminant incubation time, and then treated as described above for sample preparation. Positive Control was implemented to provide an experimental control addressing the typical 60 min coupon-contaminant contact time. After the appropriate incubation time, no decontaminant was applied, and contaminated coupons were immediately placed in an inert polypropylene bottle pre-filled with 20 mL extraction solvent. The extraction bottle was screw-capped, then swirled to facilitate contaminant extraction and let to stand for 60 min. Upon sample/control preparation, all experiments were immediately set-up for GC/MS analytical analysis.

- For dose confirmation samples, seven replicates were performed such that 2.0 uL CEPS was charged via glass syringe into an inert polypropylene bottle pre-filled with 20 mL extraction solvent. The extraction bottle was screw-capped, then swirled to facilitate contaminant extraction and let to stand for 60 min. Upon sample preparation, all experiments were immediately set-up for GC/MS analytical analysis.

TEST SPECIFIC:

Precondition Coupons:

- Coupons were conditioned for 24 h on the laboratory countertop under normal ambient air-conditioned laboratory RT (25 °C) and humidity (40-60% humidity), where no extreme temperatures or humidity fluctuations were noted.

Contamination:

- CEPS coupon contamination was performed (as previously mentioned) such that 2.0 uL total CEPS neat liquid at RT (2x 1.0 uL non-touching drops) was manually added via glass syringe. Contaminated coupons were immediately placed under an inverted Pyrex dessicator and let to stand for 60 min.
- Contamination density (CD) of coupons were optimized to utilize the entire surface area of coupons by applying two non-touching 1.0 uL contaminant drops, and by applying DeconGel decontaminant on top and evenly over the whole coupon surface area. Coupons utilized in the decontamination study of CEPS include Cadmium-plated Steel (12.75 cm^2 surface area), Carbon Steel (17.85 cm^2 surface area), Chemical Agent Resistant Coating (CARC) (19.63 cm^2 surface area), Black Rubber (16.83 cm^2 surface area), and Silicone Rubber (19.63 cm^2 surface area).

Dose Confirmation Sample Preparation:

- For dose confirmation samples, seven replicates were performed such that 2.0 uL CEPS was charged via glass syringe into an inert polypropylene bottle pre-filled with 20 mL acetonitrile extraction solvent. The extraction bottle was screw-capped, then swirled to facilitate contaminant extraction and let to stand for 60 min. Upon sample preparation, all experiments were immediately set-up for GC/MS analytical analysis.

Post-Contamination Surface Contamination Observation:

- On non-porous inert coupon surfaces such as Cadmium-plated Steel and Carbon Steel, we observed that CEPS contaminant drops initially beaded on the surface. However, after about 20 min, drops applied to Carbon Steel slowly spread out to cover roughly 2-3x the initial surface area, while contaminant drops applied to Cadmium-plated Steel stayed beaded throughout the 60 min contaminant-coupon contact hold time. After the 60 min hold time, CEPS contaminant drops were evidenced on non-porous steel surfaces as wetted drops (Cd-plated Steel) or as wetted smears (Carbon Steel).

- On porous and/or chemically active coupon surfaces such as CARC and Silicone Rubber, CEPS contaminant drops were observed to rapidly spread out from the initial drop surface area over 10 min, becoming absorbed into coupon surfaces as evidenced by a wetted surface roughly 2-3x the initial surface area that persisted for the 60 min hold time. When CEPS drops were applied on top of Black Rubber, the contaminant did not roll or spread out, but instead absorbed within minutes into the coupon surface as evidenced by a dry surface bulging that persisted throughout the 24 h hold time employed for (+)-Control 2.

Aging:

- Coupons were conditioned for 24 h on the laboratory countertop under normal ambient air-conditioned laboratory room temperature (25 ºC) and humidity (40-60% humidity), where no extreme temperature or humidity fluctuations were noted.

Post-Aging Surface Contamination Observation:

- No significant difference in appearance, size, or color of conditioned/aged coupons were noted, indicating that no significant or unexpected swelling, shrinking, deformation, decomposition, or surface drying or wetting had occurred.

Pre-Rinse:

- No pre-rinsing step of coupons was performed. Coupons were delivered to CBI Polymers directly from JPEO in a residue-free, clean state and used as received.

- Coupons were conditioned for 24 h on the laboratory countertop under normal ambient air-conditioned laboratory RT (25 ^{o}C) and humidity (40-60% humidity), where no extreme temperatures or humidity fluctuations were noted.

Decontamination:

- To decontaminate the contaminated coupons, after the 60 min contaminant-coupon surface hold time under an inverted Pyrex dessicator, 1.0-1.1 g of wet DeconGel 1101 at room temperature was poured evenly on top of the contaminated coupon throughout the whole surface area in an attempt to decontaminate all contaminant that might have spread throughout the coupon surface area.

- DeconGel was allowed to dry for 24 h, the dried gel was peeled from the contaminated surface, and the contaminated coupon was immediately placed in an inert polypropylene bottle pre-filled with 20 mL extraction solvent as to completely submerse the coupon in extraction solvent. The extraction bottle was screw-capped, then swirled to facilitate contaminant extraction and let to stand for 60 min. Upon sample preparation, all experiments were immediately set-up for GC/MS analytical analysis.

Post-Rinse:

- No post-rinsing step of DeconGel decontaminant was performed due to the chemical nature and intended utility of DeconGel; DeconGel is a viscous gel that upon surface contact first spreads out into a thin layer and then must air-dry for 12-24 h before it is peeled off of the contaminated surface. Rinsing either wet or dry DeconGel will result in partial to complete dissolution/emulsification of the decontaminant and most likely reduce its decontamination efficacy.

Drying:

- Upon addition of decontaminant to contaminated surfaces, DeconGel was allowed to dry for 24 h, the dried gel was peeled from the contaminated surface, and the contaminated coupon was immediately placed in an inert polypropylene bottle pre-filled with 20 mL extraction solvent as to completely submerse the coupon in the extraction solvent.

- Subsequent coupon post-washing or drying steps were not conducted or deemed necessary for this decontamination study.

Remaining Agent Measurement:

- Upon addition of 2.0 uL of CEPS (experimental samples and positive control) to coupon surfaces, contaminated coupons were placed under an inverted Pyrex dessicator for 60 min, and then either placed in extraction solvent as described above (for positive control) or 1.0-1.1 g of wet DeconGel 1101 was poured evenly throughout the whole coupon surface area. DeconGel was allowed to dry for 24 h, the dried gel was peeled from the contaminated surface, and the contaminated coupon was immediately placed in an inert polypropylene bottle pre-filled with 20 mL extraction solvent to completely submerse the coupon in extraction solvent. The extraction bottle was screw-capped, then swirled to facilitate contaminant extraction and let to stand for 60 min. Upon sample/control preparation, all experiments were immediately set-up for GC/MS analytical analysis in appropriate screw-cap GC vapor-tight vials (Fisher Scientific; Fair Lawn, NJ).

Chromatographic Analysis:

- A Thermo DSQ II capillary GC-double quadrupole MS with autosampler in positive mode was used to determine CEPS concentration (ppm, wt/wt) of all samples and controls using a 9-point calibration curve utilizing three independent stock solutions.

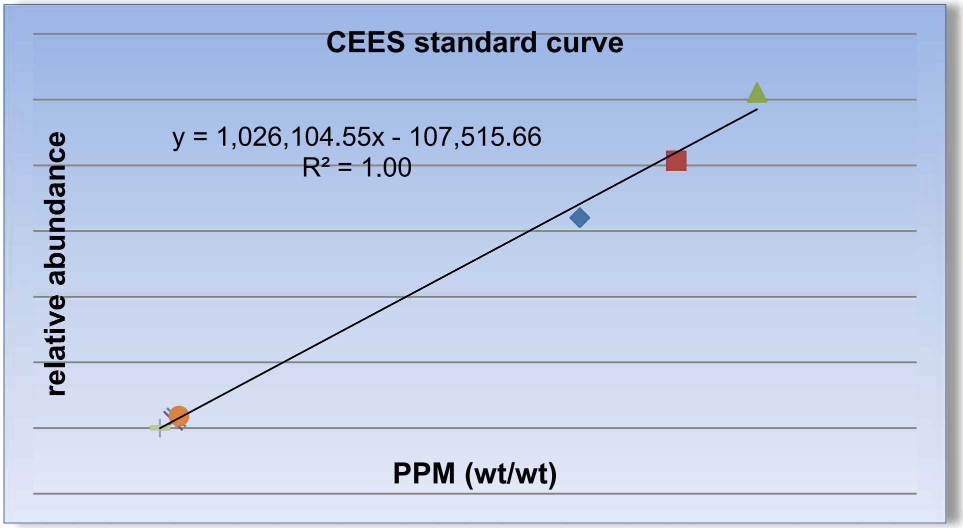
- A Restek (Bellefonte, PA) Rtx-5 polysiloxane capillary column was employed using Temperature Program: Initial at 120 ^{o}C, hold 1 min, Ramp at 30 ^{o}C/min to 320 ^{o}C, hold for 5 min.

- The calibrated range for the standard calibration curve is 0.050-200 ppm (wt/wt) = 786-3144000 ng CEPS.

- The limits of detection (LOD) and quantitation (LOQ) normal MS scan mode, the MS method used for the present decontamination study, are ≤ 40 ppb and ≤ 51 ppb, respectively, however in using either SIM (single-ion mode) or CI (chemical ionization using methane) MS capabilities the lowest limits of detection/quantitation of CEPS approach ≤ 1 ppb (with

SIM), and with CI could potentially approach ≤ 1 ppt (parts per trillion) or lower.

Figure 2. CEES Standard Calibration Curve



Reporting Statements/Summary:

- Tables 1 and 2 contain the Data Test Set, and Decontamination Efficacy and Reduction in Starting Challenge Percent Efficacy, respectively, for the sulfur mustard simulant contaminant CEPS decontamination study conducted using DeconGel 1101 as the decontaminant on a number of coupon surfaces including Cadmium-plated and Carbon Steels, CARC, and Silicone and Black Rubbers by solvent extraction.

- As seen in Table 2, when analyzing Decontamination Efficacy and reduction in starting challenge Percent Efficacy data it is noted: 1) on non-porous inert coupon surfaces such as Cadmium-plated and Carbon Steels, excellent Surface Decontamination of DeconGel 1101 on contaminant CEPS was achieved ranging from 96.3% (on Cd-plated Steel) to 97.2% (on Carbon Steel) as determined by residue analysis after solvent extraction; and 2) on porous and/or chemically active coupon surfaces DeconGel 1101 decontamination efficacy and reduction in starting challenge Percent Efficacy ranged from 51.2% (on CARC) to 51.8% (on Silicone Rubber) to 76.9% (on Black Rubber) as determined by residue analysis after solvent extraction. Overall, the decontamination study using decontaminant DeconGel 1101 on CEPS contaminant reveals DeconGel's ability to encapsulate/emulsify hydrophobic contaminants such as CEPS and other chlorinated organo-sulfur/ethyl sulfides. Additionally, due to its viscous, slow-drying, and emulisifying/encapsulating properties, DeconGel 1101 can serve as a partial chemical (vapor) barrier against agents such as sulfur mustards or other more toxic substances that might pose a significant contact risk or possess prolonged environmental persistence.

- For porous and/or chemically active coupon surfaces such as CARC and Rubbers, modest to good surface decontamination of the CEPS was realized; interestingly, CEPS was noted to appreciably absorb into such coupon surfaces, which seemed to sequester/dissolve CEPS within the coupon surfaces, hindering sufficient decontaminant-contaminant interaction and limiting DeconGel 1101's abilities to encapsulate/emulsify contaminant CEPS as reflected by low Decontamination and Percent Efficacies (Table 2).
- Since live agents may not exhibit the same bioactivity, volatility, or absorption characteristics as CEPS, live agent testing is recommended to accurately measure DeconGel effectiveness on test surfaces.

7.7. Technical Report for CWA VX-Simulant (Methyl Benzoate)

TITLE: Surface Decontamination of the VX Simulant Methyl Benzoate (MBz) by DeconGel 1101

ANALYST: Geoff Nadolski PhD, Senior Polymer Scientist, CBI Polymers, Inc.

SUPERVISOR: Garry J. Edgington PhD, Chief Scientist, CBI Polymers, Inc.

OBJECTIVE: Evaluate the decontamination efficacy of DeconGel 1101 on coupon types:

1) Cadmium-plated Steel,
2) Carbon Steel,
3) Chemical Agent Resistant Coating (CARC),
4) Silicone Rubber, and
5) Black Rubber.

- Experimentation and Reporting conducted following JPEO-CBD Source Document:
    - 2007 Chemical Decontamination Performance Evaluation Testing,
    - Section 6: Panel Contact Test to Determine Contact Hazard,
    - Section 6-E: Panel (Coupon) Extraction Method to Determine Remaining Agent;
- Sensitive (low ppb-level) GC/MS (gas chromatography/mass spectrometry) analytical methods developed.

HAZARDOUS MATERIALS RELEVANCE: VX, an organo-phosphono-thioate, is a toxic nerve agent classified as a weapon of mass destruction by the United Nations. VX can be distributed as a liquid or aerosol, both pure and as a mixture, and due to its high viscosity and low volatility is considered an environmentally persistent biohazard. VX and its simulant, methyl benzoate, (MBz) have near identical Water:Octanol Coefficients (2.09 and 2.12, respectively), and as such behave similarly in both environmental settings and biological systems.

SUMMARY RESULTS:

- On the non-porous steel surfaces tested, excellent surface decontamination was achieved by applying DeconGel 1101 onto contaminated surfaces facilitating encapsulation/emulsification of contaminants by DeconGel's active components.
    - DeconGel 1101 reduction in starting challenge Percent Efficacies, a measure comparing samples to the Dose Confirmation Control, ranged from 97.2% (on Cd-plated steel) to 99.7% (on Carbon steel) as determined by solvent extraction;
    - DeconGel 1101 Decontamination Efficacies, a measure comparing samples to Positive Control 1 (adjusted accordingly regarding particular coupon surface area), ranged from 96.7% (on Cd-plated steel) to 99.2% (on carbon steel) as determined by solvent extraction.
- On porous and/or chemically active coupon surfaces such as CARC and Black and Silicone Rubbers, modest Surface Decontamination of MBz (a volatile VX simulant) was realized; even on delicate or difficult-to-clean customized rubber, epoxy, or polyester surfaces, DeconGel is able to effectively remove hydrophobic contaminants through encapsulation/emulsification.
    - Reduction in starting challenge (Percent Efficacies of DeconGel 1101) ranged from 37.9% (on Black Rubber) to 67.0% (on Silicone Rubber) to 91.8% (on CARC) as determined by solvent extraction.
    - DeconGel 1101 Decontamination Efficacies ranged from 26.5% (on Black Rubber) to 62.3% (on Silicone Rubber) to 67.3% (on CARC) as determined by solvent extraction.
- DeconGel 1101 can serve as a partial chemical (vapor) barrier. The vapor barrier effect demonstrates the reduction in the rate of release of agent to the surrounding atmosphere as well as indicating a prolonged residence-time inside the gel layer. An extended residence-time in the gel is predicted to enhance the efficacy of neutralizing agents that are proposed to be employed in future versions of the gel. This reduction in rate of release of agents is proposed to be especially valuable in enhancing neutralization efficacies of CWAs with high reactivity and volatility.

  To support the hypothesis that DeconGel 1101 can serve as a partial chemical (vapor) barrier, additional experimentation ((+)-Control 2) was performed to demonstrate the Chemical Barrier Efficiency of DeconGel 1101.

- On CARC and Black and Silicone Rubber surfaces, DeconGel 1101 Chemical Barrier Efficiencies ranged from 99.5% (on CARC) to 78.6% (on Silicone Rubber) to 99.0% (on Black Rubber) as determined by solvent extraction, indicating a significant chemical surface barrier capability for DeconGel 1101 when applied to porous, chemically active rubber/ epoxy/polyester surfaces able to absorb and entrain contaminants;
- On non-porous, chemically inert Cd- and Carbon Steel surfaces, due to MBz's significant volatility, contaminant applied to the steel coupons was not detectable after undergoing a 24-hour coupon-contaminant incubation as implemented for positive controls ((+)-Control 2). As such, samples and controls could not be compared, and therefore Chemical Barrier Efficiencies for Cd- and Carbon Steel surfaces could not be determined.

RESULTS: Tables 1 and 2 contain the Data Test Set, and Decontamination Efficacy, Reduction in Starting Challenge Percent Efficacy, and Chemical Barrier Efficiency, respectively, for the VX simulant contaminant MBz decontamination study conducted using DeconGel 1101 as the decontaminant on a number of coupon surfaces including Cadmium-plated and Carbon Steels, CARC, Silicone Rubber and Black Rubbers by solvent extraction.

Table 1. DeconGel decontaminant Data Test Set for MBz decontamination study on multiple surfaces as determined by residue analysis after solvent extraction

Coupon Type		RA_M (ng)	CD (g/m^2)
Cd-plated Steel	(-)-Control[1]	ND[2]	ND
	Remaining Agent	60302 ± 3593	0.05 ± 0.003
	(+)-Control 1[3]	1864078 ± 11034	1.46 ± 0.01
	(+)-Control 2[4]	ND	ND
	Dose Confirmation	2161275 ± 5981	N/A[5]
Carbon Steel	(-)-Control	ND	ND
	Remaining Agent	6266 ± 305.1	0.004 ± 0.001
	(+)-Control 1	792602 ± 25573	0.44 ± 0.02
	(+)-Control 2	ND	ND
	Dose Confirmation	2161275 ± 5981	N/A
CARC	(-)-Control	ND	ND
	Remaining Agent	176724 ± 8643	0.09 ± 0.004
	(+)-Control 1	543472 ± 10586	0.28 ± 0.01
	(+)-Control 2	885.77 ± 8.36	0.0005 ± 0.00006
	Dose Confirmation	2161275 ± 5981	N/A
Silicone Rubber	(-)-Control	ND	ND
	Remaining Agent	712525 ± 13315	0.36 ± 0.01
	(+)-Control 1	1890487 ± 15744	0.96 ± 0.01
	(+)-Control 2	126980.4 ± 463.5	0.06 ± 0.0006
	Dose Confirmation	2161275 ± 5981	N/A
Black Rubber	(-)-Control	ND	ND
	Remaining Agent	1341331 ± 8618	0.80 ± 0.01
	(+)-Control 1	1827953 ± 7127	1.08 ± 0.01
	(+)-Control 2	12989.8 ± 70.59	0.008 ± 0.00006
	Dose Confirmation	2161275 ± 5981	N/A

[1] (-)-Control: no contaminant applied, then decontaminant applied

[2] ND: not detected, See Test Specific section, lower limits of detection (LOD) for MBz using GC/MS ≤ 40 ppb

[3] (+)-Control 1: contaminant applied for 1 h, no decontaminant applied

[4] (+)-Control 2: contaminant applied for 24 h, no decontaminant applied

[5] N/A: Not Applicable

RA_M: Remaining Agent (mass); CD: Contamination Density

Table 2. Decontamination Efficacy, Percent Efficacy, and Chemical Barrier Efficiency evaluation for the VX simulant MBz decontamination study on multiple surfaces as determined by residue analysis after solvent extraction

Coupon Type	Decontamination Efficacy[1] (%)	Percent Efficacy[2] (%)	Chemical Barrier Efficiency[3] (%)
Cd-plated Steel	96.7 ± 0.19	97.2 ± 0.19	ND[4]
Carbon Steel	99.2 ± 0.11	99.7 ± 0.00	ND
CARC	67.3 ± 2.34	91.8 ± 0.42	99.5 ± 0.00
Silicone Rubber	62.3 ± 1.17	67 ± 0.96	78.6 ± 0.48
Black Rubber	26.5 ± 1.88	37.9 ± 0.98	99.0 ± 0.00

[1] See Decontamination Efficacy equation (Eqn 1) in Experimental section

[2] See Percent Efficacy equation (Eqn 2) in Experimental section

[3] See Chemical Barrier Efficiency equation (Eqn 3) in Experimental section

[4] ND: not determined (no contaminant detected for samples and/or controls)

METHOD DEVELOPMENT OPTIMIZATIONS/OBSERVATIONS:

- Due to MBz's low boiling point/volatility, significant loss of contaminant MBz was noted when storing contaminated coupons in a chemical hood for the required 60 min contact hold time. Utilizing an inverted Pyrex dessicator greatly improved both volatile surface retention and contaminant-coupon interaction, as evidenced after the 60 min hold time as either a significant surface retention of wetted drops, or as a raised surface.

- In applying a small amount (2.0 uL) of neat MBz contaminant as the starting challenge, there was no need to brush the volatile liquid contaminant throughout coupon surfaces to ensure a thin layer of MBz was loaded onto coupon surfaces; using a minute amount of contaminant facilitated a favorable interaction between DeconGel and MBz without overloading the relatively small amount of applied DeconGel decontaminant (1.0-1.1 g).

- Acetonitrile solvent was used to dissolve MBz contaminant and to generate a standard calibration curve possessing a significant concentration range and favorable accuracy and precision. Acetonitrile was used as the extraction solvent due to its ability to readily solvate methyl benzoate, compatibility with GC/MS analysis, relatively low volatility, and low toxicity profile. In accordance with the JPEO guidelines, 20 mL of acetonitrile extraction solvent was effective in generating precise data as evidenced by the low standard deviation of the modestly concentrated Dose Confirmation Samples ((DCS); DCS avg= 2161275 ± 5981 ng)

- VX and its simulant methyl benzoate (MBz) have near identical Water:Octanol Coefficients (2.09 and 2.12, respectively), and as such behave similarly in both environmental settings and biological system, qualifying MBz as a suitable VX chemical warfare simulant.

- GC/MS instrumentation is a sensitive and accurate analytical tool routinely used for the qualitative and quantitative determination of small- to medium-sized, low- to medium-polarity organic compounds. A greater than ten-point calibration curve utilizing three independent stock solutions was generated, exhibiting a calibrated range for the standard calibration curve of 0.040-360 ppm (wt/wt) = 629-5659200 ng MBz. The calibration curve exhibits excellent fit as demonstrated by the coefficient of determination of linear regression R^2, where $R^2 = 0.99$ (Figure 2). The limits of detection (LOD) and quantitation (LOQ) normal MS scan mode, the MS method used for the present decontamination study, are ≤ 40 ppb and ≤ 80 ppb, respectively, however in using either SIM (single-ion mode) or CI (chemical ionization, using methane) MS capabilities, the lowest limits of detection/quantitation of MBz approach ≤ 1 ppb (with SIM), and with CI could potentially approach ≤ 1 ppt (parts per trillion) or lower.

- As seen in Table 2, for Decontamination Efficacy and reduction in starting challenge Percent Efficacy determinations, on non-porous inert coupon Steel surfaces excellent surface decontamination was achieved by applying DeconGel 1101 onto contaminated surfaces facilitating encapsulation/emulsification of contaminants by DeconGel's active components.

- On porous and/or chemically active coupon surfaces such as CARC and Rubbers, modest surface decontamination of the volatile plasticizer MBz is noted; specifically, when reviewing the Percent Efficacy achieved in comparing remaining contaminant agent versus contaminant confirmation dosage, modest (for Black and Silicone Rubbers) to good (for CARC) surface decontamination of MBz was achieved. DeconGel is able to effectively encapsulate/emulsify hydrophobic contaminants such as MBz or other phosphate/carbonate esters from such porous, delicate, or difficult-to-clean customized surfaces as rubbers and epoxy/polyesters.

- To gauge DeconGel 1101's capabilities as an effective chemical (vapor) barrier, additional experimentation ((+)-Control 2) and data compilation provided a measure of DeconGel 1101's chemical barrier capacity - termed Chemical Barrier Efficiency (see Table 2). On CARC and Black and Silicone Rubber surfaces, calculated DeconGel 1101 Chemical Barrier Efficiencies indicate a significant chemical surface barrier capability when applied to porous, chemically active rubber/ epoxy/polyester surfaces that are able to absorb and entrain contaminants. On non-porous, chemically inert Cd- and Carbon Steel surfaces, due to MBz's significant volatility, contaminant applied to the steel coupons was not detectable after undergoing a 24-hour coupon-contaminant incubation as implemented for positive controls ((+)-Control 2). As such, samples and controls could not be compared, and therefore Chemical Barrier Efficiencies for Cd- and Carbon Steel surfaces could not be determined.

EXPERIMENTAL SECTION:

Reagents:

- Reagent grade methyl benzoate (Fisher Scientific; Fair Lawn, NJ) was utilized as the starting challenge (2.0 uL) contaminant in the decontamination study.
- Proprietarily formulated DeconGel 1101 was used as the decontaminant such that 1.0-1.1 g wet DeconGel was applied to contaminated surfaces, then allowed to dry for 24 h, and finally peeled off the surface. Immediately following gel peeling, the decontaminated coupon was extracted with solvent as elaborated below.
- Acetonitrile was used as the extraction solvent due to its ability to readily solvate methyl benzoate, compatibility with GC/MS analysis, relatively low volatility, and low toxicity profile. In addition, generated analytical calibration curves using acetonitrile exhibited favorable accuracy and precision.

Equipment (see Figure 1):

- A manual 2.0 uL glass syringe (National Scientific; Rockwood, TN) with a volume dispensing range of 0.0 to 2.0 uL, 0.01 uL grading, was used to dispense MBz contaminant, such that two non-touching 1.0 uL drops were dispensed on coupon surfaces per test.
- Calibrated manual pipets (Fisher Scientific; Fair Lawn, NJ) with disposable tips were used to generate the MBz calibration curve used to analytically determine amounts of MBz.
- A plastic squeezable bottle with coned dispenser was supplied with DeconGel 1101 and used to dispense 1.0-1.1 g wet DeconGel upon contaminated surfaces.
- Extraction solvent acetonitrile (20 mL) was dispensed into a 100 mL polypropylene bottle with screwcap prior to introduction of contaminated coupons using a 50 mL graduated cylinder.
- Samples and controls were prepared without further dilution/manipulation and immediately analyzed via GC/MS upon sample preparation.
- Due to the low boiling point of MBz, the 60 min contaminant contact time with coupon surfaces was conducted under an inverted Pyrex dessicator in attempts to minimize evaporative loss of MBz contaminant.

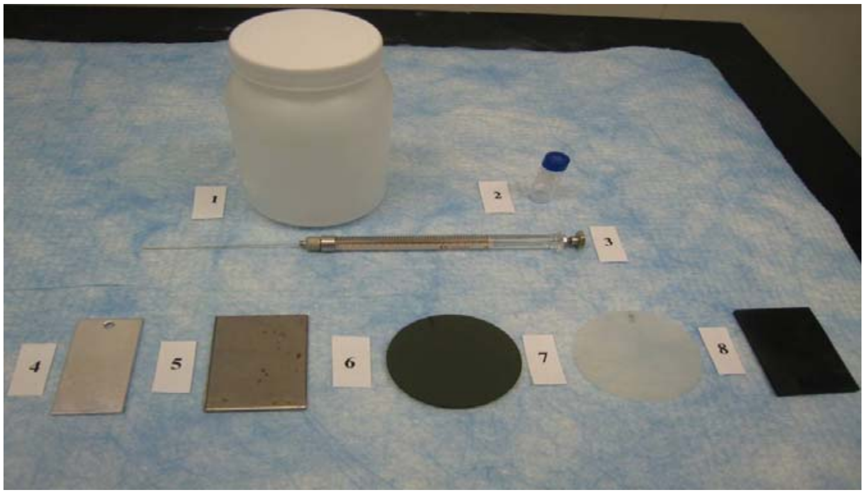
- Coupon area measurements in inches were performed using a standard ruler. Unit conversion and calculation was then conducted to achieve a coupon area in cm^2. Wet DeconGel was dispensed on coupons bordering the coupon edges and then the surface void filled in evenly as to maximize the spreading of DeconGel throughout the entire coupon surface area.

- A Thermo DSQ II capillary GC-double quadrupole MS with autosampler in positive mode was used to determine MBz concentration (ppm, wt/wt) of all samples and controls using a > 10 point calibration curve utilizing three independent stock solutions.

Materials (see Figure 1):

- Coupons utilized in the decontamination study of MBz include Cadmium-plated Steel (12.75 cm^2 surface area), Carbon Steel (17.85 cm^2 surface area), Chemical Agent Resistant Coating (CARC) (19.63 cm^2 surface area), Black Rubber (16.83 cm^2 surface area), and Silicone Rubber (19.63 cm^2 surface area). All coupons were supplied by JPEO to Dr. Garry Edgington (CBI Polymers, Inc.; Honolulu, HI) and were conditioned on a laboratory countertop overnight.

Figure 1. Equipment and Materials utilized for the VX simulant MBz decontamination study on multiple surfaces



Legend: **1.** 100 mL wide mouth extraction bottle with screw cap, **2.** GC vapor-tight sampling vial, **3.** 2 uL syringe for contaminant dispensing, **4.** Cd-plated Steel coupon, **5.** Carbon Steel coupon, **6.** CARC coupon, **7.** Silicone Rubber coupon, **8.** Black Rubber coupon.

Calculations:

- **Eqn 1.** Decontamination Efficacy

  = [(Contamination Density (CD) (g/m^2) of (+)-Control 1 – CD (g/m^2) of Remaining Agent) / CD (g/m^2) of (+)-Control 1] x 100%

- **Eqn 2.** Percent Efficacy

  = [(RA_M (ng) of Dose Confirmation Mass – RA_M (ng) of Remaining Agent Mass) / RA_M (ng) of Dose Confirmation Mass] x 100%

- **Eqn 3.** Chemical Barrier Efficiency

  = [1 – (RA_M (ng) of (+)-Control 2 / (RA_M (ng) of Remaining Agent Mass – (RA_M (ng) of (+)-Control 2))] x 100%

Detailed Test Summary:

- Experimental decontamination sampling and controls (positive and negative) were conducted on five different coupon types (as previously mentioned) in five replicates.

- Upon addition of 2.0 uL of MBz (experimental samples and positive control) to coupon surfaces, contaminated coupons were placed under an inverted Pyrex dessicator for 60 min, and then either a) placed in extraction solvent via extraction bottle (for positive controls), or b) 1.0-1.1 g of wet DeconGel 1101 was poured evenly and thoroughly throughout the coupon surface area, DeconGel was allowed to dry for 24 h, the dried gel was peeled from the contaminated surface, and the contaminated coupon was immediately placed in an inert polypropylene bottle pre-filled with 20 mL extraction solvent as to completely submerse the coupon in extraction solvent. The extraction bottle was screw-capped, then swirled to facilitate contaminant extraction and let to stand for 60 min. Upon sample/control preparation, all experiments were immediately set-up for GC/MS analytical analysis in appropriate screw-cap GC vapor-tight vials (Fisher Scientific; Fair Lawn, NJ) (Figure 1).

- For negative controls, no contaminant was applied to coupon surfaces, and instead 1.0-1.1 g of wet DeconGel 1101 was poured evenly and thoroughly throughout the coupon surface area. DeconGel was allowed to dry for 24 h, the dried gel was peeled from coupon surfaces, and the coupon placed in an inert polypropylene bottle pre-filled with 20 mL extraction solvent as to completely submerse the coupon in extraction solvent. The extraction bottle was screw-capped, then swirled and let to stand for 60 min. Upon sample/control preparation, all experiments were immediately set-up for GC/MS analytical analysis.

- For positive controls, 2 uL of contaminant was applied to coupon surfaces and applied for either a) 1 h ((+)-Control 1), or b) 24 h ((+)-Control 2) coupon-contaminant incubation time, and then treated as described above for sample preparation. Positive Control 1 was implemented to provide an experimental control addressing the typical 60 min coupon-contaminant contact time; Positive Control 2 was implemented to provide an experimental control addressing the 24 h surface residence time employed for both contaminant and decontaminant, the timeframe needed to afford both adequate drying, and optimized surface decontamination by DeconGel 1101. After the appropriate incubation time, no decontaminant was applied, and contaminated coupons were immediately placed in an inert polypropylene bottle pre-filled with 20 mL extraction solvent. The extraction bottle was screw-capped, then swirled to facilitate contaminant extraction and let to stand for 60 min. Upon sample/control preparation, all experiments were immediately set-up for GC/MS analytical analysis.
- For dose confirmation samples, seven replicates were performed such that 2.0 uL MBz was charged via glass syringe into an inert polypropylene bottle pre-filled with 20 mL extraction solvent. The extraction bottle was screw-capped, then swirled to facilitate contaminant extraction and let to stand for 60 min. Upon sample preparation, all experiments were immediately set-up for GC/MS analytical analysis.

TEST SPECIFIC:

Precondition Coupons:

- Coupons were conditioned for 24 h on the laboratory countertop under normal ambient air-conditioned laboratory RT (25 °C) and humidity (40-60% humidity), where no extreme temperatures or humidity fluctuations were noted.

Contamination:

- MBz coupon contamination was performed (as previously mentioned) such that 2.0 uL total MBz neat liquid at RT (2x 1.0 uL non-touching drops) was manually added via glass syringe. Contaminated coupons were immediately placed under an inverted Pyrex dessicator and let to stand for 60 min.

- Contamination density (CD) of coupons were optimized to utilize the entire surface area of coupons by applying two non-touching 1.0 uL contaminant drops, and by applying DeconGel decontaminant on top and evenly over the whole coupon surface area. Coupons utilized in the decontamination study of MBz include Cadmium-plated Steel (12.75 cm^2 surface area), Carbon Steel (17.85 cm^2 surface area), Chemical Agent Resistant Coating (CARC) (19.63 cm^2 surface area), Black Rubber (16.83 cm^2 surface area), and Silicone Rubber (19.63 cm^2 surface area).

Dose Confirmation Sample Preparation:

- For dose confirmation samples, seven replicates were performed such that 2.0 uL MBz was charged via glass syringe into an inert polypropylene bottle pre-filled with 20 mL acetonitrile extraction solvent. The extraction bottle was screw-capped, then swirled to facilitate contaminant extraction and let to stand for 60 min. Upon sample preparation, all experiments were immediately set-up for GC/MS analytical analysis.

Post-Contamination Surface Contamination Observation:

- On non-porous inert coupon surfaces such as Cadmium-plated Steel and Carbon Steel, we observed that MBz contaminant drops initially beaded on the surface. However, after about 20 min, drops applied to Carbon Steel slowly spread out to cover roughly 2-3x the initial surface area, while contaminant drops applied to Cadmium-plated Steel stayed beaded throughout the 60 min contaminant-coupon contact hold time. After the 60 min hold time, MBz contaminant drops were evidenced on non-porous steel surfaces as wetted drops (Cd-plated Steel) or as wetted smears (Carbon Steel); after the 24 h hold time employed for (+)-Control 2, no MBz contaminant drops were evidenced on non-porous steel surfaces as either drops or smears.

- On porous and/or chemically active coupon surfaces such as CARC and Silicone Rubber, MBz contaminant drops were observed to rapidly spread out from the initial drop surface area over 10 min, becoming absorbed into coupon surfaces as evidenced by a wetted surface roughly 2-3x the initial surface area that persisted for the 60 min hold time; after the 24 h hold time employed for (+)-Control 2, no MBz contaminant drops were evidenced on these surfaces. When MBz drops were applied on top of Black Rubber, the contaminant did not roll or spread out, but instead absorbed within minutes into the coupon surface as evidenced by a dry surface bulging that persisted throughout the 24 h hold time employed for (+)-Control 2.

Aging:

- Coupons were conditioned for 24 h on the laboratory countertop under normal ambient air-conditioned laboratory room temperature (25 ºC) and humidity (40-60% humidity), where no extreme temperature or humidity fluctuations were noted.

Post-Aging Surface Contamination Observation:

- No significant difference in appearance, size, or color of conditioned/aged coupons were noted, indicating that no significant or unexpected swelling, shrinking, deformation, decomposition, or surface drying or wetting had occurred.

Pre-Rinse:

- No pre-rinsing step of coupons was performed. Coupons were delivered to CBI Polymers directly from JPEO in a residue-free, clean state and used as received.
- Coupons were conditioned for 24 h on the laboratory countertop under normal ambient air-conditioned laboratory RT (25 ºC) and humidity (40-60% humidity), where no extreme temperatures or humidity fluctuations were noted.

Decontamination:

- To decontaminate the contaminated coupons, after the 60 min contaminant-coupon surface hold time under an inverted Pyrex dessicator, 1.0-1.1 g of wet DeconGel 1101 at room temperature was poured evenly on top of the contaminated coupon throughout the whole surface area in an attempt to decontaminate all contaminant that might have spread throughout the coupon surface area.
- DeconGel was allowed to dry for 24 h, the dried gel was peeled from the contaminated surface, and the contaminated coupon was immediately placed in an inert polypropylene bottle pre-filled with 20 mL extraction solvent as to completely submerse the coupon in extraction solvent. The extraction bottle was screw-capped, then swirled to facilitate contaminant extraction and let to stand for 60 min. Upon sample preparation, all experiments were immediately set-up for GC/MS analytical analysis.

Post-Rinse:

- No post-rinsing step of DeconGel decontaminant was performed due to the chemical nature and intended utility of DeconGel; DeconGel is a viscous gel that upon surface contact first spreads out into a thin layer and then must air-dry for 12-24 h before it is peeled off of the contaminated surface. Rinsing either wet or dry DeconGel will result in partial to complete dissolution/emulsification of the decontaminant and most likely reduce its decontamination efficacy.

Drying:

- Upon addition of decontaminant to contaminated surfaces, DeconGel was allowed to dry a RT for 24 h, the dried gel was peeled from the contaminated surface, and the contaminated coupon was immediately placed in an inert polypropylene bottle pre-filled with 20 mL extraction solvent as to completely submerse the coupon in the extraction solvent.

- Subsequent coupon post-washing or drying steps were not conducted or deemed necessary for this decontamination study.

Remaining Agent Measurement:

- Upon addition of 2.0 uL of MBz (experimental samples and positive control) to coupon surfaces, contaminated coupons were placed under an inverted Pyrex dessicator for 60 min, and then either placed in extraction solvent as described above (for positive control) or 1.0-1.1 g of wet DeconGel 1101 was poured evenly throughout the whole coupon surface area. DeconGel was allowed to dry at RT for 24 h, the dried gel was peeled from the contaminated surface, and the contaminated coupon was immediately placed in an inert polypropylene bottle pre-filled with 20 mL extraction solvent to completely submerse the coupon in extraction solvent. The extraction bottle was screw-capped, then swirled to facilitate contaminant extraction and let to stand for 60 min. Upon sample/control preparation, all experiments were immediately set-up for GC/MS analytical analysis in appropriate screw-cap GC vapor-tight vials (Fisher Scientific; Fair Lawn, NJ).

Chromatographic Analysis:

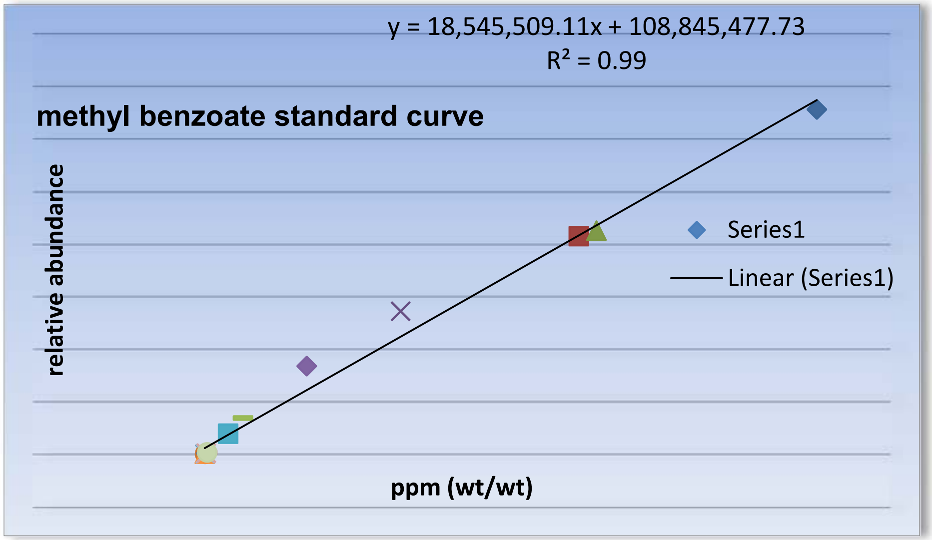
- A Thermo DSQ II capillary GC-double quadrupole MS with autosampler in positive mode was used to determine MBz concentration (ppm, wt/wt) of all samples and controls using a 15 point calibration curve utilizing three independent stock solutions.

- A Restek (Bellefonte, PA) Rtx-5 polysiloxane capillary column was employed using Temperature Program: Initial at 45 ºC, hold 1 min, Ramp at 30 ºC/min to 300 ºC, hold for 5 min.

- The calibrated range for the standard calibration curve is 0.040-360 ppm (wt/wt) = 629-5659200 ng MBz.

- The limits of detection (LOD) and quantitation (LOQ) normal MS scan mode, the MS method used for the present decontamination study, are ≤ 40 ppb and ≤ 80 ppb, respectively, however in using either SIM (single-ion mode) or CI (chemical ionization using methane) MS capabilities the lowest limits of detection/quantitation of MBz approach ≤ 1 ppb (with SIM), and with CI could potentially approach ≤ 1 ppt (parts per trillion) or lower.

Figure 2. MBz Standard Calibration Curve



Reporting Statements/Summary:

- Tables 1 and 2 contain the Data Test Set, and Decontamination Efficacy, Reduction in Starting Challenge Percent Efficacy, and Chemical Barrier Efficacy, respectively, for the VX simulant contaminant MBz decontamination study conducted using DeconGel 1101 as the decontaminant on a number of coupon surfaces including Cadmium-plated and Carbon Steels, CARC, and Silicone and Black Rubbers by solvent extraction.

- As seen in Table 2, when analyzing Decontamination Efficacy and reduction in starting challenge Percent Efficacy data it is noted: 1) on non-porous inert coupon surfaces such as Cadmium-plated and Carbon Steels, excellent Surface Decontamination of DeconGel 1101 on contaminant MBz was achieved ranging from 96.7 and 97.2% (on Cd-plated Steel) to 99.2 and 99.7% (on Carbon Steel) as determined by residue analysis after solvent extraction; and 2) on porous and/or chemically active coupon surfaces DeconGel 1101 decontamination efficacy and reduction in starting challenge Percent Efficacy ranged from 26.5 and 37.9% (on Black Rubber) to 62.3 and 67% (on Silicone Rubber) to 67.3 and 91.8% (on CARC) as determined by residue analysis after solvent extraction. Overall, the decontamination study using decontaminant DeconGel 1101 on MBz contaminant reveals DeconGel's ability to both 1) encapsulate/emulsify hydrophobic contaminants such as MBz and other phosphate/carbonate esters, and 2) act as a chemical barrier against volatile agents such as MBz or other more toxic volatiles/substances that might pose a significant contact risk or possess prolonged environmental persistence.

- For porous and/or chemically active coupon surfaces such as CARC and Rubbers, modest surface decontamination of the volatile plasticizer MBz was realized; interestingly, MBz was noted to appreciably absorb into such coupon surfaces, particularly in regard to both Silicone and Black Rubber coupons, which seemed to sequester/dissolve MBz within the coupon surfaces, hindering sufficient decontaminant-contaminant interaction and limiting DeconGel 1101's abilities to encapsulate/emulsify contaminant MBz as reflected by low Decontamination and Percent Efficacies (Table 2).

- To gauge DeconGel 1101's capabilities as an effective chemical (vapor) barrier, additional experimentation ((+)-Control 2) and data compilation provided a measure of DeconGel 1101's chemical barrier capacity - termed Chemical Barrier Efficiency (see Table 2). On CARC and Black and Silicone Rubber surfaces, calculated DeconGel 1101 Chemical Barrier Efficiencies indicates a significant chemical surface barrier capability when applied to porous, chemically active rubber/ epoxy/polyester surfaces that are able to absorb and entrain contaminants. On non-porous, chemically inert Cd- and Carbon Steel surfaces, due to MBz's significant volatility, contaminant applied to the steel coupons was not detectable after undergoing a 24-hour coupon-contaminant incubation as implemented for positive controls ((+)-Control 2). As such, Chemical Barrier Efficacies for Cd- and Carbon Steel surfaces could not be determined.

- The 60 min contact time between MBz contaminant and all coupons tested was deemed necessary, however adoption of a shorter contact time holds promise to allow the required interaction between contaminant and coupon surface while minimizing loss of volatile contaminant MBz to evaporation. Utilizing an inverted Pyrex dessicator to store contaminated coupons immediately after contamination and during the required 60 min contact time aided in retaining significant amounts of contaminant on/within the coupon surfaces.

- Since live agents may not exhibit the same bioactivity, volatility, or absorption characteristics as MBz, live agent testing is recommended to accurately measure DeconGel effectiveness on test surfaces.

7.8. Technical Report for Jet Fuel

TITLE: Surface Decontamination of Jet Fuel (Jet A) by DeconGel 1101 and 1102

ANALYST: Geoff Nadolski PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVES: Surface decontamination determination of DeconGel formulations 1101 and 1102 on stainless steel, aluminum, and concrete surfaces contaminated with Jet Fuel using GC/MS (Gas Chromatography/Mass Spectrometry) following Environmental Protection Agency (EPA) SW-846 Methods 3500C (Sampling) and 8270C (Analysis).

HAZARDOUS MATERIALS RELEVANCE: Jet Fuel (Jet A) is a C8-C16 kerosene-type flammable fuel. Jet Fuel vapors and liquid are harmful, ingestion of sufficient quantities can be fatal, therefore, Jet Fuel use requires adopting safe and proper handling, storage, and disposal procedures.

SUMMARY RESULTS:

- As seen in Table 1, excellent surface decontamination was achieved by applying DeconGel 1101 and 1102 both via brushing or pouring (non-brushed) onto contaminated surfaces, resulting in encapsulation of Jet Fuel contaminant by DeconGel's active components. Decontamination efficacies of poured DeconGel 1101 ranged from 90.3% (on concrete) to 94.4% (on stainless steel) to 94.5% (on aluminum), brushed DeconGel 1101 ranged from 98.7% (on concrete) to 99.2% (on stainless steel) to 99.4% (on aluminum), as determined by residual swipe analysis. Decontamination efficacies of poured DeconGel 1102 ranged from 89.2% (on concrete) to 92.7% (on stainless steel) to 92.9% (on aluminum), brushed DeconGel 1102 ranged from 98.9% (on concrete) to 99.4% (on stainless steel) to 99.4% (on aluminum), as determined by residual swipe analysis.
- In Table 2, excellent to acceptable surface decontamination was achieved by applying DeconGel 1101 onto contaminated surfaces, resulting in encapsulation of Jet Fuel contaminant by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 91.0% (on concrete) to 95.1% (on stainless steel) to 95.6% (on aluminum) as determined by direct DeconGel analysis. Decontamination efficacies of DeconGel 1102 ranged from 89.2% (on concrete) to 93.6% (on stainless steel) to 93.6% (on aluminum) as determined by direct DeconGel analysis.
- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for

determination of organics in polar solvent solvated samples. When necessary, the digestion methods were customized to result in the complete dissolution of the organic contaminants and to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 and 2 show the decontamination efficacies of DeconGel 1101 on stainless steel, aluminum, and concrete surfaces contaminated with Jet Fuel as determined by residual swipe testing and direct DeconGel analysis.

Table 1. Decontamination efficacies of DeconGel 1101 and 1102 on Jet Fuel (Jet A) contaminated stainless steel, aluminum, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		**Formulation**	**Formulation**
		DeconGel 1101	**DeconGel 1102**
Stainless Steel	Control	221.65 ± 1.23	221.65 ± 1.23
	Residual (non-brushed)	12.42 ± 0.47	16.19 ± 0.54
	Residual (brushed)	1.70 ± 0.02	1.30 ± 0.02
	Decon. Efficacy (non-brushed) (%)	**94.4 ± 0.22**	**92.7 ± 0.43**
	Decon. Efficacy (brushed) (%)	**99.2 ± 0.12**	**99.4 ± 0.15**
Aluminum	Control	219.92 ± 1.40	219.92 ± 1.40
	Residual (non-brushed)	12.09 ± 0.74	15.56 ± 0.55
	Residual (brushed)	1.70 ± 0.01	1.32 ± 0.04
	Decon. Efficacy (non-brushed) (%)	**94.5 ± 0.26**	**92.9 ± 0.16**
	Decon. Efficacy (brushed) (%)	**99.4 ± 0.15**	**99.4 ± 0.15**
Concrete	Control	165.20 ± 1.22	165.20 ± 1.22
	Residual (non-brushed)	16.06 ± 0.45	17.85 ± 0.59
	Residual (brushed)	2.18 ± 0.10	1.83 ± 0.10
	Decon. Efficacy (non-brushed) (%)	**90.3 ± 0.29**	**89.2 ± 0.29**
	Decon. Efficacy (brushed) (%)	**98.7 ± 0.10**	**98.9 ± 0.17**

236x dilution factor for samples and controls

Table 2. Decontamination efficacies of DeconGel 1101 and 1102 on Jet Fuel (Jet A) contaminated stainless steel, aluminum, and concrete surfaces as determined by direct DeconGel analysis.

Direct DeconGel Testing (ppm)		**Formulation**	
		DeconGel 1101	**DeconGel 1102**
Stainless Steel	Control	225.37 ± 0.74	225.50 ± 1.09
	Encapsulated in Gel (non-brushed)	214.32 ± 0.60	211.14 ± 0.68
	Decon. Efficacy (non-brushed) (%)*	**95.1 ± 0.11**	**93.6 ± 0.22**
Aluminum	Control	225.37 ± 0.74	225.50 ± 1.09
	Encapsulated in Gel (non-brushed)	214.52 ± 0.53	211.0 ± 0.68
	Decon. Efficacy (non-brushed) (%)	**95.6 ± 0.10**	**93.6 ± 0.22**
Concrete	Control	225.37 ± 0.74	225.50 ± 1.09
	Encapsulated in Gel (non-brushed)	205.13 ± 1.07	201.12 ± 0.34
	Decon. Efficacy (non-brushed) (%)	**91.0 ± 0.27**	**89.2 ± 0.28**

236x dilution factor for samples and controls

* Decontamination Efficacy by direct DeconGel testing was determined only for non-brushed films, such that brushing DeconGel onto contaminated surfaces results in a loss of some portion of contaminant on brushing tool.

TECHNICAL OBSERVATIONS:

- Application of a homogenous, thin layer of Jet Fuel contaminant on the respective substrate facilitated an optimized interaction between contaminant and DeconGel, and provided an accurate measure of DeconGel's decontamination efficacy in a scaled-down yet real-world setting. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel.

- ASTM method E1728-03, a standardized swipe testing method used for sampling of contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. Air-dried GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes wetted with methanol/DMSO (1:1) solvent were utilized in this swipe testing method.

- Direct chemical analysis of the dried peeled DeconGel samples was also utilized to provide an improved understanding of DeconGel's decontamination efficacy. Decontamination efficacy was determined only for non-brushed (poured) DeconGel films, since brushing DeconGel onto contaminated surfaces resulted in a loss of some portion of contaminant on the brushing tool used, and as such accurate decontamination efficacies for brushed DeconGel 1101 could not be determined. For brushed films, accurate decontamination efficacies were best determined utilizing swipe testing.

- Standardized EPA SW-846 Sampling Method 3500C "Organic Extraction and Sample Preparation" was followed as a guideline to prepare all samples and controls. All samples, controls, and standards were prepared using the same solvent and appropriate experimental conditions to ensure accurate and GC/MS instrumental analysis.
- GC/MS instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of organic compounds. Standardized EPA SW-846 Analytical Method 8270C "Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)" was followed as a guideline to prepare all samples and controls.
- To ensure accurate determination of DeconGel decontamination efficacy, a standard curve of the analyte of interest was prepared using sufficiently pure analyte; the respective standards were diluted to a known concentration (ppm) using the same solvent as used for samples and controls.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

Decontamination Efficacy (DeconGel Testing) =

(Contaminant (ppm) of DeconGel Direct/Contaminant (ppm) of DeconGel Control) x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.20 g Jet Fuel contaminant was evenly applied via brushing on 1) aluminum (surface area: 56.3 cm^2), 2) stainless steel (surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 or 1102 was either poured or brushed (brushed first in a top-bottom, then in a left-right fashion) onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC) wetted with methanol:DMSO (1:1) solvent (2 mL). Swipe and dried film samples were suspended in 50 mL methanol:DMSO (1:1) for 24 h. Samples were then analyzed via GC/MS (see below).

Control Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied via brushing on 1) aluminum (surface area: 56.3 cm^2), 2) stainless steel (surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC) wetted with methanol:DMSO (1:1) solvent (2 mL). Swipe and dried film samples were suspended in 50 mL methanol:DMSO (1:1) for 24 h. Samples were then analyzed via GC/MS (see below).

For DeconGel Control samples, a respectable amount of contaminant and approximately 6.0 g of dry DeconGel1101 or 1102 (pre-poured gel on the respective un-contaminated substrate and let to dry for 24 h) was suspended in 50 mL methanol:DMSO (1:1) for 24 h. Samples were then analyzed via GC/MS (see below).

Reagents and Standards

Jet Fuel (Jet A; sourced Oahu, Hawaii) was used as received.

Analytical Instrumentation

A Thermo DSQII GC/MS with autosampler in positive mode was used to determine Jet Fuel concentration (ppm, wt/wt) of all samples and controls, using a Restek (Bellefonte, PA) Rtx-5 capillary column (30 m x 0.25 mm, 0.5 um).

A 8-point standard curve derived from three independently prepared stock solutions was prepared using methanol:DMSO (1:1) as the working solvent. The calibration curve exhibits a curve fitting as approximated by the coefficient of determination of linear regression R^2, where $R^2 = 0.99$.

GC method: start at 45 °C, hold for 7 min, ramp at 30 °C/min to 320 °C, hold 10 min.

Jet Fuel (Jet A) GC/MS data: 11.0 min; $M^+ = 100$ (C_7H_{16}), 114 (C_8H_{18})

7.9. Technical Report for Lead Compounds (Lead (II) Oxide, Lead (II) Sulfate)

TITLE: Surface Decontamination of Lead Compounds (Lead (II) Oxide (PbO), Lead (II) Sulfate ($PbSO_4$)) by DeconGel 1101

ANALYSTS: Geoff Nadolski PhD, Senior Scientist, and Andreas Mylonakis PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel formulation 1101 on stainless steel, aluminum, and concrete surfaces contaminated with lead (PbO, $PbSO_4$) using ICP-OES (Inductively Coupled Plasma-Optical Emission Spectroscopy) following Environmental Protection Agency (EPA) SW-846 Methods 3005A (sampling) and 6010C (analysis).

HAZARDOUS MATERIALS RELEVANCE: Lead compounds are used in batteries, bullets, as part of solders and alloys, and as a radiation shields. Specifically, lead (II) oxide is used in the production of ceramic glazes and leaded glass, and vulcanized rubber; lead (II) sulfate is commonly used as a component of battery electrodes. Lead and its compounds accumulate in soft tissue and bone, and are potent neurotoxins. The lead compounds PbO and $PbSO_4$ were chosen as representative lead compounds for evaluating DeconGel's efficacy; DeconGel is expected to have similar efficacy towards the wide range of lead compounds.

SUMMARY RESULTS:

- As seen in Tables 1 and 3, excellent to acceptable surface decontamination was achieved by applying DeconGel 1101 onto contaminated surfaces, resulting in encapsulation of Lead contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 on lead (II) oxide ranged from 81.2% (on aluminum) to 91.2% (on concrete) to 91.5% (on stainless steel) as determined by residual swipe analysis; decontamination efficacies of DeconGel 1101 on lead (II) sulfate ranged from 99.4% (on stainless steel) to 99.8% (on concrete) to 99.8% (on aluminum) as determined by residual swipe analysis

- In Tables 2 and 4, excellent to acceptable surface decontamination was achieved by applying DeconGel 1101 onto contaminated surfaces, resulting in encapsulation of lead contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 on lead (II) oxide ranged from 78.7% (on concrete) to 93.4% (on aluminum) to 96.8% (on stainless steel) as determined by direct DeconGel analysis; decontamination efficacies of

DeconGel 1101 on lead (II) sulfate ranged from 87.5% (on concrete) to 97.7% (on aluminum) to 99.6% (on stainless steel) as determined by direct DeconGel analysis

- Lead (II) oxide is very hygroscopic and is evidenced to react with trace amounts of water (on or within both non-porous and porous test surfaces), forming a fixed residue that was not able to be completely removed by DeconGel. Nevertheless, DeconGel showed acceptable decontamination efficacy of loose lead (II) oxide contamination from such surfaces.

- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of inorganic compounds in aqueous samples. When deemed necessary, experimental methods were customized to afford complete dissolution of inorganic contaminants, and to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1-4 show the decontamination efficacies of DeconGel 1101 on stainless steel, aluminum, and concrete surfaces contaminated with lead compounds (PbO, $PbSO_4$) as determined by residual swipe testing and direct DeconGel analysis.

Table 1. Decontamination efficacies of DeconGel 1101 on lead (II) oxide contaminated stainless steel, aluminum, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		**Formulation**
		DeconGel 1101
Stainless Steel*	Control	456.7 ± 15.6
	Residual	39.0 ± 17.3
	Decon. Efficacy (%)	**91.5 ± 5.0**
Aluminum*	Control	433.9 ± 39.0
	Residual	81.4 ± 7.4
	Decon. Efficacy (%)	**81.2 ± 7.4**
Concrete*	Control	405.3 ± 35.6
	Residual	35.5 ± 13.5
	Decon. Efficacy (%)	**91.2 ± 8.7**

2000x dilution factor for samples and controls

* Lead (II) oxide is very hygroscopic and reacts with trace amounts of water (on or within both non-porous and porous test surfaces), resulting in a fixed residue on the contaminated surface that could not be completely removed by DeconGel.

Table 2. Decontamination efficacies of DeconGel 1101 on lead (II) oxide contaminated stainless steel, aluminum, and concrete surfaces as determined by direct DeconGel analysis.

Direct DeconGel Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel*	Control	447.8 ± 6.1
	Encapsulated in Gel (non-brushed)	433.5 ± 12.4
	Decon. Efficacy (non-brushed) (%)*	**96.8 ± 4.4**
Aluminum*	Control	443.6 ± 6.1
	Encapsulated in Gel (non-brushed)	414.3 ± 3.9
	Decon. Efficacy (non-brushed) (%)	**93.4 ± 4.7**
Concrete*	Control	362.5 ± 35.7
	Encapsulated in Gel (non-brushed)	285.2 ± 10.7
	Decon. Efficacy (non-brushed) (%)	**78.7 ± 8.2**

2000x dilution factor for samples and controls

* Lead (II) oxide is very hygroscopic and reacts with trace amounts of water (on or within both non-porous and porous test surfaces), resulting in a fixed residue on the contaminated surface that could not be completely removed by DeconGel.

Table 3. Decontamination efficacies of DeconGel 1101 on lead (II) sulfate contaminated stainless steel, aluminum, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	532.4 ± 14.7
	Residual	3.32 ± 4.2
	Decon. Efficacy (%)	**99.4 ± 3.5**
Aluminum	Control	539.0 ± 4.6
	Residual	0.984 ± 0.15
	Decon. Efficacy (%)	**99.8 ± 8.9**
Concrete	Control	560.4 ± 3.9
	Residual	1.13 ± 0.22
	Decon. Efficacy (%)	**99.8 ± 7.6**

2000x dilution factor for samples and controls

Table 4. Decontamination efficacies of DeconGel 1101 on lead (II) sulfate contaminated stainless steel, aluminum, and concrete surfaces as determined by direct DeconGel analysis.

Direct DeconGel Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	566.7 ± 10.0
	Encapsulated in Gel (non-brushed)	564.4 ± 12.3
	Decon. Efficacy (non-brushed) (%)	**99.6 ± 5.0**
Aluminum	Control	558.3 ± 4.6
	Encapsulated in Gel (non-brushed)	545.6 ± 9.5
	Decon. Efficacy (non-brushed) (%)	**97.7 ± 2.0**
Concrete	Control	564.7 ± 4.0
	Encapsulated in Gel (non-brushed)	494.1 ± 13.8
	Decon. Efficacy (non-brushed) (%)	**87.5 ± 12.1**

2000x dilution factor for samples and controls

TECHNICAL OBSERVATIONS:

- Application of a homogenous, thin layer of lead contaminants on the respective substrate facilitated an optimized interaction between contaminant and DeconGel, and provided an accurate measure of DeconGel's decontamination efficacy in a scaled-down yet real-world setting. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel. Lead (II) oxide and sulfate readily dissolve in aqueous acidic solutions used to prepare all samples and controls (15% HCl, 15% HNO_3, 70% DI H_2O).

- Lead (II) oxide undergoes a chemical reaction with trace amounts of water on or within both non-porous and porous test surfaces which prevents some contamination from being sampled (swipe testing) and encapsulated into dry DeconGel (direct gel testing). Nevertheless DeconGel showed acceptable decontamination efficacy against loose lead (II) oxide contamination.

- ASTM method E1728-03, a standardized swipe testing method used for sampling of inorganic contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes (pre-wetted with DI H_2O) were utilized in this swipe testing method.

- Direct chemical analysis of the dried peeled DeconGel samples was also utilized to provide an improved understanding of DeconGel's decontamination efficacy.

- Standardized EPA SW-846 Sampling Method 3005A "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy" was followed as a guideline to prepare all samples and controls. When deemed necessary, digestion methods were customized by increasing hydrochloric and nitric acid concentrations from 15% to 25% wt, and/or by heating samples to higher temperatures using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC) to afford the complete digestion of the inorganic contaminants. All samples, controls, and standards were prepared using the same dissolution solution and experimental conditions to ensure both correct instrument calibration and accurate analytical results.
- ICP-OES instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of inorganic compounds. Standardized EPA SW-846 Analytical Method 6010C "Inductively Coupled Plasma-Atomic Emission Spectrometry" was followed as a guideline to prepare all samples and controls.
- To ensure accurate determination of DeconGel decontamination efficacy, calibration standards of the analyte of interest were prepared using either a sufficiently pure analyte or an appropriate ICP-MS Standard (Ricca Chemical Company; Arlington, TX); the respective standards were diluted to a known concentration (ppm) using the same digestion method as the one used for samples and controls. Instrument blank controls were DI H_2O ($\geq$17 M-Ohm).

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

Decontamination Efficacy (DeconGel Testing) =

(Contaminant (ppm) of DeconGel Direct/Contaminant (ppm) of DeconGel Control) x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.050 g lead (II) oxide or lead (II) sulfate contaminants were evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) aluminum (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe and gel samples were suspended in 100 mL aqueous acidic solution (15% HCl, 15% HNO_3, 70% deionized (DI) H_2O) for 24 h. When deemed necessary, samples were heated to 94°C for 4-24 h to effectively complete dissolution of inorganics using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC). Samples were allowed to cool to room temperature and were then analyzed via ICP-OES (see below).

Controls Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied on 1) stainless steel (56.3 cm^2), 2) aluminum (commercial grade, 100 cm^2), or 3) concrete (industrial grade, 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 100 mL aqueous acidic solution (15% HCl, 15% HNO_3, 70% DI H_2O) for 24 h and analyzed via ICP-OES (see below).

For DeconGel Control samples, the respective amount of contaminant and approximately 6.0 g dry DeconGel 1101 was suspended in 100 mL aqueous acidic solution (15% HCl, 15% HNO_3, 70% DI H_2O) for 24 h and analyzed via ICP-OES (see below).

Reagents and Standards

Reagent grade Lead (II) Oxide, PbO, (CAS# 1317-36-8, Fisher Scientific; Fair Lawn, NJ) and Lead (II) Sulfate, $PbSO_4$, (CAS# 7446-14-2, Fisher Scientific; Fair Lawn, NJ) were used as received.

1000 ppm calibration standards were prepared using reagent grade PbO and $PbSO_4$ in freshly prepared aqueous acidic solution (15% HCl, 15% HNO_3, 70% DI H_2O). DI H_2O was used as the blank sample.

Analytical Instrumentation

A Thermo ICP-OES instrument model radial iCap 6300 was used to determine lead compound concentration (ppm) of all samples and controls using a freshly prepared 1000 ppm calibration standard. Analyte (lead) analyzed at 220.4 nm;

Pump Speed: 0.5 mL/min

7.10. Technical Report for Mercury (elemental)

TITLE: Surface Decontamination of Mercury (elemental) by DeconGel 1101

ANALYSTS: Geoff Nadolski PhD, Senior Scientist and Andreas Mylonakis PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel formulation 1101 on stainless steel, porcelain tile, composite tile (unwaxed), linoleum tile, and concrete surfaces contaminated with mercury (elemental) using Mercury Vapor Analysis Sensor (Mercury Tracker 3000) following Environmental Protection Agency (EPA) SW-846 Method 7471B: "Mercury in Solid or Semisolid Waste (Manual Cold-Vapor Technique)."

HAZARDOUS MATERIALS RELEVANCE: Mercury is used in thermometers, electronics, lighting, gold and silver refinery, and combined with other metals to form useful amalgams. Mercury and most of its compounds are extremely toxic, causing neurological damage due to inhalation of vapors/dust or ingestion.

SUMMARY RESULTS:

- As seen in Table 1, excellent to acceptable surface decontamination was achieved by applying DeconGel 1101 onto contaminated surfaces, resulting in encapsulation of mercury (elemental) by DeconGel's active components. Decontamination efficacies of poured DeconGel 1101 ranged from 66.0% (composite tile, using Zn-controlled contaminant loading) to 85.9% (porcelain tile, using Zn-controlled contaminant loading) to 90.8% (linoleum tile, using Zn-controlled contaminant loading) to 91.7% (composite tile, using pipet-controlled contaminant loading) to 95.4% (linoleum tile, using pipet-controlled contaminant loading) to 97.8% (concrete, using pipet-controlled contaminant loading) to 98.4% (stainless steel, using pipet-controlled contaminant loading) to 99.2% (porcelain tile, using pipet-controlled contaminant loading); decontamination efficacy of brushed DeconGel 1101 was 99.0% (linoleum tile, using Zn-controlled contaminant loading).
- Mercury (elemental) was evidenced to interact with most plastic surfaces such as un-waxed composite and linoleum tiles, adhering to the plastic surfaces and forming a fixed residue that was not able to be completely removed by DeconGel. Nevertheless, DeconGel showed excellent to acceptable decontamination efficacy of loose mercury (small beads <1 mm diameter) contamination from such surfaces (see Table 1).

- Optimized experimental and analytical methods were successfully developed following a standardized EPA analysis method as a guideline for determination of elemental mercury vapor on solid surfaces. When deemed necessary, experimental methods were customized to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Table 1 shows the decontamination efficacies of DeconGel 1101 on stainless steel, concrete, porcelain tile, composite tile, and linoleum tile surfaces as determined by mercury vapor analysis.

Table 1. Decontamination efficacies of DeconGel 1101 on Mercury (elemental) contaminated stainless steel, concrete, porcelain tile, composite tile, and linoleum tile surfaces as determined by mercury vapor analysis.

Mercury Vapor Analysis Testing (ppt)		**Formulation**
		DeconGel 1101
Stainless Steel	Control [1]	18.6 ± 3.0
	Residual [1]	0.3 ± 0.12
	Decon. Efficacy (%) [1]	**98.4 ± 0.7**
Concrete	Control [1]	18.6 ± 8.9
	Residual [1]	0.4 ± 0.13
	Decon. Efficacy (%) [1]	**97.8 ± 0.7**
Porcelain Tile*	Control [1]	12.2 ± 1.5
	Control [2]	6.4 ± 1.2
	Residual [1]	0.1 ± 0.04
	Residual [2]	0.9 ± 0.6
	Decon. Efficacy (%) [1]	**99.2 ± 0.4**
	Decon. Efficacy (%) [2]	**85.9 ± 11.7**
Composite Tile*	Control [1]	72.1 ± 39.1
	Control [2]	5.0 ± 2.7
	Residual [1]	6.0 ± 1.4
	Residual [2]	1.7 ± 0.8
	Decon. Efficacy (%) [1]	**91.7 ± 2.1**
	Decon. Efficacy (%) [2]	**66.0 ± 11.7**
Linoleum Tile*	Control [1]	60.4 ± 6.6
	Control [2]	20.7 ± 10.0
	Control [3]	20.7 ± 10.0
	Residual [1]	2.8 ± 0.5
	Residual [2]	1.9 ± 1.1
	Residual [3]	0.2 ± 0.1
	Decon. Efficacy (%) [1]	**95.4 ± 0.9**
	Decon. Efficacy (%) [2]	**90.8 ± 5.8**
	Decon. Efficacy (%) [3]	**99.0 ± 0.2**

* Mercury (elemental) has an affinity for plastics, mercury in the form of small beads was noted to adhere to plastic test surfaces (composite tile and linoleum tile), resulting in a fixed residue on the contaminated surface that could not be completely removed by DeconGel.

[1] Contaminant administration controlled using pipet-aided removal of bulk mercury contamination to yield contaminant as very small liquid beads (<1 mm diameter); DeconGel was poured onto contaminated surface

[2] Contaminant administration controlled using zinc dust-assisted removal of bulk mercury contamination to yield contaminant as very small liquid beads (<1 mm diameter); DeconGel was poured onto contaminated surface

[3] Contaminant administration controlled using zinc dust-assisted removal of bulk mercury liquid to yield contaminant as very small liquid beads (<1 mm diameter); DeconGel was brushed onto contaminated surface

TECHNICAL OBSERVATIONS:

- Application of homogeneously dispersed mercury (elemental) contaminant in the form of very small beads (<1 mm diameter) on the respective substrates facilitated an optimized interaction between contaminant and DeconGel, and provided an accurate measure of DeconGel's decontamination efficacy in a scaled-down yet real-world setting. Contaminant administration was controlled using either 1) pipet-aided test surface spreading and then removal of bulk mercury liquid (approximately 0.25 g) initially loaded onto test surfaces, or 2) zinc dust-assisted test surface spreading and then removal (using mercury removal kit, Lab Safety Supply Inc., Janesville, WI) of bulk mercury liquid (approximately 0.25 g) initially loaded onto test surfaces, to yield mercury contaminant in the form of very small liquid beads (<1 mm diameter) spread homogeneously throughout the test surfaces. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel. Mercury (elemental) vapor was analyzed using a mercury vapor analyzer (see below).

- Mercury (elemental) was evidenced to interact with plastic surfaces such as coated porcelain, composite, and linoleum tiles, adhering to the plastic surfaces and forming a fixed residue that was not able to be completely removed by DeconGel. Nevertheless, DeconGel showed excellent to acceptable decontamination efficacy of loose mercury (small beads <1 mm diameter) contamination from such surfaces, as determined using a mercury vapor analyzer (see below).

- Standardized EPA SW-846 Sampling Method 7471B: "Mercury in Solid or Semisolid Waste (Manual Cold-Vapor Technique)" was followed as a guideline to prepare all samples and controls, to ensure both accurate and precise analytical testing results.

- Mercury Vapor Analysis using the Mercury Tracker 3000 (rented from Mercury Instruments USA; Littleton, CO) allows for the continuous measurement of mercury (elemental) concentration (parts per trillion (ppt)) in ambient air, instrument determination of mercury concentration utilizes mercury resonance absorption of 253.7 nm. The Mercury Tracker 3000 contains an internalized mercury lamp used for instrument calibration, and during use the instrument routinely performs instrument calibration to provide accurate analytical determination.

- Direct chemical analysis of the dried peeled DeconGel samples was also utilized to provide an improved understanding of DeconGel's decontamination efficacy.

CALCULATIONS:

Decontamination Efficacy (Mercury Vapor Testing) =

[(Contaminant (ppt) of Control) – (Contaminant (ppt) of Residual)/Contaminant (ppt) of Control] x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, approximately 0.25 g of mercury (elemental) was pipetted onto 1) stainless steel (surface area: 56.3 cm^2), 2) plastic-coated porcelain tile (surface area: 48.8 cm^2), 3) non-waxed composite tile (surface area: 48.8 cm^2), 4) linoleum tile (surface area: 48.8 cm^2), or 5) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Contaminant administration was controlled using either 1) pipet-aided test surface spreading and then removal of bulk mercury contamination initially loaded onto test surfaces, or 2) zinc dust-assisted test surface spreading and then removal (using mercury removal kit, Lab Safety Supply Inc., Janesville, WI) of bulk mercury liquid initially loaded onto test surfaces, to yield mercury contaminant in the form of very small liquid beads (<1 mm diameter) spread homogeneously throughout the test surfaces Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was analyzed for mercury vapor using the Mercury Tracker 3000 (Mercury Instruments USA; Littleton, CO) (see below).

Control Method

For Control samples, a respectable amount of contaminant (see above in Sample Method) was evenly applied on 1) stainless steel (56.3 cm^2), 2) plastic-coated porcelain tile (surface area: 48.8 cm^2), 3) non-waxed composite tile (surface area: 48.8 cm^2), 4) linoleum tile (surface area: 48.8 cm^2), or 5) concrete (industrial grade, surface area: 56.3 cm^2) coupons and the surface was analyzed for mercury vapor using the Mercury Tracker 3000 (Mercury Instruments USA; Littleton, CO) (see below).

Reagents and Standards

Mercury (elemental) (CAS# 7439-97-6, Fisher Scientific; Fair Lawn, NJ) was used as received.

Analytical Instrumentation

A Mercury Tracker 3000 (rented from Mercury Instruments USA; Littleton, CO) was used to determine mercury (elemental) surface vapor concentration (ppt) of all samples and controls. Precise vapor measurements were conducted using a hand-held open-ended wand connected to the instrument detector, such that the wand was passed just above (<0.5 cm) the contaminated surface at a 45 degree, passing the wand first in a top-bottom, then in a left-right fashion.

7.11. Technical Report for Motor Oil

TITLE: Surface Decontamination of Motor Oil by DeconGel 1101 and 1102

ANALYST: Geoff Nadolski PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVES: Surface decontamination determination of DeconGel formulation 1101 and 1102 on aluminum, stainless steel, and concrete surfaces contaminated with Motor Oil (polyaromatic hydrocarbons fraction) using LC/MS (Liquid Chromatography/Mass Spectrometry) following Environmental Protection Agency (EPA) SW-846 Methods 3500C (Sampling), 8321B (Analysis), and 1654A (Analysis of PAH Content of Oil).

HAZARDOUS MATERIALS RELEVANCE: Motor oil is used throughout the world as a combustion engine lubricant. Motor oil is derived from petroleum-based materials including crude oil, and is composed of a variety of saturated and unsaturated hydrocarbons including polyaromatic hydrocarbons (PAHs) (up to 6%). PAHs are widespread organic pollutants known for their carcinogenic, mutagenic, and tetratogenic properties.

SUMMARY RESULTS:

- As seen in Table 1, excellent surface decontamination was achieved by applying DeconGel 1101 and 1102 both via brushing or pouring (non-brushed) onto contaminated surfaces, resulting in encapsulation of Motor oil contaminant by DeconGel's active components. Decontamination efficacies of poured DeconGel 1101 ranged from 87.2% (on concrete) to 96.2% (on aluminum) to 96.2% (on stainless steel), brushed DeconGel 1101 ranged from 98.0% (on concrete) to 99.4% (on stainless steel) to 99.4% (on aluminum), as determined by residual swipe analysis. Decontamination efficacies of poured DeconGel 1102 ranged from 85.6% (on concrete) to 95.3% (on stainless steel) to 95.1% (on aluminum), brushed DeconGel 1102 ranged from 98.5% (on concrete) to 99.5% (on stainless steel) to 99.5% (on aluminum), as determined by residual swipe analysis.
- In Table 2, excellent to acceptable surface decontamination was achieved by applying DeconGel 1101 onto contaminated surfaces, resulting in encapsulation of Motor oil contaminant by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 87.8% (on concrete) to 96.4% (on stainless steel) to 96.8% (on aluminum) as determined by direct DeconGel analysis. Decontamination efficacies of DeconGel 1102 ranged from 86.0% (on concrete) to 95.5% (on stainless steel) to 96.4% (on aluminum) as determined by direct DeconGel analysis.

- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of organics in polar solvent solvated samples. When necessary, the digestion methods were customized to result in the complete dissolution of the organic contaminants and to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 and 2 show the decontamination efficacies of DeconGel 1101 on stainless steel, aluminum, and concrete surfaces contaminated with Motor oil as determined by residual swipe testing and direct DeconGel analysis.

Table 1. Decontamination efficacies of DeconGel 1101 and 1102 on Motor Oil contaminated stainless steel, aluminum, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		**Formulation**	**Formulation**
		DeconGel 1101	**DeconGel 1102**
Stainless Steel	Control	283.30 ± 2.38	283.30 ± 2.38
	Residual (non-brushed)	10.84 ± 0.49	13.26 ± 0.49
	Residual (brushed)	1.70 ± 0.10	1.33 ± 0.10
	Decon. Efficacy (non-brushed) (%)	**96.2 ± 0.16**	**95.3 ± 0.16**
	Decon. Efficacy (brushed) (%)	**99.4 ± 0.12**	**99.5 ± 0.12**
Aluminum	Control	282.06 ± 2.82	282.06 ± 2.82
	Residual (non-brushed)	10.67 ± 0.12	13.13 ± 0.53
	Residual (brushed)	1.71 ± 0.15	1.40 ± 0.04
	Decon. Efficacy (non-brushed) (%)	**96.2 ± 0.16**	**95.3 ± 0.10**
	Decon. Efficacy (brushed) (%)	**99.4 ± 0.16**	**99.5 ± 0.10**
Concrete	Control	204.91 ± 1.64	204.91 ± 1.64
	Residual (non-brushed)	26.23 ± 1.58	29.51 ± 1.11
	Residual (brushed)	4.11 ± 0.19	3.09 ± 0.28
	Decon. Efficacy (non-brushed) (%)	**87.2 ± 0.91**	**85.6 ± 0.64**
	Decon. Efficacy (brushed) (%)	**98.0 ± 0.15**	**98.5 ± 0.15**

11260x dilution factor for samples and controls

Table 2. Decontamination efficacies of DeconGel 1101 and 1102 on Motor Oil contaminated stainless steel, aluminum, and concrete surfaces as determined by direct DeconGel analysis.

Direct DeconGel Testing (ppm)		Formulation	
		DeconGel 1101	**DeconGel 1102**
Stainless Steel	Control	305.6 ± 1.50	303.54 ± 1.25
	Encapsulated in Gel (non-brushed)	294.59 ± 0.73	289.99 ± 1.36
	Decon. Efficacy (non-brushed) (%)*	**96.4 ± 0.24**	**95.5 ± 0.22**
Aluminum	Control	305.6 ± 1.50	303.54 ± 1.25
	Encapsulated in Gel (non-brushed)	295.89 ± 0.21	292.56 ± 1.55
	Decon. Efficacy (non-brushed) (%)	**96.8 ± 0.10**	**96.4 ± 0.16**
Concrete	Control	305.6 ± 1.50	303.54 ± 1.25
	Encapsulated in Gel (non-brushed)	268.32 ± 2.05	261.04 ± 2.44
	Decon. Efficacy (non-brushed) (%)	**87.8 ± 0.75**	**86.0 ± 0.58**

11260x dilution factor for samples and controls

* Decontamination Efficacy by direct DeconGel testing was determined only for non-brushed films, such that brushing DeconGel onto contaminated surfaces results in a loss of some portion of contaminant on brushing tool.

TECHNICAL OBSERVATIONS:

- Application of a homogenous, thin layer of Motor oil contaminant on the respective substrate facilitated an optimized interaction between contaminant and DeconGel, and provided an accurate measure of DeconGel's decontamination efficacy in a scaled-down yet real-world setting. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel.

- ASTM method E1728-03, a standardized swipe testing method used for sampling of contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. Air-dried GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes wetted with methanol/DMSO (1:1) solvent were utilized in this swipe testing method.

- Direct chemical analysis of the dried peeled DeconGel samples was also utilized to provide an improved understanding of DeconGel's decontamination efficacy. Decontamination efficacy was determined only for non-brushed (poured) DeconGel films, since brushing DeconGel onto contaminated surfaces resulted in a loss of some portion of contaminant on the brushing tool used, and as such accurate decontamination efficacies for brushed DeconGel 1101 could not be determined. For brushed films, accurate decontamination efficacies were best determined utilizing swipe testing.

- Standardized EPA SW-846 Sampling Method 3500C "Organic Extraction and Sample Preparation" and EPA Method 1654A "PAH Content of Oil by

HPLC/UV) were followed as a guideline to prepare all samples and controls. All samples, controls, and standards were prepared using the same solvent and appropriate experimental conditions to ensure accurate and LC/MS instrumental analysis.

- LC/MS instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of organic compounds. Standardized EPA SW-846 Analytical Method 8321B "Solvent-Extractable Nonvolatile Compounds by High-Performance Liquid Chromatography/Thermospray/Mass Spectrometry (HPLC/TS/MS) or Ultraviolet (UV) Detection" was followed as a guideline to prepare all samples and controls.
- To ensure accurate determination of DeconGel decontamination efficacy, a standard curve of the analyte of interest was prepared using sufficiently pure analyte; the respective standards were diluted to a known concentration (ppm) using the same solvent as used for samples and controls.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

Decontamination Efficacy (DeconGel Testing) =

(Contaminant (ppm) of DeconGel Direct/Contaminant (ppm) of DeconGel Control) x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.07 g (1.0 mL of 3.5 g/50 mL hexane) Motor oil contaminant was evenly applied via brushing on 1) aluminum (surface area: 56.3 cm^2), 2) stainless steel (surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and then the hexane carrier solvent allowed to evaporate for 20 min. Approximately 6.0 g of DeconGel 1101 or 1102 was either poured or brushed (brushed first in a top-bottom, then in a left-right fashion) onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC) wetted with methanol:DMSO (1:1) solvent (2 mL). Swipe and dried film samples were suspended in 50 mL methanol:DMSO (1:1) for 24 h. Samples were then analyzed via LC/MS (see below).

Control Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied via brushing on 1) aluminum (surface area: 56.3 cm^2), 2) stainless steel (surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC) wetted with methanol:DMSO (1:1) solvent (2 mL). Swipe samples were suspended in 50 mL methanol:DMSO (1:1) for 24 h. Samples were then analyzed via LC/MS (see below).

For DeconGel Control samples, the respective amount of contaminant and approximately 6.0 g of dry DeconGel1101 or 1102 (pre-poured gel on the respective un-contaminated substrate and let to dry for 24 h) was suspended in 50 mL methanol:DMSO (1:1) for 24 h. Samples were then analyzed via LC/MS (see below).

Reagents and Standards

Motor oil (SAE 5W-30, Valvoline) was suspended in methanol/DMSO (1:1) solvent mixture, sonicated for 10 min, let to stand for 24 h, and then the liquid decanted from undissolved material used to generate standard curves.

Analytical Instrumentation

A Thermo LCQ LC/MS with autosampler in positive mode was used to determine PAHs (polyaromatic hydrocarbons) fraction concentration (ppm, wt/wt) of all samples and controls, using a Grace Davison (Deerfield, IL) C18-Select analytical column (150 x 4.6 mm, 5 um).

A 7-point standard curve derived from three independently prepared stock solutions (motor oil dissolved in methanol:DMSO (1:1) was prepared.

LC method using A= water (0.1% formic acid), B=acetonitrile (0.1% formic acid); start at 95%A to 3 min at 70%A, to 6 min at 50%A, to 12 min at 30%A, to 15 min at 90%B, to 16 min at 100%B, hold until 22 min at 100% B, to 23 min at 95%A, hold until 24 min.

Motor oil (PAHs fraction) LC/MS data: 19.8 min; lambda max = 284 nm; no data acquired for MS.

7.12. Technical Report for Navy Dye Marker

TITLE: Surface Decontamination of Navy Dye Marker by DeconGel 1101

ANALYST: Geoff Nadolski PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVES: Surface decontamination determination of DeconGel formulation 1101 on aluminum, stainless steel, and concrete surfaces contaminated with Navy Dye Marker using LC/MS (Liquid Chromatography/Mass Spectrometry) following Environmental Protection Agency (EPA) SW-846 Methods 3500C (Sampling) and 8321B (Analysis).

HAZARDOUS MATERIALS RELEVANCE: Navy Dye Marker is a water-soluble dye used as an ocean marker for a variety of applications and uses. Navy Dye Marker resists short-term environment-mediated degradation and possesses a strong chromophore, and as such even small amounts of dye are plainly visible on both porous and non-porous surfaces, complicating the complete and facile removal of Navy Dye Marker from commonly utilized surfaces.

SUMMARY RESULTS:

- As seen in Table 1, excellent surface decontamination was achieved by applying DeconGel 1101 onto contaminated surfaces, resulting in encapsulation of Navy Dye Marker contaminant by DeconGel's active components. Decontamination efficacies of poured DeconGel 1101 ranged from 99.1% (on concrete) to 99.3% (on aluminum) to 99.4% (on stainless steel) as determined by residual swipe/solvent extraction analysis. Additionally, for concrete substrate, no residual dye was detected visually when adding droplets of water to the concrete panels after DeconGel administration and film peel.
- In Table 2, excellent surface decontamination was achieved by applying DeconGel 1101 onto contaminated surfaces, resulting in encapsulation of Navy Dye Marker contaminant by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 97.8% (on concrete) to 99.1% (on aluminum) to 99.2% (on stainless steel) as determined by direct DeconGel analysis.
- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of organics in polar solvent solvated samples. When necessary, the digestion methods were customized to result in the complete dissolution of

the organic contaminants and to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 and 2 show the decontamination efficacies of DeconGel 1101 on stainless steel, aluminum, and concrete surfaces contaminated with Navy Dye Marker as determined by residual swipe/solvent extraction testing and direct DeconGel analysis.

Table 1. Decontamination efficacies of DeconGel 1101 on Navy Dye Marker contaminated stainless steel, aluminum, and concrete surfaces as determined by residual swipe testing/solvent extraction.

Swipe/Extraction/Visual Testing (ppm)		**Formulation**
		DeconGel 1101
Stainless Steel*	Control	198.63 ± 0.12
	Residual	1.28 ± 0.10
	Decon. Efficacy (%)	**99.4 ± 0.10**
Aluminum*	Control	198.71 ± 0.14
	Residual	1.34 ± 0.10
	Decon. Efficacy (%)	**99.3 ± 0.10**
Concrete**	Control	199.38 ± 0.58
	Residual	1.87 ± 0.14
	Visual inspection	ND***
	Decon. Efficacy (%)	**99.1 ± 0.10**

10000x dilution factor for samples and controls
* Decontamination efficacy determined using residual swipe testing
** Decontamination efficacy determined using solvent extraction
*** ND: not detected (residual dye not detected visually after 5, 24 hours after adding droplets of water to decontaminated concrete panel)

Table 2. Decontamination efficacies of DeconGel 1101 on Navy Dye Marker contaminated stainless steel, aluminum, and concrete surfaces as determined by direct DeconGel analysis.

Direct DeconGel Testing (ppm)		**Formulation**
		DeconGel 1101
Stainless Steel	Control	201.71 ± 0.42
	Encapsulated in Gel (non-brushed)	200.02 ± 0.19
	Decon. Efficacy (non-brushed) (%)*	**99.2 ± 0.10**
Aluminum	Control	201.71 ± 0.42
	Encapsulated in Gel (non-brushed)	199.93 ± 0.21
	Decon. Efficacy (non-brushed) (%)	**99.1 ± 0.10**
Concrete	Control	201.71 ± 0.42
	Encapsulated in Gel (non-brushed)	197.21 ± 1.21
	Decon. Efficacy (non-brushed) (%)	**97.8 ± 0.46**

10000x dilution factor for samples and controls

* Decontamination Efficacy by direct DeconGel testing was determined only for non-brushed films, such that brushing DeconGel onto contaminated surfaces results in a loss of some portion of contaminant on brushing tool.

TECHNICAL OBSERVATIONS:

- Application of a homogenous, thin layer of Navy Dye Marker contaminant on the respective substrate facilitated an optimized interaction between contaminant and DeconGel, and provided an accurate measure of DeconGel's decontamination efficacy in a scaled-down yet real-world setting. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel.

- ASTM method E1728-03, a standardized swipe testing method used for sampling of contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. Pre-wetted (with water) GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes were utilized in this swipe testing method.

- For concrete testing surface, both control and sample coupons contaminated with Navy Dye Marker were extracted with deionized water in a suitably-sized closed plastic bottle, such that concrete coupons were completely submerged in water (50 mL) for 24 h to afford the complete dissolution of analyte in such a porous substrate such as concrete.

- To evaluate leaching dynamics of the Dye in porous substrates such as concrete, after decontamination using DeconGel, water droplets were added to the decontaminated concrete panels to determine if any residual Dye remained on or within the substrate surfaces. Applied water droplets were inspected after 5 and 24 hours of administration, and gratifyingly, no residual dye/coloration was noted for any of the water droplets/concrete surfaces tested.

- Direct chemical analysis of the dried peeled DeconGel samples was also utilized to provide an improved understanding of DeconGel's decontamination efficacy.
- Standardized EPA SW-846 Sampling Method 3500C "Organic Extraction and Sample Preparation" was followed as a guideline to prepare all samples and controls. All samples, controls, and standards were prepared using the same solvent and appropriate experimental conditions to ensure accurate and LC/MS instrumental analysis.
- LC/MS instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of organic compounds. Standardized EPA SW-846 Analytical Method 8321B "Solvent-Extractable Nonvolatile Compounds by High-Performance Liquid Chromatography/Thermospray/Mass Spectrometry (HPLC/TS/MS) or Ultraviolet (UV) Detection" was followed as a guideline to prepare all samples and controls.
- To ensure accurate determination of DeconGel decontamination efficacy, a standard curve of the analyte of interest was prepared using sufficiently pure analyte; the respective standards were diluted to a known concentration (ppm) using the same solvent as used for samples and controls.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

Decontamination Efficacy (Solvent Extraction Testing) =

[(Contaminant (ppm) of Solvent Extraction Control) – (Contaminant (ppm) of Residual Solvent Extraction)/Contaminant (ppm) of Solvent Extraction Control] x 100%

Decontamination Efficacy (DeconGel Testing) =

(Contaminant (ppm) of DeconGel Direct/Contaminant (ppm) of DeconGel Control) x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.005 g (0.50 mL of 0.5 g/50 mL deoinized water) Navy Dye Marker contaminant was evenly applied via brushing on 1) aluminum (surface area: 56.3 cm^2), 2) stainless steel (surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was either 1) swipe tested (ASTM method E1728-03) using pre-wetted (with water) GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes (for aluminum and stainless steel coupons); or 2) extracted with deionized water (50 mL) in a suitably-sized closed plastic bottle as to completely submerge contaminated coupon surfaces with solvent, and let to stand for 24 h to afford the complete dissolution of analyte (for concrete coupons). Swipe and dried film samples were suspended in 50 mL deionized water for 24 h. All samples were then analyzed via LC/MS (see below). To evaluate leaching dynamics of the Dye in concrete (porous substrate), after decontamination using DeconGel, small water droplets (deionized water, 2 mL) were added throughout the area of the decontaminated concrete panels. Applied water droplets were inspected visually after 5 and 24 hours of administration for any notable coloration on the surface or within water droplets.

Control Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied via brushing on 1) aluminum (surface area: 56.3 cm^2) or 2) stainless steel (surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using pre-wetted (with water) GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes. Swipe samples were suspended in 50 mL deionized water for 24 h, and then analyzed via LC/MS (see below).

For Solvent Extraction Control samples, a respectable amount of contaminant was evenly applied via brushing on concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the coupons were extracted with deionized water (50 mL) in a suitably-sized closed plastic bottle as to completely submerge contaminated coupon surfaces with solvent, and let to stand for 24 h to afford the complete dissolution of analyte, and then analyzed via LC/MS (see below).

For DeconGel Control samples, the respectable amount of contaminant and approximately 6.0 g of dry DeconGel1101 was suspended in 50 mL deionized water for 24 h. Samples were then analyzed via LC/MS (see below).

Reagents and Standards

Navy Dye Marker powder (sourced in Oahu, Hawaii) was dissolved in water and used to generate standard curves.

Analytical Instrumentation

A Thermo LCQ LC/MS with autosampler in positive mode was used to determine Navy Dye Marker concentration (ppm, wt/wt) of all samples and controls, using a Grace Davison (Deerfield, IL) C18-Select analytical column (150 x 4.6 mm, 5 um).
A 5-point standard curve derived from three independently prepared stock solutions was prepared using deionized water as the working solvent.
LC method using A= water (0.1% formic acid), B=acetonitrile (0.1% formic acid); start at 90%A to 2 min at 70%A, to 3 min at 50%A, to 5 min at 30%A, to 6 min at 100%B, hold until 16 min at 90% A hold until 17 min.
Navy Dye Marker LC/MS data: 7.58 min; lambda max = 266, 293 nm; M^+ = 333.

7.13. Technical Report for Tin Compounds

TITLE: Surface Decontamination of Tin compounds (tributyltin chloride) by DeconGel 1101

ANALYST: Geoff Nadolski PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVES: Surface decontamination efficacy of DeconGel 1101 on stainless steel, aluminum, and concrete surfaces contaminated with organotin (tributyltin chloride) compound; experimentation and associated analyses using ICP-OES (Inductively Coupled Plasma-Optical Emission Spectroscopy) following Environmental Protection Agency (EPA) SW-846 Methods 3005A (sampling) and 6010C (analysis).

HAZARDOUS MATERIALS RELEVANCE: Tin compounds (organotin) have been extensively used as biocides, wood preservatives, and as anti-biofouling agents, however, concerns over potent toxicity to marine life have led to a worldwide ban by the International Maritime Organization. Organotin compounds are considered environmentally persistent pollutants.

SUMMARY RESULTS:

- As seen in Table 1, excellent surface decontamination was achieved by applying DeconGel 1101 via brushing onto contaminated surfaces, resulting in encapsulation of organotin contaminant by DeconGel's active components. Decontamination efficacies of brushed DeconGel 1101 ranged from 99.0% (on concrete) to 99.3% (on aluminum) to 99.4% (on stainless steel) as determined by residual swipe analysis; decontamination efficacies of poured DeconGel 1101 ranged from 86.9% (on concrete) to 96.2% (on stainless steel) to 96.3% (on aluminum) as determined by residual swipe analysis.

- In Table 2, excellent to acceptable surface decontamination was achieved by applying DeconGel 1101 onto contaminated surfaces, resulting in encapsulation of organotin contaminant by DeconGel's active components. Decontamination efficacies of DeconGel 1101 ranged from 84.9% (on concrete) to 95.2% (on stainless steel) to 95.4% (on aluminum) as determined by direct DeconGel analysis.

- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of inorganics/organometallics in aqueous/polar aprotic solvated samples. When necessary, the digestion methods were customized to result in the complete dissolution of the inorganic contaminants and to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 and 2 show the decontamination efficacies of DeconGel 1101 on stainless steel, aluminum, and concrete surfaces contaminated with organotin compound (tributyltin chloride) as determined by residual swipe testing and direct DeconGel analysis.

Table 1. Decontamination efficacies of DeconGel 1101 on Organotin (tributyltin chloride) contaminated stainless steel, aluminum, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	465.03 ± 2.80
	Residual (non-brushed)	17.84 ± 0.88
	Residual (brushed)	2.82 ± 0.60
	Decon. Efficacy (non-brushed) (%)	**96.2 ± 0.21**
	Decon. Efficacy (brushed) (%)	**99.4 ± 0.10**
Aluminum	Control	468.37 ± 3.39
	Residual (non-brushed)	17.24 ± 0.83
	Residual (brushed)	3.34 ± 0.28
	Decon. Efficacy (non-brushed) (%)	**96.3 ± 0.21**
	Decon. Efficacy (brushed) (%)	**99.3 ± 0.10**
Concrete	Control	341.73 ± 3.79
	Residual (non-brushed)	44.82 ± 0.77
	Residual (brushed)	3.47 ± 0.45
	Decon. Efficacy (non-brushed) (%)	**86.9 ± 0.16**
	Decon. Efficacy (brushed) (%)	**99.0 ± 0.12**

764x dilution factor for samples and controls

Table 2. Decontamination efficacies of DeconGel 1101 on Organotin (tributyltin chloride) contaminated stainless steel, aluminum, and concrete surfaces as determined by direct DeconGel analysis.

Direct DeconGel Testing (ppm)		**Formulation**
		DeconGel 1101
Stainless Steel	Control	466.97 ± 5.58
	Encapsulated in Gel (non-brushed)	444.77 ± 1.63
	Decon. Efficacy (non-brushed) (%)*	**95.2 ± 0.42**
Aluminum	Control	466.97 ± 5.58
	Encapsulated in Gel (non-brushed)	445.37 ± 0.93
	Decon. Efficacy (non-brushed) (%)	**95.4 ± 0.48**
Concrete	Control	466.97 ± 5.58
	Encapsulated in Gel (non-brushed)	396.3 ± 1.25
	Decon. Efficacy (non-brushed) (%)	**84.9 ± 0.88**

764x dilution factor for samples and controls

* Decontamination Efficacy by direct DeconGel testing was determined only for non-brushed application, since brushing DeconGel onto contaminated surfaces results in a loss of some portion of contaminant on brushing tool.

TECHNICAL OBSERVATIONS:

- Application of a homogenous, thin layer of the organotin liquid contaminant on the respective substrate facilitated an optimized interaction between contaminant and DeconGel, and provided an accurate measure of DeconGel's decontamination efficacy in a scaled-down yet real-world setting. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel.

- ASTM method E1728-03, a standardized swipe testing method used for sampling of contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. Air-dried GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes wetted with DMSO solvent were utilized in this swipe testing method.

- Direct chemical analysis of the dried peeled DeconGel samples was also utilized to provide an improved understanding of DeconGel's decontamination efficacy. Decontamination efficacy was determined only for non-brushed (poured) DeconGel films, since brushing DeconGel onto contaminated surfaces resulted in a loss of some portion of contaminant on the brushing tool used, and as such accurate decontamination efficacies for brushed DeconGel 1101 could not be determined. For brushed films, accurate decontamination efficacies were best determined utilizing swipe testing.

- Standardized EPA SW-846 Sampling Method 3005A "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy" was followed as a guideline to prepare all samples and controls. All samples, controls, and standards were prepared using the same dissolution solution and experimental conditions to ensure both correct instrument calibration and accurate analytical results.

- ICP-OES instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of inorganic compounds. Standardized EPA SW-846 Analytical Method 6010C "Inductively Coupled Plasma-Atomic Emission Spectrometry" was followed as a guideline to prepare all samples and controls.

- To ensure accurate determination of DeconGel decontamination efficacy, calibration standards of the analyte of interest were prepared using either a sufficiently pure analyte or an appropriate ICP-MS Standard (Ricca Chemical Company; Arlington, TX); the respective standards were diluted to a known concentration (ppm) using the same digestion method as the one used for samples and controls. Instrument blank controls were DI H_2O ($\geq$17 M-Ohm) and DMSO solvent.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

Decontamination Efficacy (DeconGel Testing) =

(Contaminant (ppm) of DeconGel Direct/Contaminant (ppm) of DeconGel Control) x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.080 g tributyltin chloride contaminant was evenly applied via brushing on 1) aluminum (surface area: 56.3 cm^2), 2) stainless steel (surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was either poured or brushed (brushed first in a top-bottom, then in a left-right fashion) onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC) wetted with DMSO solvent (2 mL). Swipe and dried film samples were suspended in 50 mL DMSO for 24 h. Samples were then analyzed using ICP-OES following EPA SW-846 Method 6010C (analysis).

Control Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied via brushing on 1) aluminum (surface area: 56.3 cm^2), 2) stainless steel (surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC) wetted with DMSO solvent (2 mL). Swipe samples were suspended in 50 mL DMSO for 24 h. Samples were then analyzed using ICP-OES following EPA SW-846 Method 6010C (analysis).

For DeconGel Control samples, a respectable amount of contaminant and approximately 6.0 g of dry DeconGel1101 (pre-poured gel on the respective un-contaminated substrate and let to dry for 24 h) was suspended in 50 mL DMSO for 24 h. Samples were then analyzed using ICP-OES following EPA SW-846 Method 6010C (analysis).

Reagents and Standards

Tributyltin chloride, liquid, (CAS# 1461-22-9, Fisher Scientific; Fair Lawn, NJ), was used as received.

A 1000 ppm calibration standard of contaminant was prepared using tributyltin chloride in DMSO solvent. DMSO was used as the blank sample.

Analytical Instrumentation

A Thermo ICP-OES instrument model radial iCap 6300 was used to determine tin concentration (ppm) of all samples and controls using a freshly prepared 1000 ppm calibration standard.

Analyte (aluminum) analyzed at 189.9 nm

Pump Speed: 0.5 mL/min

7.14. Technical Report for PCBs (In-House, Navy Site Testing)

TITLE: Surface Decontamination of PCBs (PCB-laden oil) by DeconGel 1101 and 1102

ANALYSTS: Geoff Nadolski PhD, Senior Polymer Scientist, Andreas Mylonakis PhD, Senior Polymer Scientist, and Garry Edgington PhD, Chief Scientist, CBI Polymers, Inc.

OBJECTIVES: 1) Surface decontamination determination DeconGel 1102 (optimized for the decontamination of hydrophobic contaminants) on multiple contaminated sites aboard the USS Missouri (BB-63) maritime museum vessel, 2) "In house" surface decontamination determination of DeconGel 1101 and 1102 on stainless steel, aluminum, and concrete surfaces contaminated with PCB (polychlorinated biphenyls) oil; experimentation and associated analyses using GC/MS (Gas Chromatography/Mass Spectrometry) following Environmental Protection Agency (EPA) SW-846 Methods 3500C (Sampling) and 8270C (Analysis), and PCB surface limit standards following EPA Regulations 761.79, 761.123.

HAZARDOUS MATERIALS RELEVANCE: PCBs were widely used as industrial dielectric additives in transformer and capacitor coolants and insulating fluids. Due to undesirable carcinogenicity, PCB production has been banned since the 1970s. PCBs are considered chemically inert and resist environmental degradation, and as such remain environmentally persistent pollutants.

SUMMARY RESULTS:

- USS Missouri maritime museum vessel contains several areas with limited access to the public due to PCB oil contamination. Field testing aboard the USS Missouri maritime museum vessel showed that excellent surface decontamination was achieved by applying DeconGel 1102 onto contaminated surfaces, resulting in encapsulation of PCB contaminant by DeconGel's active components. For all contaminated surfaces tested, EPA PCB limit standards (Regulations 761.79, 761.123) of ≤ 10 ug/100 cm^2 were achieved upon decontamination with DeconGel 1102. Table 1 below summarizes these results.

- As seen in Table 2, “in house” tests show that excellent surface decontamination was achieved by applying both DeconGel 1101 and 1102 via brushing onto contaminated surfaces, resulting in encapsulation of PCB contaminant by DeconGel’s active components. Decontamination efficacies of brushed DeconGel 1101 and 1102 were 100% on concrete, aluminum, and stainless steel as determined by residual swipe analysis.

- “In house” tests show decontamination efficacies of poured DeconGel 1101 ranging from 83.9% (1st application) to 98.6% (2nd application, 3 days after 1st application) to 98.8% (2nd application, 7 days after 1st application) on concrete, 92.4% on aluminum (one application), and 92.4% on stainless steel (one application), as determined by residual swipe analysis. Decontamination efficacies of poured DeconGel 1102 ranged from 87.2% (1st application) to 98.8% (2nd application, 3 days after 1st application) to 99.0% (2nd application, 7 days after 1st application) on concrete, 94.1% on aluminum (one application), and 94.1% on stainless steel (one application) as determined by residual swipe analysis.

- As seen in Table 3, “in house” tests show that excellent to acceptable surface decontamination was achieved using both DeconGel 1101 and 1102 as determined by direct DeconGel analysis. Decontamination efficacies of DeconGel 1101 were 93.3% on stainless steel, 93.3% on aluminum, and 80.4% on concrete as determined by for direct DeconGel analysis; decontamination efficacies of DeconGel 1102 were 95.6% on stainless steel, 95.4% on aluminum, and 82.9% on concrete as determined by for direct DeconGel analysis.

- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of organic compounds dissolved in a suitable solvent able to completely solvate PCBs as well as DeconGel components. When deemed necessary, experimental methods were customized to afford complete dissolution of organic contaminants. Additionally, analytical methods and associated equipment (GC column, GC gradient program, MS sample ionization parameters, PCB analyses for maritime museum vessel) were appropriately developed and outsourced to ensure accurate decontamination determination of DeconGel.

RESULTS: Figure 1 on the next page shows photos taken during the field testing aboard the USS Missouri pcb decontamination field testing. Table 1 shows the decontamination efficacies of DeconGel 1102 on multiple contaminated sites aboard the USS Missouri maritime museum vessel; Table 2 shows the decontamination efficacies of DeconGel 1101 and 1102 in “in house” tests on stainless steel, aluminum, and concrete surfaces contaminated with PCB oil as determined by residual swipe testing; Table 3 shows the decontamination efficacies of DeconGel 1101 and 1102 in “in house” tests on stainless steel, aluminum, and concrete surfaces contaminated with PCB oil as determined by direct DeconGel analysis.

Figure 1. Following series of photo's taken during field testing aboard USS Missouri maritime museum vessel.

Table 1. Decontamination efficacies of DeconGel 1102 on PCB oil contaminated stainless steel, cementacious leveling compound surface, painted deck plate, and brass surfaces as determined by residual swipe testing.

Description	CONTAMINATED SURFACE MATERIAL	Before Decon (ug/100cm^2)	DeconGel1102* After 1st application (ug/100cm^2)	DeconGel1102* After 2nd application (ug/100cm^2)
2-111-2-Q Gen Wksp Oil on #8 Radial Press	**Stainless steel**	89.1ug/ 100cm^2 Aroclor 1254	<1.00ug/ 100 cm^2 (wire brush utilized)	<1.00ug/ 100cm^2 (wire brush utilized)
2-68-0-L Crew Living Space oil on deck from winch (underlayment)	**Cementacious leveling compounds**	14.7ug/ 100cm^2 Aroclor 1254	1.82 ug/ 100 cm^2 Aroclor 1260 (Stand up wire brush utilized)	6.14ug/ 100cm^2 Aroclor 1260 (Stand up wire brush utilized)
4-79-2-C Fwd Battery Plot oil on deck from overhead metal panel	**Painted metal deck plate**	661ug/ 100cm^2 Aroclor 1254	<1.00ug/ 100 cm^2 (brushing with a paint brush)	<1.00ug/ 100cm^2 (brushing with a paint brush)
			3.32 ug/ 100 cm^2 Aroclor 1260 (scrubbing)	<1.00ug/ 100cm^2 (scrubbing)
4-74-2-M 16" FWD Magazine cosmolene film on brass	**Brass**	132ug/ 100cm^2 Aroclor 1254 213ug/ 100cm^2 Aroclor 1260 Total PCBs 345 ug/ 100cm^2	<1.00ug/ 100cm^2 (scrubbing)	<1.00ug/ 100cm^2 (scrubbing)

Above data are from an independent lab. Sample analysis performed by SGS North America Inc.; Environmental Services (Anchorage, AK).

* PCB surface limit standards (following EPA Regulations 761.79, 761.123) $\leq$ 10 ug/100 cm^2.

Table 2. Decontamination efficacies of DeconGel 1101 and 1102 on PCB oil contaminated stainless steel, aluminum, and concrete surfaces as determined by residual swipe testing. (In house evaluations)

Swipe Testing (ppm)		**Formulation**	**Formulation**
		DeconGel 1101	**DeconGel 1102**
Stainless Steel	Control	762.3 ± 4.93	762.3 ± 4.93
	Residual (non-brushed)	57.68 ± 0.42	44.72 ± 0.51
	Residual (brushed)	ND*	ND
	Decon. Efficacy (non-brushed) (%)	**92.4 ± 0.10**	**94.1 ± 0.10**
	Decon. Efficacy (brushed) (%)	**100****	**100****
Aluminum	Control	764.4 ± 5.32	764.4 ± 5.32
	Residual (non-brushed)	57.78 ± 0.31	44.86 ± 0.41
	Residual (brushed)	ND	ND
	Decon. Efficacy (non-brushed) (%)	**92.4 ± 0.10**	**94.1 ± 0.10**
	Decon. Efficacy (brushed) (%)	**100****	**100****
Concrete	Control	645.6 ± 3.69	645.6 ± 3.69
	Residual 1[1] (non-brushed)	103.7 ± 0.88	82.58 ± 0.60
	Residual 1 (brushed)	ND	ND
	Decon. Efficacy (1st Application) (non-brushed) (%)	**83.9 ± 0.18**	**87.2 ± 0.11**
	Decon. Efficacy (1st Application) (brushed) (%)	**100****	**100****
	Residual 2[2] (non-brushed)	9.18 ± 0.79	7.68 ± 0.78
	Residual 2 (brushed)	ND	ND
	Decon. Efficacy (2nd Application/ 72hrs after 1st Application) (non-brushed) (%)	**98.6 ± 0.10**	**98.8 ± 0.15**
	Decon. Efficacy (2nd Application/ 72hrs after 1st Application) (brushed) (%)	**100**	**100**
	Residual 3[3] (non-brushed)	7.87 ± 0.45	6.61 ± 0.66
	Residual 3(brushed)	ND	ND
	Decon. Efficacy (2nd Application/ 168 hrs after 1st Application) (non-brushed) (%)	**98.8 ± 0.10**	**99.0 ± 0.12**
	Decon. Efficacy (2nd Application/ 168 hrs after 1st Application) (brushed) (%)	**100**	**100**

3860x dilution factor for samples and controls

* ND: not detected, limit of detection (LOD) for PCBs approximates 250 ppb

** An ND value for residual experiments results in a Decontamination Efficacy of 100%

[1] Standard 24 h decontamination protocol utilized (24 h required DeconGel drying time)

[2] 2nd DeconGel application (3 days/72 h after 1st application)

[3] 2nd DeconGel application (7 days/168 h after 1st application)

Table 3. Decontamination efficacies of DeconGel 1101 and 1102 on PCB oil on stainless steel, aluminum, and concrete surfaces as determined by direct DeconGel analysis (in-house evaluations).

Direct DeconGel Testing (ppm)		Formulation	
		DeconGel 1101	**DeconGel 1102**
Stainless Steel	Control	814.68 ± 0.73	814.68 ± 0.73
	Encapsulated in Gel (non-brushed)	759.71 ± 1.26	778.85 ± 0.80
	Decon. Efficacy (non-brushed) (%)*	**93.3 ± 0.16**	**95.6 ± 0.46**
Aluminum	Control	814.68 ± 0.73	814.68 ± 0.73
	Encapsulated in Gel (non-brushed)	759.75 ± 1.09	777.57 ± 0.56
	Decon. Efficacy (non-brushed) (%)	**93.3 ± 0.16**	**95.4 ± 0.38**
Concrete	Control	814.68 ± 0.73	814.68 ± 0.73
	Encapsulated in Gel (non-brushed)	654.63 ± 2.13	675.54 ± 1.68
	Decon. Efficacy (non-brushed) (%)	**80.4 ± 0.31**	**82.9 ± 0.25**

3860x dilution factor for samples and controls

* Decontamination Efficacy by direct DeconGel testing was determined only for non-brushed films, such that brushing DeconGel onto contaminated surfaces results in a loss of some portion of contaminant on brushing tool.

TECHNICAL OBSERVATIONS:

- For decontamination studies on the maritime museum vessel USS Missouri, PCB surface limit standards following EPA Regulations 761.79, 761.123 were used as references to determine surface decontamination efficacies; for all surfaces decontaminated, PCB levels were determined to be below the ≤ 10 ug/100 cm^2 limit (see Table 1).

- Gauze pads pre-wetted with hexanes were used to sample 100cm^2 of coverage area before and after decon at the sites of USS Missouri that were decontaminated. Sample wipes were put into tightly sealed pre-labeled amber glass containers and sent to SGS North America Inc.; Environmental Services (Anchorage, AK) for independent lab sample testing of PCB oil.

- ASTM method E1728-03, a standardized swipe testing method used for sampling of contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. Air-dried GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes wetted with DMSO solvent were utilized in this swipe testing method (In house evaluations).

- Direct chemical analysis of the dried peeled DeconGel samples was also utilized to provide an improved understanding of DeconGel's decontamination efficacy. Decontamination efficacy was determined only for non-brushed (poured) DeconGel films, such that brushing DeconGel onto contaminated surfaces resulted in a loss of some portion of contaminant on the brushing tool used, and as such accurate decontamination efficacies for brushed DeconGel 1101 and 1102 could not be determined. For brushed films, accurate decontamination efficacies were best determined utilizing swipe testing.

- Standardized EPA SW-846 Sampling Method 3500C “Organic Extraction and Sample Preparation” was followed as a guideline to prepare all samples and controls. All samples, controls, and standards were prepared using the same solvent and appropriate experimental conditions to ensure accurate and GC/MS instrumental analysis.

- GC/MS instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of organic compounds. Standardized EPA SW-846 Analytical Method 8270C “Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)” and EPA SW-846 Method 8082A “Determination of PCBs by Gas Chromatography” were followed as a guideline to prepare all samples and controls.

- To ensure accurate determination of DeconGel decontamination efficacy, a standard curve of the analyte of interest was prepared using sufficiently pure analyte; the respective standards were diluted to a known concentration (ppm) using the same solvent as used for samples and controls.

- For decontamination studies conducted at CBI Polymers, due to the dynamic nature of PCB oil contaminant leaching/migration within porous substrates such as concrete, consecutive DeconGel film applications were conducted in attempts to provide superior PCB oil decontamination over what is achieved when utilizing a singular DeconGel film application on porous surfaces. After the first decontamination round, a second application of DeconGel was applied for both the poured- (non-brushed) and brushed-film decontamination series performed either 3 days or 7 days after the first DeconGel film application. Gratifyingly, employing a second DeconGel film application facilitated excellent surface decontamination (see Table 2).

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

Decontamination Efficacy (DeconGel Testing) =

(Contaminant (ppm) of DeconGel Direct/Contaminant (ppm) of DeconGel Control) x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.215 g PCB-laden mineral oil (approx. 33145 ppm (wt/wt)) contaminant was evenly applied via brushing on 1) aluminum (surface area: 56.3 cm^2), 2) stainless steel (surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 or 1102 was poured onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC) wetted with DMSO solvent (2 mL). Swipe samples were suspended in 50 mL DMSO for 24 h. Samples were then analyzed via GC/MS following EPA SW-846 Method 8082A, "Determination of PCBs by Gas Chromatography" (see below).

Control Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied via brushing on 1) aluminum (surface area: 56.3 cm^2), 2) stainless steel (surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC) wetted with DMSO solvent (2 mL). Swipe samples were suspended in 50 mL DMSO for 24 h. Samples were then analyzed via GC/MS following EPA SW-846 Method 8082A, "Determination of PCBs by Gas Chromatography" (see below).

For DeconGel Control samples, a respectable amount of contaminant and approximately 6.0 g of either dry DeconGel 1101 or 1102 (pre-poured gel on the respective uncontaminated substrate and let to dry for 24 h) was suspended in 50 mL DMSO for 24 h and analyzed via GC/MS (see below).

Reagents and Standards

PCB standard Aroclor 1016 (CAS# 12674-11-2, Ultra Scientific; Kingston, RI) was dissolved in DMSO solvent used to generate standard curves.

PCB-laden mineral oil was confirmed to be Aroclor 1016 by GC/MS analysis and was found to have an Aroclor 1016 concentration of approximately 33145 ppm (wt/wt).

Analytical Instrumentation

A Thermo DSQII GC/MS with autosampler in positive mode was used to determine PCB (Aroclor 1016) concentration (ppm, wt/wt) of all samples and controls, using a Restek (Bellefonte, PA) Rtx-5 capillary column (30 m x 0.25 mm, 0.5 um).

A 7-point standard curve derived from three independently prepared stock solutions was prepared using DMSO as the working solvent.

GC method: start at 100 ^{o}C, hold for 1 min, ramp at 15 ^{o}C/min to 320 ^{o}C, hold 5 min.

PCB (di-chlorinated biphenyl) GC/MS data: 10.5 min; $M^{+} = 222$.

7.15. End-User Report for Asbestos

TITLE: Surface Decontamination of Asbestos by DeconGel 1101

AUTHOR: Andreas Mylonakis PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVES: Determine the surface decontamination efficacy of DeconGel 1101 on linoleum tile, painted drywall and concrete surfaces contaminated with Asbestos fibers (Chrysotile fibers) using Transmission Electron Microscopy (TEM; ASTM standard test method D6480-05) and Polarized Light Microscopy (PLM; EPA approved method for analysis of asbestos from bulk samples).

HAZARDOUS MATERIALS RELEVANCE: Asbestos is a set of six naturally occurring silicate minerals exploited commercially for their desirable physical properties. They all have in common their long and thin fibrous crystals. The inhalation of asbestos fibers can cause serious illnesses, including malignant lung cancer, mesothelioma, and asbestosis.

HIGHLIGHTS

- Excellent surface decontamination was achieved by applying DeconGel 1101 onto surfaces contaminated with Asbestos. Decontamination efficacies (wt% based on the residual asbestos fibers on the surface of interest) were 99.9+% from painted drywall, linoleum tile and concrete surfaces.

- Both semi-qualitative (tape lift adhesion sampling method followed by EPA approved PLM analysis) and semi-quantitative (ASTM standard test swipe sampling method followed by TEM analysis) methods have been utilized in these evaluations.

- Application of asbestos contamination on the respective substrate and sampling tests were performed in CBI Polymers labs; Analysis of the samples were performed at independent labs (EMSL Analytical, Centennial, CO, and Bureau Veritas North America, Inc., Kennesaw, GA)

RESULTS

Tables 1 and 2 show the decontamination efficacies of DeconGel 1101 against asbestos on painted drywall and linoleum tile as determined by the tape lift adhesion test method and EPA approved PLM analysis method and on linoleum tile and concrete as determined by ASTM 6480-05 swipe sampling test method and TEM analysis respectively

Table 1. Decontamination efficacies of DeconGel 1101 against Asbestos contaminated painted drywall and linoleum tile as determined by the tape lift adhesion sampling and PLM analysis method.

Tape Lift Sampling Testing (10 cm^2 sample area)		Formulation
		DeconGel 1101
Linoleum tile	Before Decon	Trace amounts of chrysotile Asbestos*
	After Decon	None Detected**
	Decon. Efficacy (%)	**100%**
Painted drywall	Before Decon	Trace amounts of chrysotile Asbestos*
	After Decon	None Detected**
	Decon. Efficacy (%)	**100%**

Surface area sampled: 10 cm^2

* Samples for which asbestos is detected under the Polarized Light Microscope at <1%are reported as trace.

** "None Detected" indicates that no asbestos fibers were observed under the Polarized Light Microscope

Table 2. Decontamination efficacies of DeconGel 1101 against Asbestos contaminated linoleum tile and concrete surfaces as determined by ASTM pre-wetted wipe sampling and TEM analysis method.

Swipe Testing Method (100 cm^2 sample area) CONCENTRATION (structures/cm²)		Formulation
		DeconGel 1101
Linoleum tile*	Before Decon	117000000
	After Decon	44900
	Decon. Efficacy (%)	**99.97%**
Concrete	Before Decon	2670000
	After Decon	None Detected (<1970)
	Decon. Efficacy (%)	**100%**

Surface area sampled: 100 cm^2

* High loading of asbestos fibers was used for these evaluations.

NOTES

- Chrysotile, which is the most prevalent type of asbestos, was used in these evaluation studies. Chrysotile is a group of fibrous minerals of the serpentine group that have the nominal composition $Mg_3Si_2O_5(OH)_4$ and have the crystal structure of either clinochrysotile, orthochrysotile, or parachrysotile. Most natural chrysotile deviates little from this nominal composition. Chrysotile may be partially dehydrated or magnesium-leached both in nature and in building materials. In some varieties of chrysotile, minor substitution of silicon by aluminum may occur.

- A method that mimics a scaled-down yet real-world setting where decontamination of facilities contaminated with asbestos takes place after the wetting of the substrate to be decontaminated was followed. During these evaluations the procedure followed includes contamination of the substrate of interest with a solution containing asbestos fibers (chrysotile) dispersed in water followed by partial evaporation of the excess water and the application of DeconGel 1101 on top of the wet asbestos contamination.

- ASTM method D 6480-05 is a standardized procedure used to sample and analyze asbestos fibers where pre-wetted wipes are utilized to sample asbestos from surfaces. This method provides an estimate of the concentration of asbestos reported as the number of asbestos structures per unit area of sampled surface.

- Pre-wetted wipers (Model: TX1084 QuanSat with Vectra Quantex from ITW Texwipe, NJ) wetted with 70% isopropanol and 30% deionized water (sealed-border at the edges) were used for the swipe sample tests.

- Tape lift sampling method is a semi-qualitative quick and reliable sampling test method for analysis and monitoring of asbestos contamination. Analysis of the amount of asbestos that has been sampled with this type of sampling method is performed with polarized light microscopy which is an EPA approved method for analysis of asbestos structures in bulk samples (EPA/600/R-93/116).

- Clear (transparent) 3M Scotch tape type was utilized for the tape lift adhesion sampling method. Before and after decon tape lift samples were sent to an independent lab for analysis of the number of asbestos structures per unit area by polarized light microscopy (PLM).

CALCULATIONS

Decontamination Efficacy (Swipe Testing Method) =

[(Concentration (structures/cm^2) of Swipe Control) – (Concentration (structures/cm^2) of Residual Swipe)/ Concentration (structures/cm^2) of Swipe Control] x 100%

MATERIALS AND METHODS

Asbestos solution

Asbestos fibers (Chrysotile, SPI Supplies / Structure Probe, Inc, West Chester, PA) were dispersed in Deionized Water (DI, ≥17MOhm) utilizing a mixer. 0.0971 g of Asbestos fibers were dispersed in 89.3 g of DI water (0.1 wt%).

Application of Asbestos solution on the substrates

Dispersed asbestos fibers in water were applied on top of the surface of interest on a predetermined spot (10cm^2 surface area for the tape lift sample tests; 100cm^2 surface area for the swipe sample tests) and were let to dry up to semi-wet (dampened state) before DeconGel 1101 was applied.

Polarized Light Microscopy (PLM, Analytical Method for Asbestos in Bulk Samples)

Use of EPA/600/R-93/116 satisfies applicable requirements of the USEPA's "Interim Method for the Determination of Asbestos in Bulk Insulation Sample", EPA-600/M4-82-020, December 1982, published as Appendix E to Subpart E of 40CFR763. Bulk samples analyzed by New York State methods follow stratified point counting methods (198.1) or Method 198.6 for PLM non-friable organically bound materials (NYSDOH Lab Code 11645). Percentages are visual estimations of asbestos >10:1 aspect ratio. The reliable limit of quantification of the method is 1%, although asbestos may be qualitatively detected at concentrations less than 1%. Samples for which asbestos is detected at <1% are reported as trace, "<1%". "None Detected" indicates that no asbestos fibers were observed.

SAMPLE AND ANALYSIS METHODS

Tape lift adhesion sampling followed by PLM analysis (semi qualitative test)

1g of asbestos solution (0.1wt% of asbestos in DI H_2O) was applied on top of the linoleum tiles (10cm^2 surface area sampled). 1g of asbestos solution (0.1wt% of asbestos in DI H_2O) was applied on top of painted drywall panels (10cm^2 surface area for the swipe sample tests). Linoleum tile panels and painted drywall were sampled before and after decon with Scotch tape. Scotch tape samples were put into tightly sealed pre-labeled plastic containers (free of dust) and sent to Bureau Veritas North America, Inc. Labs (Kennesaw, GA) for analysis by PLM according to an EPA approved method for analysis of asbestos in bulk samples (EPA/600/R-93/116 satisfies applicable requirements of the USEPA's "Interim Method for the Determination of Asbestos in Bulk Insulation Sample", EPA-600/M4-82-020, December 1982, published as Appendix E to Subpart E of 40CFR763).

Swipe sampling with prewetted wipes (ASTM standard method) followed by TEM analysis (semi quantitative test)

5.52g of asbestos solution (0.1wt% of asbestos in DI H_2O) was applied on top of the concrete panels (100cm^2 surface area for the swipe sample tests). 10g of asbestos solution (0.1wt% of asbestos in DI H_2O) was applied on top of the linoleum tile panels (100cm^2 surface area for the swipe sample tests). Concrete and linoleum tile panels were sampled before and after decon with a pre-wetted wipe (containing 70% Isopropanol/30% Water) according to ASTM 6480-05.

Sample wipes were put into tightly sealed pre-labeled plastic containers (free of dust) and sent to EMSL labs for analysis by TEM according to the ASTM standard test method 6480-05 “Standard Test Method for Wipe Sampling of Surfaces, Indirect Preparation, and Analysis for Asbestos Structure Number Concentration by Transmission Electron Microscopy”.

Analytical Instrumentation

Polarized Light Microscopy (PLM, work performed at Bureau Veritas North America, Inc., Kennesaw, GA 30144) was used as the method of analysis of the tape lift adhesion samples (EPA approved method for analysis of asbestos in bulk samples).

Transmission Electron Microscopy (TEM, EMSL Analytical, Centennial, CO) was used as the method of analysis of the swipe samples (ASTM standard test method for sampling and analysis of asbestos contamination on a surface).

APPLICATION INSTRUCTIONS FOR END-USERS

Pre-wet the surface suspected to be contaminated with asbestos. Use product directly as is from container. DO NOT DILUTE. Masking or painters tape can be applied along one edge of the area that is to be decontaminated to aid creating a peeled edge to grip for peeling the dried film. Apply DeconGel using a paint brush, a trowel, a handheld sprayer, or an industrial grade sprayer (use DeconGel 1120 or 1121 for spray application).

The thickness of the gel and the number of coats is dictated by the surface to be decontaminated. Coating thickness required for good peel characteristics varies with substrate and generally increases with substrate porosity. It is recommended that first time customers test DeconGel on a small sample area to confirm the required film thickness and dry time for their specific application.

If the film is difficult to peel, please apply an additional coat. A razor blade is useful to start the peel. Lay the blade nearly flat and fillet the edge of the film to create a tab that can be pulled. For surfaces that the gel adheres to well, such as concrete, 12” – 24” strips can be cut in the film resulting in less force being required to peel the film.

- Let DeconGel dry for 24 hours

Dry time will vary depending on humidity, temperature, air flow and thickness of the DeconGel. This can take from as little time as an hour for thin coats in a dry environment with plenty of airflow, to overnight or longer if thicker coats are applied in humid environments. Dry times exceeding 24 hours may sometimes be required for good peel performance on bare concrete, wood and other highly porous substrates and substrates with deep cracks or grooves. However, 18-24 hrs is often sufficient dry time on good quality concrete. It is recommended that users test a small area to determine drying time prior to applying DeconGel for an entire job. Supplemental heat or air circulation will accelerate DeconGel’s drying time for any job.

- Peel DeconGel off the surface by starting from one of the edges

When dry, the product locks the contaminants into a polymer matrix. The film containing the encapsulated contamination can then be peeled. DeconGel peels from most non-porous and porous hard surfaces if the dried film is thick enough. If the film is difficult to peel, add another coat, let dry, and peel. In most cases the DeconGel will come off in a single sheet but for odd shaped surfaces you may be required to score DeconGel in order to be able to peel it off.

- Dispose of the dried DeconGel in accordance with the local, state and Federal disposal regulations of the contaminant/substance you are removing. DeconGel itself has no special disposal restrictions.

For more information on application techniques visit our website:
www.decongel.com

7.16. End-User Report for Chromium Compound (Potassium Chromate)

TITLE: Surface Decontamination of Chromium Compounds (Potassium Chromate (K_2CrO_4) by DeconGel 1101

ANALYSTS: Geoff Nadolski PhD, Senior Scientist and Andreas Mylonakis PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel formulation 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with Chromium (K_2CrO_4) using ICP-OES (Inductively Coupled Plasma-Optical Emission Spectroscopy) following Environmental Protection Agency (EPA) SW-846 Methods 3005A (sampling) and 6010C (analysis).

HAZARDOUS MATERIALS RELEVANCE: Potassium chromate is a strong oxidizing agent and is used as a chemical indicator for chloride ion content. Potassium chromate is very toxic and may be fatal if swallowed, and can cause cancer on inhalation. Potassium chromate was chosen as a representative chromium compound for evaluating DeconGel's efficacy; DeconGel is expected to have similar efficacy towards the wide range of chromium compounds.

SUMMARY RESULTS:

- As seen in Table 1, excellent to acceptable surface decontamination was achieved by applying DeconGel 1101 onto contaminated surfaces, resulting in encapsulation of Chromium contaminant by DeconGel's active components. Decontamination efficacies of poured DeconGel 1101 ranged from 91.9% (on concrete) to 99.2% (on carbon steel) to 99.6% (on stainless steel) as determined by residual swipe analysis.

- Potassium chromate was evidenced to react with concete surfaces, forming a fixed residue that was not able to be completely removed by DeconGel. Nevertheless, DeconGel showed acceptable decontamination efficacy of loose potassium chromate contamination from such surfaces.

- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of inorganic compounds in aqueous samples. When deemed necessary, experimental methods were customized to afford complete dissolution of inorganic contaminants, and to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Table 1 shows the decontamination efficacies of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with chromium compound (K_2CrO_4) as determined by residual swipe testing.

Table 1. Decontamination efficacies of DeconGel 1101 on Potassium Chromate contaminated stainless steel, carbon steel, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	140.5 ± 7.9
	Residual	0.58 ± 0.16
	Decon. Efficacy (%)	**99.6 ± 5.9**
Carbon Steel	Control	144.8 ± 10.4
	Residual	1.23 ± 0.24
	Decon. Efficacy (%)	**99.2 ± 9.6**
Concrete*	Control	144.0 ± 21.3
	Residual	11.7 ± 1.3
	Decon. Efficacy (%)	**91.9 ± 3.5**

2000x dilution factor for samples and controls

* Potassium chromate reacted with concrete surface, resulting in a fixed residue on the contaminated surface that could not be completely removed by DeconGel.

NOTES:

- Application of a homogenous, thin layer of Chromium contaminant on the respective substrate facilitated an optimized interaction between contaminant and DeconGel, and provided an accurate measure of DeconGel's decontamination efficacy in a scaled-down yet real-world setting. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel. Potassium chromate readily dissolves in aqueous acidic solutions used to prepare all samples and controls (15% HCl, 15% HNO_3, 70% DI H_2O).

- Potassium chromate undergoes a chemical reaction with concrete surface which prevents some contamination from being sampled (swipe testing). Nevertheless DeconGel showed acceptable decontamination efficacy against loose potassium chromate contamination.

- ASTM method E1728-03, a standardized swipe testing method used for sampling of inorganic contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes (pre-wetted with DI H_2O) were utilized in this swipe testing method.

- Standardized EPA SW-846 Sampling Method 3005A "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy" was followed as a guideline to prepare all samples and controls. When deemed necessary, digestion methods were customized by increasing hydrochloric and nitric acid concentrations from 15% to 25% wt, and/or by heating samples to higher temperatures using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC) to afford the complete digestion of the inorganic contaminants. All samples, controls, and standards were prepared using the same dissolution solution and experimental conditions to ensure both correct instrument calibration and accurate analytical results.

- ICP-OES instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of inorganic compounds. Standardized EPA SW-846 Analytical Method 6010C "Inductively Coupled Plasma-Atomic Emission Spectrometry" was followed as a guideline to prepare all samples and controls.

- To ensure accurate determination of DeconGel decontamination efficacy, calibration standards of the analyte of interest were prepared using either a sufficiently pure analyte or an appropriate ICP-MS Standard (Ricca Chemical Company; Arlington, TX); the respective standards were diluted to a known concentration (ppm) using the same digestion method as the one used for samples and controls. Instrument blank controls were DI H_2O ($\geq$17 M-Ohm).

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.050 g potassium chromate contaminant was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) carbon steel (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe and gel samples were suspended in 100 mL aqueous acidic solution (15% HCl, 15% HNO_3, 70% deionized (DI) H_2O) for 24 h. When deemed necessary, samples were heated to 94°C for 4-24 h to effectively complete dissolution of inorganics using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC). Samples were allowed to cool to room temperature and were then analyzed via ICP-OES (see below).

Controls Methods

For Swipe Controls samples, a respectable amount of contaminant was evenly applied on 1) stainless steel (56.3 cm^2), 2) carbon steel (commercial grade, 100 cm^2), or 3) concrete (industrial grade, 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 100 mL aqueous acidic solution (15% HCl, 15% HNO_3, 70% DI H_2O) for 24 h and analyzed via ICP-OES (see below).

Reagents and Standards

Reagent grade Potassium Chromate, K_2CrO_4, (CAS# 7789-00-6, Fisher Scientific; Fair Lawn, NJ) was used as received.

1000 ppm calibration standards were prepared using reagent grade K_2CrO_4 in freshly prepared aqueous acidic solution (15% HCl, 15% HNO_3, 70% DI H_2O). DI H_2O was used as the blank sample.

Analytical Instrumentation

A Thermo ICP-OES instrument model radial iCap 6300 was used to determine chromium compound concentration (ppm) of all samples and controls using a freshly prepared 1000 ppm calibration standard.

Analyte (chromium) analyzed at 283.5 nm

Pump Speed: 0.5 mL/min

APPLICATION INSTRUCTIONS FOR END-USERS

Use product directly as is from container. DO NOT DILUTE. Masking or painters tape can be applied along one edge of the area that is to be decontaminated to aid creating a peeled edge to grip for peeling the dried film. Apply DeconGel using a paint brush, a trowel, a handheld sprayer, or an industrial grade sprayer (use DeconGel 1120 or 1121 for spray application).

The thickness of the gel and the number of coats is dictated by the surface to be decontaminated. Coating thickness required for good peel characteristics varies with substrate and generally increases with substrate porosity. It is recommended that first time customers test DeconGel on a small sample area to confirm the required film thickness and dry time for their specific application. If the film is difficult to peel, please apply an additional coat. A razor blade is useful to start the peel. Lay the blade nearly flat and fillet the edge of the film to create a tab that can be pulled. For surfaces that the gel adheres to well, such as concrete, 12" – 24" strips can be cut in the film resulting in less force being required to peel the film.

- Let DeconGel dry for 24 hours

Dry time will vary depending on humidity, temperature, air flow and thickness of the DeconGel. This can take from as little time as an hour for thin coats in a dry environment with plenty of airflow, to overnight or longer if thicker coats are applied in humid environments. Dry times exceeding 24 hours may sometimes be required for good peel performance on bare concrete, wood and other highly porous substrates and substrates with deep cracks or grooves. However, 18-24 hrs is often sufficient dry time on good quality concrete. It is recommended that users test a small area to determine drying time prior to applying DeconGel for an entire job. Supplemental heat or air circulation will accelerate DeconGel's drying time for any job.

- Peel DeconGel off the surface by starting from one of the edges

When dry, the product locks the contaminants into a polymer matrix. The film containing the encapsulated contamination can then be peeled. DeconGel peels from most non-porous and porous hard surfaces if the dried film is thick enough. If the film is difficult to peel, add another coat, let dry, and peel. In most cases the DeconGel will come off in a single sheet but for odd shaped surfaces you may be required to score DeconGel in order to be able to peel it off.

- Dispose of the dried DeconGel in accordance with the local, state and Federal disposal regulations of the contaminant/substance you are removing. DeconGel itself has no special disposal restrictions.

For more information on application techniques visit our website: www.decongel.com

7.17. End-User Report for Crude Oil

TITLE: Surface Decontamination of Crude Oil by DeconGel 1101 and 1102

ANALYST: Geoff Nadolski PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVES: Surface decontamination determination of DeconGel formulation 1101 and 1102 on aluminum, stainless steel, and concrete surfaces contaminated with Crude Oil (polyaromatic hydrocarbons fraction) using LC/MS (Liquid Chromatography/Mass Spectrometry) following Environmental Protection Agency (EPA) SW-846 Methods 3500C (Sampling), 8321B (Analysis), and 1654A (Analysis of PAH Content of Oil).

HAZARDOUS MATERIALS RELEVANCE: Crude oil is used throughout the world as an energy source, and is used to provide a broad range of valuable and useful materials. Crude oil and its decomposition products are environmentally persistent toxins, and when released into the environment require extensive remediation typically resulting in the generation of large quantities of toxic contamination. Crude oil is comprised of 0.2-7% polyaromatic hydrocarbons (PAHs), widespread organic pollutants known for their carcinogenic, mutagenic, and tetratogenic properties.

SUMMARY RESULTS:

- As seen in Table 1, excellent surface decontamination was achieved by applying DeconGel 1101 and 1102 both via brushing or pouring (non-brushed) onto contaminated surfaces, resulting in encapsulation of Crude oil contaminant by DeconGel's active components. Decontamination efficacies of poured DeconGel 1101 ranged from 85.9% (on concrete) to 93.9% (on aluminum) to 93.9% (on stainless steel), brushed DeconGel 1101 ranged from 98.4% (on concrete) to 99.0% (on stainless steel) to 99.1% (on aluminum), as determined by residual swipe analysis. Decontamination efficacies of poured DeconGel 1102 ranged from 86.6% (on concrete) to 91.3% (on stainless steel) to 94.1% (on aluminum), brushed DeconGel 1102 ranged from 98.9% (on concrete) to 99.5% (on stainless steel) to 99.6% (on aluminum), as determined by residual swipe analysis.
- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of organics in polar solvent solvated samples. When necessary, the digestion methods were customized to result in the complete dissolution of the organic contaminants and to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Table 1 shows the decontamination efficacies of DeconGel 1101 on stainless steel, aluminum, and concrete surfaces contaminated with Crude oil as determined by residual swipe testing.

Table 1. Decontamination efficacies of DeconGel 1101 and 1102 on Crude Oil contaminated stainless steel, aluminum, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation	Formulation
		DeconGel 1101	**DeconGel 1102**
Stainless Steel	Control	213.63 ± 1.60	213.63 ± 1.60
	Residual (non-brushed)	12.88 ± 0.29	18.63 ± 0.33
	Residual (brushed)	2.04 ± 0.02	0.97 ± 0.03
	Decon. Efficacy (non-brushed) (%)	**93.9 ± 0.10**	**91.3 ± 0.16**
	Decon. Efficacy (brushed) (%)	**99.0 ± 0.10**	**99.5 ± 0.10**
Aluminum	Control	215.82 ± 1.07	215.82 ± 1.07
	Residual (non-brushed)	13.16 ± 0.66	19.10 ± 0.41
	Residual (brushed)	2.02 ± 0.01	0.93 ± 0.02
	Decon. Efficacy (non-brushed) (%)	**93.9 ± 0.10**	**94.1 ± 0.34**
	Decon. Efficacy (brushed) (%)	**99.1 ± 0.10**	**99.6 ± 0.10**
Concrete	Control	156.03 ± 1.93	156.03 ± 1.93
	Residual (non-brushed)	22.02 ± 0.26	20.88 ± 0.12
	Residual (brushed)	2.44 ± 0.10	1.79 ± 0.02
	Decon. Efficacy (non-brushed) (%)	**85.9 ± 0.18**	**86.6 ± 0.36**
	Decon. Efficacy (brushed) (%)	**98.4 ± 0.10**	**98.9 ± 0.10**

13133x dilution factor for samples and controls

NOTES:

- Application of a homogenous, thin layer of Crude oil contaminant on the respective substrate facilitated an optimized interaction between contaminant and DeconGel, and provided an accurate measure of DeconGel’s decontamination efficacy in a scaled-down yet real-world setting. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel.

- ASTM method E1728-03, a standardized swipe testing method used for sampling of contaminants was the integral method used to accurately evaluate DeconGel’s decontamination efficacy. Air-dried GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes wetted with methanol/DMSO (1:1) solvent were utilized in this swipe testing method.

- Standardized EPA SW-846 Sampling Method 3500C “Organic Extraction and Sample Preparation” and EPA Method 1654A “PAH Content of Oil by HPLC/UV) were followed as a guideline to prepare all samples and controls. All samples, controls, and standards were prepared using the same solvent and appropriate experimental conditions to ensure accurate and LC/MS instrumental analysis.
- LC/MS instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of organic compounds. Standardized EPA SW-846 Analytical Method 8321B “Solvent-Extractable Nonvolatile Compounds by High-Performance Liquid Chromatography/Thermospray/Mass Spectrometry (HPLC/TS/MS) or Ultraviolet (UV) Detection” was followed as a guideline to prepare all samples and controls.
- To ensure accurate determination of DeconGel decontamination efficacy, a standard curve of the analyte of interest was prepared using sufficiently pure analyte; the respective standards were diluted to a known concentration (ppm) using the same solvent as used for samples and controls.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.10 g (1.0 mL of 5 g/50 mL hexane) Crude oil contaminant was evenly applied via brushing on 1) aluminum (surface area: 56.3 cm^2), 2) stainless steel (surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and then the hexane carrier solvent allowed to evaporate for 20 min. Approximately 6.0 g of DeconGel 1101 or 1102 was either poured or brushed (brushed first in a top-bottom, then in a left-right fashion) onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC) wetted with methanol:DMSO (1:1) solvent (2 mL). Swipe and dried film samples were suspended in 50 mL methanol:DMSO (1:1) for 24 h. Samples were then analyzed via LC/MS (see below).

Control Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied via brushing on 1) aluminum (surface area: 56.3 cm^2), 2) stainless steel (surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC) wetted with methanol:DMSO (1:1) solvent (2 mL). Swipe and dried film samples were suspended in 50 mL methanol:DMSO (1:1) for 24 h. Samples were then analyzed via LC/MS (see below).

Reagents and Standards

Crude oil (light, sweet Chevron crude oil sourced Oahu, Hawaii) was suspended in methanol/DMSO (1:1) solvent mixture, sonicated for 10 min, let to stand for 24 h, and then the yellow liquid decanted from undissolved solids used to generate standard curves.

Analytical Instrumentation

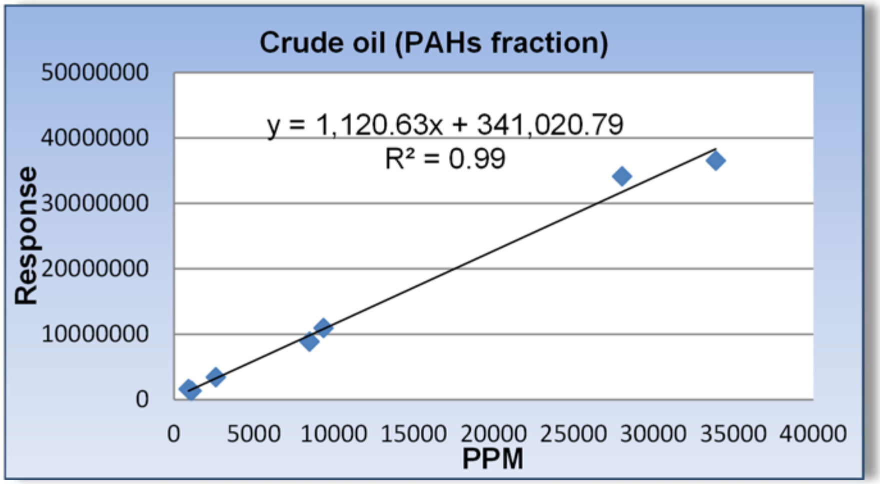
A Thermo LCQ LC/MS with autosampler in positive mode was used to determine PAHs (polyaromatic hydrocarbons) fraction concentration (ppm, wt/wt) of all samples and controls, using a Grace Davison (Deerfield, IL) C18-Select analytical column (150 x 4.6 mm, 5 um).

A 7-point standard curve derived from three independently prepared stock solutions (crude oil dissolved in methanol:DMSO (1:1) was prepared. The calibration curve exhibits a curve fitting as approximated by the coefficient of determination of linear regression R^2, where $R^2 = 0.99$ (see Figure 1).

LC method using A= water (0.1% formic acid), B=acetonitrile (0.1% formic acid); start at 90%A to 2 min at 70%A, to 3 min at 50%A, to 5 min at 30%A, to 6 min at 100%B, hold until 16 min at 90% A hold until 17 min.

Crude oil (PAHs fraction) LC/MS data: 9.2 min; lambda max = 275 nm; no data acquired for MS.

Figure 1. Crude oil (PAHs fraction) Standard Calibration Curve



APPLICATION INSTRUCTIONS FOR END-USERS

Use product directly as is from container. DO NOT DILUTE. Masking or painters tape can be applied along one edge of the area that is to be decontaminated to aid creating a peeled edge to grip for peeling the dried film. Apply DeconGel using a paint brush, a trowel, a handheld sprayer, or an industrial grade sprayer (use DeconGel 1120 or 1121 for spray application).

The thickness of the gel and the number of coats is dictated by the surface to be decontaminated. Coating thickness required for good peel characteristics varies with substrate and generally increases with substrate porosity. It is recommended that first time customers test DeconGel on a small sample area to

confirm the required film thickness and dry time for their specific application. If the film is difficult to peel, please apply an additional coat. A razor blade is useful to start the peel. Lay the blade nearly flat and fillet the edge of the film to create a tab that can be pulled. For surfaces that the gel adheres to well, such as concrete, 12” – 24” strips can be cut in the film resulting in less force being required to peel the film.

- Let DeconGel dry for 24 hours

Dry time will vary depending on humidity, temperature, air flow and thickness of the DeconGel. This can take from as little time as an hour for thin coats in a dry environment with plenty of airflow, to overnight or longer if thicker coats are applied in humid environments. Dry times exceeding 24 hours may sometimes be required for good peel performance on bare concrete, wood and other highly porous substrates and substrates with deep cracks or grooves. However, 18-24 hrs is often sufficient dry time on good quality concrete. It is recommended that users test a small area to determine drying time prior to applying DeconGel for an entire job. Supplemental heat or air circulation will accelerate DeconGel’s drying time for any job.

- Peel DeconGel off the surface by starting from one of the edges

When dry, the product locks the contaminants into a polymer matrix. The film containing the encapsulated contamination can then be peeled. DeconGel peels from most non-porous and porous hard surfaces if the dried film is thick enough. If the film is difficult to peel, add another coat, let dry, and peel. In most cases the DeconGel will come off in a single sheet but for odd shaped surfaces you may be required to score DeconGel in order to be able to peel it off.

- Dispose of the dried DeconGel in accordance with the local, state and Federal disposal regulations of the contaminant/substance you are removing. DeconGel itself has no special disposal restrictions.

For more information on application techniques visit our website:
www.decongel.com

7.18. End-User Report for Copper Compounds (Elemental Copper, Copper (I) Oxide)

TITLE: Surface Decontamination of Copper Compounds (Copper (elemental), Copper (I) Oxide) by DeconGel 1101

ANALYSTS: Geoff Nadolski PhD, Senior Scientist and Andreas Mylonakis PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel 1101 on stainless steel, carbon steel, concrete, Lexan, and rubber surfaces contaminated with elemental copper and copper (I) oxide (Cu_2O) using ICP-OES (Inductively Coupled Plasma-Optical Emission Spectroscopy) following Environmental Protection Agency (EPA) SW-846 Methods 3005A (sampling) and 6010C (analysis).

HAZARDOUS MATERIALS RELEVANCE: Copper and its compounds such as copper (I) oxide are commonly used as components of semiconductors, pigments, fungicides, and as antifouling agents for marine paints. Due to its toxicity toward aquatic organisms, copper is considered an environmentally persistent pollutant. Elemental copper and copper (I) oxide were chosen as a representative copper compounds for evaluating DeconGel's efficacy; DeconGel is expected to have similar efficacy towards the wide range of copper compounds.

SUMMARY RESULTS:

- As seen in Tables 1 and 2, excellent surface decontamination was achieved by applying DeconGel 1101 onto contaminated surfaces, resulting in encapsulation of Copper contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 on copper elemental ranged from 98.0% (on concrete) to 99.4% (on rubber) to 99.5% (on Lexan) to 99.5% (on carbon steel) to 99.9% (on stainless steel) as determined by residual swipe analysis; decontamination efficacies of DeconGel 1101 on copper (I) oxide ranged from 94.7% (on concrete) to 98.9% (on carbon steel) to 99.7% (on stainless steel) as determined by residual swipe analysis.
- Copper (I) oxide was evidenced to react with concete surfaces, forming a fixed residue that was not able to be completely removed by DeconGel. Nevertheless, DeconGel showed acceptable decontamination efficacy of loose copper (I) oxide contamination from such surfaces.
- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of inorganic compounds in aqueous samples. When deemed

necessary, experimental methods were customized to afford complete dissolution of inorganic contaminants, and to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 and 2 show the decontamination efficacies of DeconGel 1101 on stainless steel, carbon steel, and concrete surfaces contaminated with copper compound (Cu_2O) as determined by residual swipe testing.

Table 1. Decontamination efficacies of DeconGel 1101 on Copper (elemental) contaminated stainless steel, carbon steel, concrete, rubber, and Lexan surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel	Control	396.3 ± 16.3
	Residual	0.56 ± 0.59
	Decon. Efficacy (%)	**99.9 ± 8.9**
Carbon Steel	Control	396.1 ± 10.1
	Residual	1.81 ± 0.81
	Decon. Efficacy (%)	**99.5 ± 7.0**
Concrete	Control	352.0 ± 8.2
	Residual	7.0 ± 4.0
	Decon. Efficacy (%)	**98.0 ± 2.9**
Rubber*	Control	61.0 ± 6.0
	Residual	0.34 ± 0.40
	Decon. Efficacy (%)	**99.4 ± 9.9**
Lexan*	Control	59.7 ± 1.2
	Residual	0.28 ± 0.34
	Decon. Efficacy (%)	**99.5 ± 4.9**

2000x dilution factor for samples and controls
*12000x dilution factor for samples and controls

Table 2. Decontamination efficacies of DeconGel 1101 on Copper (I) Oxide contaminated stainless steel, carbon steel, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation DeconGel 1101
Stainless Steel	Control	526.5 ± 41.9
	Residual	1.50 ± 0.40
	Decon. Efficacy (%)	**99.7 ± 8.7**
Carbon Steel	Control	499.7 ± 33.9
	Residual	5.52 ± 3.53
	Decon. Efficacy (%)	**98.9 ± 6.9**
Concrete*,[1]	Control	55.8 ± 1.4
	Residual	2.95 ± 1.67
	Decon. Efficacy (%)	**94.7 ± 3.9**

2000x dilution factor for samples and controls

[1] 12000x dilution factor for samples

* Copper (I) oxide reacted with concrete surface, resulting in a fixed residue on the contaminated surface that could not be completely removed by DeconGel.

NOTES:

- Application of a homogenous, thin layer of Copper contaminant on the respective substrate facilitated an optimized interaction between contaminant and DeconGel, and provided an accurate measure of DeconGel's decontamination efficacy in a scaled-down yet real-world setting. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel. Copper (elemental) and copper (I) oxide readily dissolve in aqueous acidic solutions used to prepare all samples and controls (20% HCl, 15% HNO_3, 65% DI H_2O).

- Copper (I) oxide undergoes a chemical reaction with concrete surface which prevents some contamination from being sampled (swipe testing) and encapsulated into dry DeconGel (direct gel testing). Nevertheless DeconGel showed acceptable decontamination efficacy against loose copper (I) oxide contamination.

- ASTM method E1728-03, a standardized swipe testing method used for sampling of inorganic contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes (pre-wetted with DI H_2O) were utilized in this swipe testing method.

- Standardized EPA SW-846 Sampling Method 3005A "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy" was followed as a guideline to prepare all samples and controls. When deemed necessary, digestion methods were customized by increasing hydrochloric and nitric acid concentrations from 15% to 25% wt, and/or by heating samples to higher temperatures using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC) to afford the complete digestion of the inorganic contaminants. All samples, controls, and standards were prepared using the same dissolution solution and experimental conditions to ensure both correct instrument calibration and accurate analytical results.

- ICP-OES instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of inorganic compounds. Standardized EPA SW-846 Analytical Method 6010C "Inductively Coupled Plasma-Atomic Emission Spectrometry" was followed as a guideline to prepare all samples and controls.

- To ensure accurate determination of DeconGel decontamination efficacy, calibration standards of the analyte of interest were prepared using either a sufficiently pure analyte or an appropriate ICP-MS Standard (Ricca Chemical Company; Arlington, TX); the respective standards were diluted to a known concentration (ppm) using the same digestion method as the one used for samples and controls. Instrument blank controls were DI H_2O ($\geq$17 M-Ohm).

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.050 g copper contaminant was evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) carbon steel (surface area: 100 cm^2), 3) concrete (industrial grade, surface area: 56.3 cm^2), 4) rubber (surface area: 56.3 cm^2), or 5) Lexan (surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe and gel samples were suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% deionized (DI) H_2O) for 24 h. When deemed necessary, samples were heated to 94°C for 4-24 h to effectively complete dissolution of inorganics using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC).

Samples were allowed to cool to room temperature and were then analyzed via ICP-OES (see below).

Controls Methods

For Swipe Controls samples, a respectable amount of contaminant was evenly applied on 1) stainless steel (56.3 cm^2), 2) carbon steel (commercial grade, 100 cm^2), or 3) concrete (industrial grade, 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 100 mL aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O) for 24 h and analyzed via ICP-OES (see below).

Reagents and Standards

Reagent grade Copper (elemental), (CAS# 7440-50-8, Fisher Scientific; Fair Lawn, NJ) was used as received.

Reagent grade Copper (I) Oxide, Cu_2O, (CAS# 1317-39-1, Fisher Scientific; Fair Lawn, NJ) was used as received.

1000 ppm calibration standards were prepared using reagent grade copper (elemental) and Cu_2O in freshly prepared aqueous acidic solution (20% HCl, 15% HNO_3, 65% DI H_2O). DI H_2O was used as the blank sample.

Analytical Instrumentation

A Thermo ICP-OES instrument model radial iCap 6300 was used to determine copper compound concentration (ppm) of all samples and controls using a freshly prepared 1000 ppm calibration standard.

Analyte (copper) analyzed at 327.4 nm

Pump Speed: 0.5 mL/min

APPLICATION INSTRUCTIONS FOR END-USERS

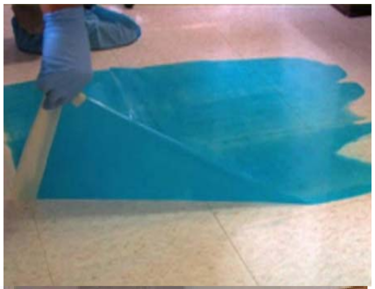
Use product directly as is from container. DO NOT DILUTE. Masking or painters tape can be applied along one edge of the area that is to be decontaminated to aid creating a peeled edge to grip for peeling the dried film. Apply DeconGel using a paint brush, a trowel, a handheld sprayer, or an industrial grade sprayer (use DeconGel 1120 or 1121 for spray application).

The thickness of the gel and the number of coats is dictated by the surface to be decontaminated. Coating thickness required for good peel characteristics varies with substrate and generally increases with substrate porosity. It is recommended that first time customers test DeconGel on a small sample area to confirm the required film thickness and dry time for their specific application. If the film is difficult to peel, please apply an additional coat. A razor blade is useful to start the peel. Lay the blade nearly flat and fillet the edge of the film to create a tab that can be pulled. For surfaces that the gel adheres to well, such as concrete, 12” – 24” strips can be cut in the film resulting in less force being required to peel the film.

- Let DeconGel dry for 24 hours

Dry time will vary depending on humidity, temperature, air flow and thickness of the DeconGel. This can take from as little time as an hour for thin coats in a dry environment with plenty of airflow, to overnight or longer if thicker coats are applied in humid environments. Dry times exceeding 24 hours may sometimes be required for good peel performance on bare concrete, wood and other highly porous substrates and substrates with deep cracks or grooves. However, 18-24 hrs is often sufficient dry time on good quality concrete. It is recommended that users test a small area to determine drying time prior to applying DeconGel for an entire job. Supplemental heat or air circulation will accelerate DeconGel’s drying time for any job.

- Peel DeconGel off the surface by starting from one of the edges



When dry, the product locks the contaminants into a polymer matrix. The film containing the encapsulated contamination can then be peeled. DeconGel peels from most non-porous and porous hard surfaces if the dried film is thick enough. If the film is difficult to peel, add another coat, let dry, and peel. In most cases the DeconGel will come off in a single sheet but for odd shaped surfaces you may be required to score DeconGel in order to be able to peel it off.

➢ Dispose of the dried DeconGel in accordance with the local, state and Federal disposal regulations of the contaminant/substance you are removing. DeconGel itself has no special disposal restrictions.

For more information on application techniques visit our website: www.decongel.com

7.19. End-User Report for CWA Sulfur Mustard-Simulant (CEES)

TITLE: Surface Decontamination of the Sulfur Mustard Simulant 2-Chloroethyl Ethyl Sulfide (CEES) by DeconGel 1101

ANALYST: Geoff Nadolski PhD, Senior Polymer Scientist, CBI Polymers, Inc.

SUPERVISOR: Garry J. Edgington PhD, Chief Scientist, CBI Polymers, Inc.

OBJECTIVE: Evaluate the decontamination efficacy of DeconGel 1101 on coupon types:

1) Cadmium-plated Steel,

2) Carbon Steel,

3) Chemical agent resistant coating (CARC),

4) Silicone Rubber, and

5) Black Rubber surfaces contaminated with the Sulfur Mustard simulant CEES.

HAZARDOUS MATERIALS RELEVANCE: Sulfur Mustard, bis-(2-chloroethyl) sulfide, is a powerful vesicant, cytotoxic agent regulated under the 1993 Chemical Weapons Convention (CWC). Sulfur mustard can be deployed by spraying, or more typically distributed via artillery shells and bombs. For experimental testing purposes, the half-mustard 2-chloroethyl ethyl sulfide (CEES) is used as an effective yet less toxic simulant for sulfur mustard chemical warfare agents.

SUMMARY RESULTS:

- Because none of the highly volatile contaminant CEES was detected for either samples on the non-porous steel surfaces tested, or Positive Control 1, an adequate measure of surface decontamination could not be calculated for Cd-plated and Carbon Steel coupons.

- On porous and/or chemically active coupon surfaces such as CARC and Black and Silicone Rubbers, modest to excellent Surface Decontamination of volatile CEES was realized; even on delicate or difficult-to-clean customized rubber, epoxy, or polyester surfaces, DeconGel is able to effectively remove hydrophobic contaminants through encapsulation/emulsification.

  - Reduction in starting challenge (Percent Efficacies of DeconGel 1101) ranged from 80.5% (on Silicone Rubber) to 97.0% (on Black Rubber) to 99.9% (on CARC) as determined by solvent extraction.

  - DeconGel 1101 Decontamination Efficacies ranged from 28.6% (on CARC) to 54.6% (on Silicone Rubber) to 93.2% (on Black Rubber) as determined by solvent extraction.

- DeconGel 1101 can serve as a partial chemical (vapor) barrier. The vapor barrier effect demonstrates the reduction in the rate of release of agent to the surrounding atmosphere as well as indicating a prolonged residence-time inside the gel layer. An extended residence-time in the gel is predicted to enhance the efficacy of neutralizing agents that are proposed to be employed in future versions of the gel. This reduction in rate of release of agents is proposed to be especially valuable in enhancing neutralization efficacies of CWAs with high reactivity and volatility.To support the hypothesis that DeconGel 1101 can serve as an partial chemical (vapor) barrier, additional experimentation ((+)-Control 2) was performed to demonstrate the Chemical Barrier Efficiency of DeconGel 1101.

  - On Black and Silicone Rubber surfaces, DeconGel 1101 Chemical Barrier Efficiencies ranged from 84.7% (on Silicone Rubber) to 85.8% (on Black Rubber) as determined by solvent extraction, indicating a significant chemical surface barrier capability for DeconGel 1101 when applied to porous, chemically active rubber/ epoxy/polyester surfaces able to absorb and entrain contaminants;

  - On non-porous, chemically inert Cd- and Carbon Steel surfaces, as well as the porous CARC coupon, due to CEES's significant volatility, contaminant applied to the steel coupons was not detectable after undergoing a 24-hour coupon-contaminant incubation as implemented for positive controls ((+)-Control 2). As such, samples and controls could not be compared, and therefore Chemical Barrier Efficiencies for Cd- and Carbon Steels and CARC surfaces could not be determined.

RESULTS: Tables 1 and 2 contain the Data Test Set, and Decontamination Efficacy, Reduction in Starting Challenge Percent Efficacy, and Chemical Barrier Efficiency, respectively, for the sulfur mustard simulant contaminant CEES decontamination study conducted using DeconGel 1101 as the decontaminant on a number of coupon surfaces including Cadmium-plated and Carbon Steels, CARC, Silicone Rubber and Black Rubbers by solvent extraction.

Table 1. DeconGel decontaminant Data Test Set for CEES decontamination study on multiple surfaces as determined by residue analysis after solvent extraction

Coupon Type		RA_M (ng)	CD (g/m^2)
Cd-plated Steel	(-)-Control[1]	ND[2]	ND
	Remaining Agent	ND	ND
	(+)-Control 1[3]	ND	ND
	(+)-Control 2[4]	ND	ND
	Dose Confirmation	2127253 ± 8925	N/A[5]
Carbon Steel	(-)-Control	ND	ND
	Remaining Agent	ND	ND
	(+)-Control 1	ND	ND
	(+)-Control 2	ND	ND
	Dose Confirmation	2127253 ± 8925	N/A
CARC	(-)-Control	ND	ND
	Remaining Agent	1776.4 ± 0.0	0.0009 ± 0.0
	(+)-Control 1	2490.05 ± 14.06	0.0013 ± 0.0001
	(+)-Control 2	ND	ND
	Dose Confirmation	2127253 ± 8925	N/A
Silicone Rubber	(-)-Control	ND	ND
	Remaining Agent	415762.6 ± 1529.8	0.211 ± 0.001
	(+)-Control 1	915092.6 ± 4302.8	0.466 ± 0.002
	(+)-Control 2	55177.7 ± 216.3	0.028 ± 0.0
	Dose Confirmation	2127253 ± 8925	N/A
Black Rubber	(-)-Control	ND	ND
	Remaining Agent	63515.08 ± 1144.5	0.038 ± 0.001
	(+)-Control 1	930026.6 ± 23048.3	0.553 ± 0.014
	(+)-Control 2	7849.5 ± 86.58	0.005 ± 0.0003
	Dose Confirmation	2127253 ± 8925	N/A

[1] (-)-Control: no contaminant applied, then decontaminant applied

[2] ND: not detected, See Test Specific section, lower limits of detection (LOD) for CEES using GC/MS ≤ 80 ppb

[3] (+)-Control 1: contaminant applied for 1 h, no decontaminant applied

[4] (+)-Control 2: contaminant applied for 24 h, no decontaminant applied

[5] N/A: Not Applicable

RA_M: Remaining Agent (mass); CD: Contamination Density

Table 2. Decontamination Efficacy, Percent Efficacy, and Chemical Barrier Efficiency evaluation for the sulfur mustard simulant CEES decontamination study on multiple surfaces as determined by residue analysis after solvent extraction

Coupon Type	Decontamination Efficacy[1] (%)	Percent Efficacy[2] (%)	Chemical Barrier Efficacy[3] (%)
Cd-plated Steel	ND[4]	ND	ND
Carbon Steel	ND	ND	ND
CARC	28.6 ± 0.0	99.9 ± 0.0	ND
Silicone Rubber	54.6 ± 0.20	80.5 ± 0.11	84.7 ± 0.10
Black Rubber	93.2 ± 0.12	97.0 ± 0.05	85.8 ± 0.27

[1] See Decontamination Efficacy equation (Eqn 1) in Experimental section

[2] See Percent Efficacy equation (Eqn 2) in Experimental section

[3] See Chemical Barrier Efficacy equation (Eqn 3) in Experimental section

[4] ND: not determined (no contaminant detected for samples and/or controls)

NOTES:

- Due to CEES's low boiling point/volatility, significant loss of contaminant CEES was noted when storing contaminated coupons in a chemical hood for the required 60 min contact hold time. Utilizing an inverted Pyrex dessicator greatly improved both volatile surface retention and contaminant-coupon interaction, as evidenced after the 60 min hold time as either a significant surface retention of wetted drops, or as a raised surface.

- In applying a small amount (2.0 uL) of neat CEES contaminant as the starting challenge, there was no need to brush the volatile liquid contaminant throughout coupon surfaces to ensure a thin layer of CEES was loaded onto coupon surfaces; using a minute amount of contaminant facilitated a favorable interaction between DeconGel and CEES without overloading the relatively small amount of applied DeconGel decontaminant (1.0-1.1 g).

- Acetonitrile solvent was used to dissolve CEES contaminant and to generate a standard calibration curve possessing a significant concentration range and favorable accuracy and precision. Acetonitrile was used as the extraction solvent due to its ability to readily solvate CEES, compatibility with GC/MS analysis, relatively low volatility, and low toxicity profile. In accordance with the JPEO guidelines, 20 mL of acetonitrile extraction solvent was effective in generating precise data as evidenced by the low standard deviation of the modestly concentrated Dose Confirmation Samples ((DCS); DCS avg= 2127253 ± 8925 ng)

- Sulfur Mustard and its simulant CEES are nearly chemically equivalent ethyl sulfides, and as such behave similarly in both environmental settings and biological system, qualifying CEES as a suitable sulfur mustard chemical warfare simulant.

- GC/MS instrumentation is a sensitive and accurate analytical tool routinely used for the qualitative and quantitative determination of small- to medium-sized, low- to medium-polarity organic compounds. A nine-point calibration curve utilizing three independent stock solutions was generated, exhibiting a calibrated range for the standard calibration curve of 0.080-150 ppm (wt/wt) = 1200-2358000 ng CEES. The calibration curve exhibits excellent fit as demonstrated by the coefficient of determination of linear regression R^2, where $R^2 = 1.0$ (Figure 2). The limits of detection (LOD) and quantitation (LOQ) normal MS scan mode, the MS method used for the present decontamination study, are ≤ 40 ppb and ≤ 80 ppb, respectively, however in using either SIM (single-ion mode) or CI (chemical ionization, using methane) MS capabilities, the lowest limits of detection/quantitation of CEES approach ≤ 1 ppb (with SIM), and with CI could potentially approach ≤ 1 ppt (parts per trillion) or lower.

- As seen in Table 2, for Decontamination Efficacy and reduction in starting challenge Percent Efficacy determinations, on the non-porous Steel surfaces tested, because none of the highly volatile contaminant CEES was detected for either samples or Positive Control 1, an adequate measure of surface decontamination could not be calculated for Cd-plated and Carbon Steel coupons.

- On porous and/or chemically active coupon surfaces such as CARC and Black and Silicone Rubbers, modest to excellent Surface Decontamination of volatile CEES was realized; even on delicate or difficult-to-clean customized rubber, epoxy, or polyester surfaces, DeconGel is able to effectively remove hydrophobic contaminants through encapsulation/emulsification.

- To gauge DeconGel 1101's capabilities as an effective chemical (vapor) barrier, additional experimentation ((+)-Control 2) and data compilation provided a measure of DeconGel 1101's chemical barrier capacity - termed Chemical Barrier Efficiency (see Table 2): 1) On Black and Silicone Rubber surfaces, calculated DeconGel 1101 Chemical Barrier Efficiencies indicate a significant chemical surface barrier capability for DeconGel 1101 when applied to porous, chemically active rubber/ epoxy/polyester surfaces able to absorb and entrain contaminants; and 2) On non-porous, chemically inert Cd- and Carbon Steel surfaces, as well as the porous CARC coupon, due to CEES's significant volatility, contaminant applied to the steel coupons was not detectable after undergoing a 24-hour coupon-contaminant incubation as implemented for positive controls ((+)-Control 2). As such, samples and controls could not be compared, and therefore Chemical Barrier Efficiencies for Cd- and Carbon Steels and CARC surfaces could not be determined.

EXPERIMENTAL SECTION:

Reagents:

- Reagent grade CEES (Sigma-Aldrich) was utilized as the starting challenge (2.0 uL) contaminant in the decontamination study.

- Proprietarily formulated DeconGel 1101 was used as the decontaminant such that 1.0-1.1 g wet DeconGel was applied to contaminated surfaces, then allowed to dry for 24 h, and finally peeled off the surface. Immediately following gel peeling, the decontaminated coupon was extracted with solvent as elaborated below.

- Acetonitrile was used as the extraction solvent due to its ability to readily solvate CEES, compatibility with GC/MS analysis, relatively low volatility, and low toxicity profile. In addition, generated analytical calibration curves using acetonitrile exhibited favorable accuracy and precision.

Equipment (see Figure 1):

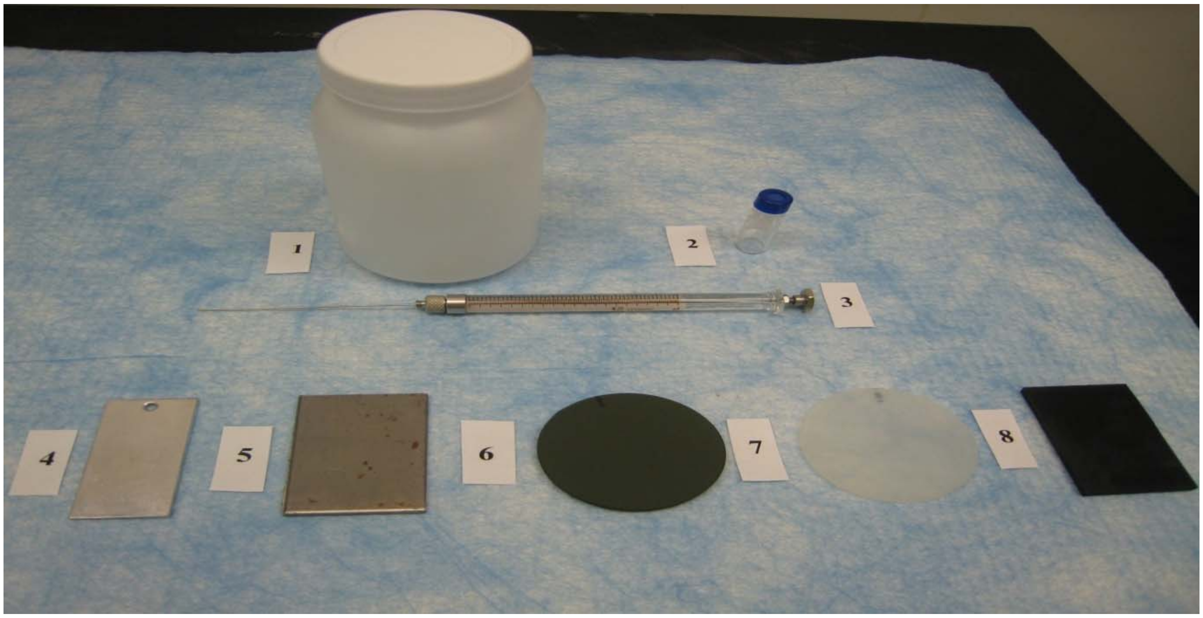
- A manual 2.0 uL glass syringe (National Scientific; Rockwood, TN) with a volume dispensing range of 0.0 to 2.0 uL, 0.01 uL grading, was used to dispense CEES contaminant, such that two non-touching 1.0 uL drops were dispensed on coupon surfaces per test.
- Calibrated manual pipets (Fisher Scientific; Fair Lawn, NJ) with disposable tips were used to generate the CEES calibration curve used to analytically determine amounts of CEES.
- A plastic squeezable bottle with coned dispenser was supplied with DeconGel 1101 and used to dispense 1.0-1.1 g wet DeconGel upon contaminated surfaces.
- Extraction solvent acetonitrile (20 mL) was dispensed into a 100 mL polypropylene bottle with screwcap prior to introduction of contaminated coupons using a 50 mL graduated cylinder.
- Samples and controls were prepared without further dilution/manipulation and immediately analyzed via GC/MS upon sample preparation.
- Due to the low boiling point of CEES, the 60 min contaminant contact time with coupon surfaces was conducted under an inverted Pyrex dessicator in attempts to minimize evaporative loss of CEES contaminant.
- A coupon area measurement in inches was performed using a standard ruler. Unit conversion and calculation was then conducted to achieve a coupon area in cm^2. Wet DeconGel was dispensed on coupons bordering the coupon edges and then the surface void filled in evenly as to maximize the spreading of DeconGel throughout the entire coupon surface area.
- A Thermo DSQ II capillary GC-double quadrupole MS with autosampler in positive mode was used to determine CEES concentration (ppm, wt/wt) of all samples and controls using a 9-point calibration curve utilizing three independent stock solutions.

Materials (see Figure 1):

- Coupons utilized in the decontamination study of CEES include Cadmium-plated Steel (12.75 cm^2 surface area), Carbon Steel (17.85 cm^2 surface area), Chemical Agent Resistant Coating (CARC) (19.63 cm^2 surface area), Black Rubber (16.83 cm^2 surface area), and Silicone

Rubber (19.63 cm^2 surface area). All coupons were supplied by JPEO to Dr. Garry Edgington (CBI Polymers, Inc.; Honolulu, HI) and were conditioned at RT on a laboratory countertop overnight.

Figure 1. Equipment and Materials utilized for the sulfur mustard simulant CEES decontamination study on multiple surfaces



Legend: **1.** 100 mL wide mouth extraction bottle with screw cap, **2.** GC vapor-tight sampling vial, **3.** 2 uL syringe for contaminant dispensing, **4.** Cd-plated Steel coupon, **5.** Carbon Steel coupon, **6.** CARC coupon, **7.** Silicone Rubber coupon, **8.** Black Rubber coupon.

Calculations:

- **Eqn 1.** Decontamination Efficacy

  = [(Contamination Density (CD) (g/m^2) of (+)-Control 1 – CD (g/m^2) of Remaining Agent) / CD (g/m^2) of (+)-Control 1] x 100%

- **Eqn 2.** Percent Efficacy

  = [(RA_M (ng) of Dose Confirmation Mass – RA_M (ng) of Remaining Agent Mass) / RA_M (ng) of Dose Confirmation Mass] x 100%

- **Eqn 3.** Chemical Barrier Efficiency

  = [1 – (RA_M (ng) of (+)-Control 2 / (RA_M (ng) of Remaining Agent Mass – (RA_M (ng) of (+)-Control 2))] x 100%

Detailed Test Summary:

- Experimental decontamination sampling and controls (positive and negative) were conducted on five different coupon types (as previously mentioned) in five replicates.

- Upon addition of 2.0 uL of CEES (experimental samples and positive controls) to coupon surfaces, contaminated coupons were placed under an inverted Pyrex dessicator for 60 min, and then either a) placed in extraction solvent via extraction bottle (for positive controls), or b) 1.0-1.1 g of wet DeconGel 1101 was poured evenly and thoroughly throughout the coupon surface area, DeconGel was allowed to dry for 24 h, the dried gel was peeled from the contaminated surface, and the contaminated coupon was immediately placed in an inert polypropylene bottle pre-filled with 20 mL extraction solvent as to completely submerse the coupon in extraction solvent. The extraction bottle was screw-capped, then swirled to facilitate contaminant extraction and let to stand for 60 min. Upon sample/control preparation, all experiments were immediately set-up for GC/MS analytical analysis in appropriate screw-cap GC vapor-tight vials (Fisher Scientific; Fair Lawn, NJ) (Figure 1).

- For negative controls, no contaminant was applied to coupon surfaces, and instead 1.0-1.1 g of wet DeconGel 1101 was poured evenly and thoroughly throughout the coupon surface area. DeconGel was allowed to dry for 24 h, the dried gel was peeled from coupon surfaces, and the coupon placed in an inert polypropylene bottle pre-filled with 20 mL extraction solvent as to completely submerse the coupon in extraction solvent. The extraction bottle was screw-capped, then swirled and let to stand for 60 min. Upon sample/control preparation, all experiments were immediately set-up for GC/MS analytical analysis.

- For positive controls, 2 uL of contaminant was applied to coupon surfaces and applied for either a) 1 h ((+)-Control 1), or b) 24 h ((+)-Control 2) coupon-contaminant incubation time, and then treated as described above for sample preparation. Positive Control 1 was implemented to provide an experimental control addressing the typical 60 min coupon-contaminant contact time; Positive Control 2 was implemented to provide an experimental control addressing the 24 h surface residence time employed for both contaminant and decontaminant, the timeframe needed to afford both adequate drying, and optimized surface decontamination by DeconGel 1101. After the appropriate incubation time, no decontaminant was applied, and contaminated coupons were immediately placed in an inert polypropylene bottle pre-filled with 20 mL extraction solvent. The extraction bottle was screw-capped, then swirled to facilitate contaminant extraction and let to stand for 60 min. Upon sample/control preparation, all experiments were immediately set-up for GC/MS analytical analysis.

- For dose confirmation samples, seven replicates were performed such that 2.0 uL CEES was charged via glass syringe into an inert polypropylene bottle pre-filled with 20 mL extraction solvent. The extraction bottle was screw-capped, then swirled to facilitate contaminant extraction and let to stand for 60 min. Upon sample preparation, all experiments were immediately set-up for GC/MS analytical analysis.

TEST SPECIFIC:

Precondition Coupons:

- Coupons were conditioned for 24 h on the laboratory countertop under normal ambient air-conditioned laboratory RT (25 ^{o}C) and humidity (40-60% humidity), where no extreme temperatures or humidity fluctuations were noted.

Contamination:

- CEES coupon contamination was performed (as previously mentioned) such that 2.0 uL total CEES neat liquid at RT (2x 1.0 uL non-touching drops) was manually added via glass syringe. Contaminated coupons were immediately placed under an inverted Pyrex dessicator and let to stand for 60 min.

- Contamination density (CD) of coupons were optimized to utilize the entire surface area of coupons by applying two non-touching 1.0 uL contaminant drops, and by applying DeconGel decontaminant on top and evenly over the whole coupon surface area. Coupons utilized in the decontamination study of CEES include Cadmium-plated Steel (12.75 cm^2 surface area), Carbon Steel (17.85 cm^2 surface area), Chemical Agent Resistant Coating (CARC) (19.63 cm^2 surface area), Black Rubber (16.83 cm^2 surface area), and Silicone Rubber (19.63 cm^2 surface area).

Dose Confirmation Sample Preparation:

- For dose confirmation samples, seven replicates were performed such that 2.0 uL CEES was charged via glass syringe into an inert polypropylene bottle pre-filled with 20 mL acetonitrile extraction solvent. The extraction bottle was screw-capped, then swirled to facilitate contaminant extraction and let to stand for 60 min. Upon sample preparation, all experiments were immediately set-up for GC/MS analytical analysis.

Post-Contamination Surface Contamination Observation:

- On non-porous inert coupon surfaces such as Cadmium-plated Steel and Carbon Steel, we observed that CEES contaminant drops initially beaded on the surface. However, after about 20 min, drops applied to Carbon Steel slowly spread out to cover roughly 2-3x the initial surface area, while contaminant drops applied to Cadmium-plated Steel stayed beaded throughout the 60 min contaminant-coupon contact hold time. After the 60 min hold time, CEES contaminant drops were evidenced on non-porous steel surfaces as wetted drops (Cd-plated Steel) or as wetted smears (Carbon Steel); after the 24 h hold time employed for (+)-Control 2, no CEES contaminant drops were evidenced on non-porous steel surfaces as either drops or smears.
- On porous and/or chemically active coupon surfaces such as CARC and Silicone Rubber, CEES contaminant drops were observed to rapidly spread out from the initial drop surface area over 10 min, becoming absorbed into coupon surfaces as evidenced by a wetted surface roughly 2-3x the initial surface area that persisted for the 60 min hold time; after the 24 h hold time employed for (+)-Control 2, no CEES contaminant drops were evidenced on these surfaces. When CEES drops were applied on top of Black Rubber, the contaminant did not roll or spread out, but instead absorbed within minutes into the coupon surface as evidenced by a dry surface bulging that persisted throughout the 24 h hold time employed for (+)-Control 2.

Aging:

- Coupons were conditioned for 24 h on the laboratory countertop under normal ambient air-conditioned laboratory room temperature (25 ^{o}C) and humidity (40-60% humidity), where no extreme temperature or humidity fluctuations were noted.

Post-Aging Surface Contamination Observation:

- No significant difference in appearance, size, or color of conditioned/aged coupons were noted, indicating that no significant or unexpected swelling, shrinking, deformation, decomposition, or surface drying or wetting had occurred.

Pre-Rinse:

- No pre-rinsing step of coupons was performed. Coupons were delivered to CBI Polymers directly from JPEO in a residue-free, clean state and used as received.
- Coupons were conditioned for 24 h on the laboratory countertop under normal ambient air-conditioned laboratory RT (25 ^{o}C) and humidity (40-60% humidity), where no extreme temperatures or humidity fluctuations were noted.

Decontamination:

- To decontaminate the contaminated coupons, after the 60 min contaminant-coupon surface hold time under an inverted Pyrex dessicator, 1.0-1.1 g of wet DeconGel 1101 at room temperature was poured evenly on top of the contaminated coupon throughout the whole surface area in an attempt to decontaminate all contaminant that might have spread throughout the coupon surface area.
- DeconGel was allowed to dry for 24 h, the dried gel was peeled from the contaminated surface, and the contaminated coupon was immediately placed in an inert polypropylene bottle pre-filled with 20 mL extraction solvent as to completely submerse the coupon in extraction solvent. The extraction bottle was screw-capped, then swirled to facilitate contaminant extraction and let to stand for 60 min. Upon sample preparation, all experiments were immediately set-up for GC/MS analytical analysis.

Post-Rinse:

- No post-rinsing step of DeconGel decontaminant was performed due to the chemical nature and intended utility of DeconGel; DeconGel is a viscous gel that upon surface contact first spreads out into a thin layer and then must air-dry for 12-24 h before it is peeled off of the contaminated surface. Rinsing either wet or dry DeconGel will result in partial to complete dissolution/emulsification of the decontaminant and most likely reduce its decontamination efficacy.

Drying:

- Upon addition of decontaminant to contaminated surfaces, DeconGel was allowed to dry for 24 h, the dried gel was peeled from the contaminated surface, and the contaminated coupon was immediately placed in an inert polypropylene bottle pre-filled with 20 mL extraction solvent as to completely submerse the coupon in the extraction solvent.

- Subsequent coupon post-washing or drying steps were not conducted or deemed necessary for this decontamination study.

Remaining Agent Measurement:

- Upon addition of 2.0 uL of CEES (experimental samples and positive controls) to coupon surfaces, contaminated coupons were placed under an inverted Pyrex dessicator for 60 min, and then either placed in extraction solvent as described above (for positive control) or 1.0-1.1 g of wet DeconGel 1101 was poured evenly throughout the whole coupon surface area. DeconGel was allowed to dry for 24 h, the dried gel was peeled from the contaminated surface, and the contaminated coupon was immediately placed in an inert polypropylene bottle pre-filled with 20 mL extraction solvent to completely submerse the coupon in extraction solvent. The extraction bottle was screw-capped, then swirled to facilitate contaminant extraction and let to stand for 60 min. Upon sample/control preparation, all experiments were immediately set-up for GC/MS analytical analysis in appropriate screw-cap GC vapor-tight vials (Fisher Scientific; Fair Lawn, NJ).

Chromatographic Analysis:

- A Thermo DSQ II capillary GC-double quadrupole MS with autosampler in positive mode was used to determine CEES concentration (ppm, wt/wt) of all samples and controls using a 9-point calibration curve utilizing three independent stock solutions (Figure 2).

- A Restek (Bellefonte, PA) Rtx-5 polysiloxane capillary column was employed using Temperature Program: Initial at 45 ^{o}C, hold 1 min, Ramp at 30 ^{o}C/min to 300 ^{o}C, hold for 5 min.

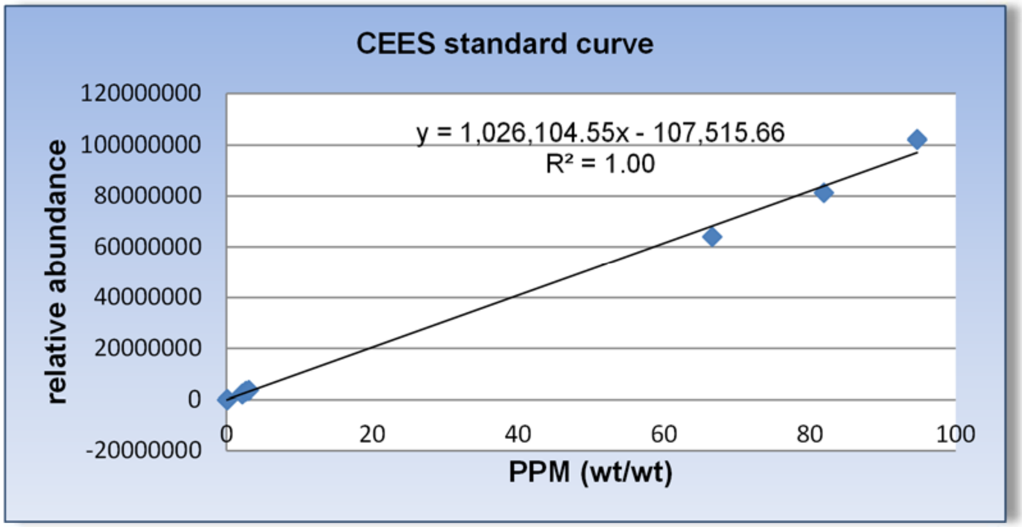
- The calibrated range for the standard calibration curve is 0.080-150 ppm (wt/wt) = 1200-2358000 ng CEES.

- The limits of detection (LOD) and quantitation (LOQ) normal MS scan mode, the MS method used for the present decontamination study, are ≤ 40 ppb and ≤ 80 ppb, respectively, however in using either SIM (single-ion mode) or CI (chemical ionization using methane) MS capabilities the lowest limits of detection/quantitation of CEES approach ≤ 1 ppb (with

SIM), and with CI could potentially approach ≤ 1 ppt (parts per trillion) or lower.

- The calibration curve exhibits a curve fitting as approximated by the coefficient of determination of linear regression R^2, where $R^2 = 1.0$ (Figure 2).

Figure 2. CEES Standard Calibration Curve



Reporting Statements/Summary:

- Tables 1 and 2 contain the Data Test Set, and Decontamination Efficacy, Reduction in Starting Challenge Percent Efficacy, and Chemical Barrier Efficacy, respectively, for the sulfur mustard simulant contaminant CEES decontamination study conducted using DeconGel 1101 as the decontaminant on a number of coupon surfaces including Cadmium-plated and Carbon Steels, CARC, and Silicone and Black Rubbers by solvent extraction.

- As seen in Table 2, for Decontamination Efficacy and reduction in starting challenge Percent Efficacy determinations it is noted: 1) On the non-porous Steel surfaces tested, because none of the highly volatile contaminant CEES was detected for either samples or Positive Control 1, an adequate measure of surface decontamination could not be calculated for Cd-plated and Carbon Steel coupons; and 2) On porous and/or chemically active coupon surfaces DeconGel 1101 decontamination efficacy and reduction in starting challenge Percent Efficacy ranged from 28.6 and 99.9% (on CARC) to 54.6 and 80.5% (on Silicone Rubber) to 93.2 and 97.0% (on Black Rubber) as determined by residue analysis after solvent extraction. Overall, the decontamination study using decontaminant DeconGel 1101 on CEES contaminant reveals DeconGel's ability to both 1) encapsulate/emulsify hydrophobic contaminants such as CEES and other chlorinated ethyl sulfide/sulfur mustards, and 2) act as a chemical barrier against volatile agents such as CEES or other more toxic volatiles/substances that might pose a significant contact risk or possess prolonged environmental persistence.

- For porous and/or chemically active coupon surfaces such as CARC and Rubbers, modest to excellent surface decontamination of volatile CEES was realized; interestingly, CEES was noted to appreciably absorb into such coupon surfaces, which seemed to sequester/dissolve CEES within the coupon surfaces, hindering sufficient decontaminant-contaminant interaction and limiting DeconGel 1101's abilities to encapsulate/emulsify contaminant CEES as reflected by low Decontamination and Percent Efficacies (Table 2). On the other hand, CEES' limiting volatility introduced difficulties in accurately determining Surface Decontamination for all coupon types tested; rapid surface evaporation of CEES when applied to coupon surfaces could result in falsely inflated Decontamination Efficacies and Percent Efficacies.

- To gauge DeconGel 1101's capabilities as an effective chemical (vapor) barrier, additional experimentation ((+)-Control 2) and data compilation provided a measure of DeconGel 1101's chemical barrier capacity - termed Chemical Barrier Efficiency (see Table 2): 1) On Black and Silicone Rubber surfaces, calculated DeconGel 1101 Chemical Barrier Efficiencies indicate a significant chemical surface barrier capability for DeconGel 1101 when applied to porous, chemically active rubber/epoxy/polyester surfaces able to absorb and entrain contaminants; and 2) On non-porous, chemically inert Cd- and Carbon Steel surfaces, as well as the porous CARC coupon, due to CEES's significant volatility, contaminant applied to the steel coupons was not detectable after undergoing a 24-hour coupon-contaminant incubation as implemented for positive controls ((+)-Control 2). As such, samples and controls could not be compared, and therefore Chemical Barrier Efficiencies for Cd- and Carbon Steels and CARC surfaces could not be determined.

- The 60 min contact time between CEES contaminant and all coupons tested was deemed necessary, however adoption of a shorter contact time holds promise to allow the required interaction between contaminant and coupon surface while minimizing loss of volatile contaminant CEES to evaporation. Utilizing an inverted Pyrex dessicator to store contaminated coupons immediately after contamination and during the required 60 min contact time aided in retaining significant amounts of contaminant on/within the coupon surfaces.

- Since live agents may not exhibit the same bioactivity, volatility, or absorption characteristics as CEES, live agent testing is recommended to accurately measure DeconGel effectiveness on test surfaces.

APPLICATION INSTRUCTIONS FOR END-USERS

Use product directly as is from container. DO NOT DILUTE. Masking or painters tape can be applied along one edge of the area that is to be decontaminated to aid creating a peeled edge to grip for peeling the dried film. Apply DeconGel using a paint brush, a trowel, a handheld sprayer, or an industrial grade sprayer (use DeconGel 1120 or 1121 for spray application).

The thickness of the gel and the number of coats is dictated by the surface to be decontaminated. Coating thickness required for good peel characteristics varies with substrate and generally increases with substrate porosity. It is recommended that first time customers test DeconGel on a small sample area to confirm the required film thickness and dry time for their specific application. If the film is difficult to peel, please apply an additional coat. A razor blade is useful to start the peel. Lay the blade nearly flat and fillet the edge of the film to create a tab that can be pulled. For surfaces that the gel adheres to well, such as concrete, 12" – 24" strips can be cut in the film resulting in less force being required to peel the film.

- Let DeconGel dry for 24 hours

Dry time will vary depending on humidity, temperature, air flow and thickness of the DeconGel. This can take from as little time as an hour for thin coats in a dry environment with plenty of airflow, to overnight or longer if thicker coats are applied in humid environments. Dry times exceeding 24 hours may sometimes be required for good peel performance on bare concrete, wood and other highly porous substrates and substrates with deep cracks or grooves. However, 18-24 hrs is often sufficient dry time on good quality concrete. It is recommended that users test a small area to determine drying time prior to applying DeconGel for an entire job. Supplemental heat or air circulation will accelerate DeconGel's drying time for any job.

- Peel DeconGel off the surface by starting from one of the edges

When dry, the product locks the contaminants into a polymer matrix. The film containing the encapsulated contamination can then be peeled. DeconGel peels from most non-porous and porous hard surfaces if the dried film is thick enough. If the film is difficult to peel, add another coat, let dry, and peel. In most cases the DeconGel will come off in a single sheet but for odd shaped surfaces you may be required to score DeconGel in order to be able to peel it off.

- Dispose of the dried DeconGel in accordance with the local, state and Federal disposal regulations of the contaminant/substance you are removing. DeconGel itself has no special disposal restrictions.

For more information on application techniques visit our website:
www.decongel.com

7.20. End-User Report for CWA Sulfur Mustard-Simulant (CEPS)

TITLE: Surface Decontamination of the Sulfur Mustard Simulant 2-Chloroethyl Phenyl Sulfide (CEPS) by DeconGel 1101

ANALYST: Geoff Nadolski PhD, Senior Polymer Scientist, CBI Polymers, Inc.

SUPERVISOR: Garry J. Edgington PhD, Chief Scientist, CBI Polymers, Inc.

OBJECTIVE: Evaluate the decontamination efficacy of DeconGel 1101 on coupon types:

1) Cadmium-plated Steel,
2) Carbon Steel,
3) Chemical agent resistant coating (CARC),
4) Silicone Rubber, and
5) Black Rubber surfaces contaminated with the Sulfur Mustard simulant CEPS.

- Experimentation and Reporting conducted following JPEO-CBD Source Document:
  - 2007 Chemical Decontamination Performance Evaluation Testing,
  - Section 6: Panel Contact Test to Determine Contact Hazard,
  - Section 6-E: Panel (Coupon) Extraction Method to Determine Remaining Agent;
- Sensitive (low ppb-level) GC/MS (gas chromatography/mass spectrometry) analytical methods developed.

HAZARDOUS MATERIALS RELEVANCE: Sulfur Mustard, bis-(2-chloroethyl) sulfide, is a powerful vesicant, cytotoxic agent regulated under the 1993 Chemical Weapons Convention (CWC). Sulfur mustard can be deployed by spraying, or more typically distributed via artillery shells and bombs. For experimental testing purposes, the half-mustard 2-chloroethyl phenyl sulfide (CEPS) is commonly used as an effective yet less toxic simulant for sulfur mustard chemical warfare agents.

SUMMARY RESULTS:

- On the non-porous steel surfaces tested, excellent surface decontamination was achieved by applying DeconGel 1101 onto contaminated surfaces facilitating encapsulation/emulsification of contaminants by DeconGel's active components.
    - DeconGel 1101 reduction in starting challenge Percent Efficacies, a measure comparing samples to the Dose Confirmation Control, ranged from 96.3% (on Cd-plated steel) to 97.2% (on Carbon steel) as determined by solvent extraction;
    - DeconGel 1101 Decontamination Efficacies, a measure comparing samples to the Positive Control (adjusted accordingly regarding particular coupon surface area), ranged from 96.3% (on Cd-plated steel) to 97.2% (on carbon steel) as determined by solvent extraction.
- On porous and/or chemically active coupon surfaces such as CARC and Black and Silicone Rubbers, modest Surface Decontamination of CEPS was realized; even on delicate or difficult-to-clean customized rubber, epoxy, or polyester surfaces, DeconGel is able to effectively remove hydrophobic contaminants through encapsulation/emulsification.
    - Reduction in starting challenge Percent Efficacies of DeconGel 1101 ranged from 51.2% (on CARC) to 51.8% (on Silicone Rubber) to 76.9% (on Black Rubber) as determined by solvent extraction.
    - DeconGel 1101 Decontamination Efficacies ranged from 51.2% (on CARC) to 51.9% (on Silicone Rubber) to 76.9% (on Black Rubber) as determined by solvent extraction.
- Due to its viscous, slow-drying, and emulisifying/encapsulating properties, DeconGel 1101 can serve as a partial chemical (vapor) barrier. This chemical barrier effect can serve to reduce the release of contaminant to the surrounding environment, as well as promote a prolonged residence-time inside the gel layer. An extended residence-time in the gel is predicted to enhance the efficacy of neutralizing agents that are proposed to be employed in future versions of the gel. This reduction in rate of release of agents is proposed to be especially valuable in enhancing neutralization efficacies of CWAs with high reactivity such as sulfur mustards and phosphono-esters.

RESULTS: Tables 1 and 2 contain the Data Test Set, and Decontamination Efficacy and Reduction in Starting Challenge Percent Efficacy, respectively, for the sulfur mustard simulant contaminant CEPS decontamination study conducted using DeconGel 1101 as the decontaminant on a number of coupon surfaces including Cadmium-plated and Carbon Steels, CARC, Silicone Rubber and Black Rubbers by solvent extraction.

Table 1. DeconGel decontaminant Data Test Set for CEPS decontamination study on multiple surfaces as determined by residue analysis after solvent extraction

Coupon Type		**RA_M** (ng)	**CD** (g/m^2)
Cd-plated Steel	(-)-Control[1]	ND[2]	ND
	Remaining Agent	85048.3 ± 826.0	0.067 ± 0.0007
	(+)-Control 1[3]	2338224 ± 7223.0	1.83 ± 0.0057
	Dose Confirmation	2334869 ± 8674	N/A[4]
Carbon Steel	(-)-Control	ND	ND
	Remaining Agent	64382.8 ± 751.3	0.036 ± 0.0003
	(+)-Control 1	2335394 ± 9478.8	1.31 ± 0.0055
	Dose Confirmation	2334869 ± 8674	N/A
CARC	(-)-Control	ND	ND
	Remaining Agent	1138814 ± 5408.6	0.580 ± 0.0028
	(+)-Control 1	2335112 ± 7818.6	1.19 ± 0.0041
	Dose Confirmation	2334869 ± 8674	N/A
Silicone Rubber	(-)-Control	ND	ND
	Remaining Agent	1125300 ± 3701.8	0.573 ± 0.0019
	(+)-Control 1	2337627 ± 8288.8	1.19 ± 0.0042
	Dose Confirmation	2334869 ± 8674	N/A
Black Rubber	(-)-Control	ND	ND
	Remaining Agent	539647.6 ± 1167.7	0.321 ± 0.0007
	(+)-Control 1	2337564 ± 8147.2	1.39 ± 0.0048
	Dose Confirmation	2334869 ± 8674	N/A

[1] (-)-Control: no contaminant applied, then decontaminant applied

[2] ND: not detected, See Test Specific section, lower limits of detection (LOD) for CEPS using GC/MS ≤ 50 ppb

[3] (+)-Control 1: contaminant applied for 1 h, no decontaminant applied

[4] N/A: Not Applicable

RA_M: Remaining Agent (mass); CD: Contamination Density

Table 2. Decontamination Efficacy and Percent Efficacy evaluation for the sulfur mustard simulant CEPS decontamination study on multiple surfaces as determined by residue analysis after solvent extraction

Coupon Type	**Decontamination Efficacy[1] (%)**	**Percent Efficacy[2] (%)**
Cd-plated Steel	96.3 ± 0.1	96.3 ± 0.1
Carbon Steel	97.2 ± 0.1	97.2 ± 0.1
CARC	51.2 ± 0.51	51.2 ± 0.47
Silicone Rubber	51.9 ± 0.32	51.8 ± 0.25
Black Rubber	76.9 ± 0.11	76.9 ± 0.1

[1] See Decontamination Efficacy equation (Eqn 1) in Experimental section

[2] See Percent Efficacy equation (Eqn 2) in Experimental section

METHOD DEVELOPMENT OPTIMIZATIONS/OBSERVATIONS:

- In applying a small amount (2.0 uL) of neat CEPS contaminant as the starting challenge, there was no need to brush the contaminant throughout coupon surfaces to ensure a thin layer of CEPS was loaded onto coupon surfaces; using a minute amount of contaminant facilitated a favorable interaction between DeconGel and CEPS without overloading the relatively small amount of applied DeconGel decontaminant (1.0-1.1 g).

- Acetonitrile solvent was used to dissolve CEPS contaminant and to generate a standard calibration curve possessing a significant concentration range and favorable accuracy and precision. Acetonitrile was used as the extraction solvent due to its ability to readily solvate CEPS, compatibility with GC/MS analysis, relatively low volatility, and low toxicity profile. In accordance with the JPEO guidelines, 20 mL of acetonitrile extraction solvent was effective in generating precise data as evidenced by the low standard deviation of the modestly concentrated Dose Confirmation Samples ((DCS); DCS avg= 2224869 ± 8674 ng)

- Sulfur Mustard and its simulant CEPS are nearly chemically equivalent ethyl sulfides, and as such behave similarly in both environmental settings and biological system, qualifying CEPS as a suitable sulfur mustard chemical warfare simulant.

- GC/MS instrumentation is a sensitive and accurate analytical tool routinely used for the qualitative and quantitative determination of small- to medium-sized, low- to medium-polarity organic compounds. A nine-point calibration curve utilizing three independent stock solutions was generated, exhibiting a calibrated range for the standard calibration curve of 0.050-200 ppm (wt/wt) = 786-3144000 ng CEPS. The calibration curve exhibits excellent fit as demonstrated by the coefficient of determination of linear regression R^2, where $R^2 = 1.0$ (Figure 2). The limits of detection (LOD) and quantitation (LOQ) normal MS scan mode, the MS method used for the present decontamination study, are ≤ 40 ppb and ≤ 51 ppb, respectively, however in using either SIM (single-ion mode) or CI (chemical ionization, using methane) MS capabilities, the lowest limits of detection/quantitation of CEPS approach ≤ 1 ppb (with SIM), and with CI could potentially approach ≤ 1 ppt (parts per trillion) or lower.

- As seen in Table 2, for Decontamination Efficacy and reduction in starting challenge Percent Efficacy determinations, on non-porous inert coupon Steel surfaces excellent surface decontamination was achieved by applying DeconGel 1101 onto contaminated surfaces facilitating encapsulation/emulsification of contaminants by DeconGel's active components.

- On porous and/or chemically active coupon surfaces such as CARC and Rubbers, modest surface decontamination of CEPS is noted; specifically, when reviewing the Percent Efficacy achieved in comparing remaining contaminant agent versus contaminant confirmation dosage, modest (for CARC and Silicone Rubber) to good (for Black Rubber) surface decontamination of CEPS was achieved. DeconGel is able to effectively encapsulate/emulsify hydrophobic contaminants such as CEPS or other chlorinated organo-sulfur/ethyl sulfides from such porous, delicate, or difficult-to-clean customized rubber, epoxy, or polyester surfaces.

EXPERIMENTAL SECTION:

Reagents:

- Reagent grade CEPS (Sigma-Aldrich, USA) was utilized as the starting challenge (2.0 uL) contaminant in the decontamination study.
- Proprietarily formulated DeconGel 1101 was used as the decontaminant such that 1.0-1.1 g wet DeconGel was applied to contaminated surfaces, then allowed to dry for 24 h, and finally peeled off the surface. Immediately following gel peeling, the decontaminated coupon was extracted with solvent as elaborated below.
- Acetonitrile was used as the extraction solvent due to its ability to readily solvate methyl benzoate, compatibility with GC/MS analysis, relatively low volatility, and low toxicity profile. In addition, generated analytical calibration curves using acetonitrile exhibited favorable accuracy and precision.

Equipment (see Figure 1):

- A manual 2.0 uL glass syringe (National Scientific; Rockwood, TN) with a volume dispensing range of 0.0 to 2.0 uL, 0.01 uL grading, was used to dispense CEPS contaminant, such that two non-touching 1.0 uL drops were dispensed on coupon surfaces per test.
- Calibrated manual pipets (Fisher Scientific; Fair Lawn, NJ) with disposable tips were used to generate the CEPS calibration curve used to analytically determine amounts of CEPS.
- A plastic squeezable bottle with coned dispenser was supplied with DeconGel 1101 and used to dispense 1.0-1.1 g wet DeconGel upon contaminated surfaces.
- Extraction solvent acetonitrile (20 mL) was dispensed into a 100 mL polypropylene bottle with screwcap prior to introduction of contaminated coupons using a 50 mL graduated cylinder.
- Samples and controls were prepared without further dilution/manipulation and immediately analyzed via GC/MS upon sample preparation.
- A coupon area measurement in inches was performed using a standard ruler. Unit conversion and calculation was then conducted to achieve a coupon area in cm^2. Wet DeconGel was dispensed on coupons bordering the coupon edges and then the surface void filled in evenly as

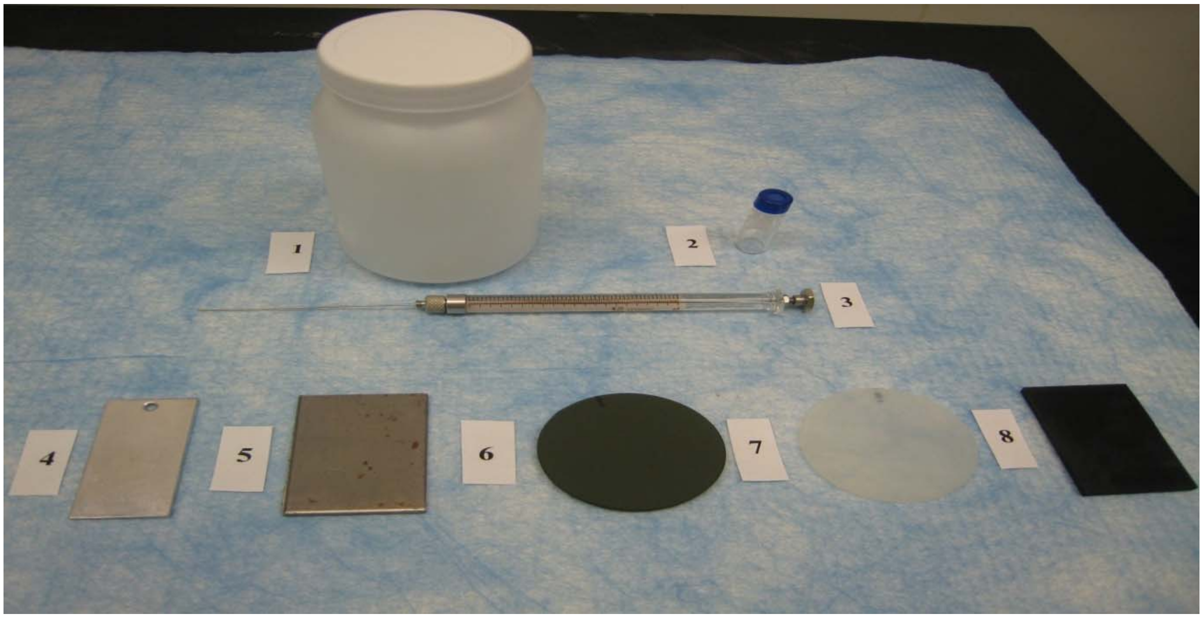
to maximize the spreading of DeconGel throughout the entire coupon surface area.

- A Thermo DSQ II capillary GC-double quadrupole MS with autosampler in positive mode was used to determine CEPS concentration (ppm, wt/wt) of all samples and controls using a 9-point calibration curve utilizing three independent stock solutions.

Materials (see Figure 1):

- Coupons utilized in the decontamination study of CEPS include Cadmium-plated Steel (12.75 cm^2 surface area), Carbon Steel (17.85 cm^2 surface area), Chemical Agent Resistant Coating (CARC) (19.63 cm^2 surface area), Black Rubber (16.83 cm^2 surface area), and Silicone Rubber (19.63 cm^2 surface area). All coupons were supplied by JPEO to Dr. Garry Edgington (CBI Polymers, Inc.; Honolulu, HI) and were conditioned at RT on a laboratory countertop overnight.

Figure 1. Equipment and Materials utilized for the sulfur mustard simulant CEPS decontamination study on multiple surfaces



Legend: **1.** 100 mL wide mouth extraction bottle with screw cap, **2.** GC vapor-tight sampling vial, **3.** 2 uL syringe for contaminant dispensing, **4.** Cd-plated Steel coupon, **5.** Carbon Steel coupon, **6.** CARC coupon, **7.** Silicone Rubber coupon, **8.** Black Rubber coupon.

Calculations:

- **Eqn 1.** Decontamination Efficacy

  = [(Contamination Density (CD) (g/m^2) of (+)-Control 1 – CD (g/m^2) of Remaining Agent) / CD (g/m^2) of (+)-Control 1] x 100%

- **Eqn 2.** Percent Efficacy

  = [(RA_M (ng) of Dose Confirmation Mass – RA_M (ng) of Remaining Agent Mass) / RA_M (ng) of Dose Confirmation Mass] x 100%

Detailed Test Summary:

- Experimental decontamination sampling and controls (positive and negative) were conducted on five different coupon types (as previously mentioned) in five replicates.

- Upon addition of 2.0 uL of CEPS (experimental samples and positive control) to coupon surfaces, contaminated coupons were placed under an inverted Pyrex dessicator for 60 min, and then either a) placed in extraction solvent via extraction bottle (for positive controls), or b) 1.0-1.1 g of wet DeconGel 1101 was poured evenly and thoroughly throughout the coupon surface area, DeconGel was allowed to dry for 24 h, the dried gel was peeled from the contaminated surface, and the contaminated coupon was immediately placed in an inert polypropylene bottle pre-filled with 20 mL extraction solvent as to completely submerse the coupon in extraction solvent. The extraction bottle was screw-capped, then swirled to facilitate contaminant extraction and let to stand for 60 min. Upon sample/control preparation, all experiments were immediately set-up for GC/MS analytical analysis in appropriate screw-cap GC vapor-tight vials (Fisher Scientific; Fair Lawn, NJ) (Figure 1).

- For negative controls, no contaminant was applied to coupon surfaces, and instead 1.0-1.1 g of wet DeconGel 1101 was poured evenly and thoroughly throughout the coupon surface area. DeconGel was allowed to dry for 24 h, the dried gel was peeled from coupon surfaces, and the coupon placed in an inert polypropylene bottle pre-filled with 20 mL extraction solvent as to completely submerse the coupon in extraction solvent. The extraction bottle was screw-capped, then swirled and let to stand for 60 min. Upon sample/control preparation, all experiments were immediately set-up for GC/MS analytical analysis.

- For positive controls, 2 uL of contaminant was applied to coupon surfaces and applied for 1 h ((+)-Control 1) coupon-contaminant incubation time, and then treated as described above for sample preparation. Positive Control was implemented to provide an experimental control addressing the typical 60 min coupon-contaminant contact time. After the appropriate incubation time, no decontaminant was applied, and contaminated coupons were immediately placed in an inert polypropylene bottle pre-filled with 20 mL extraction solvent. The extraction bottle was screw-capped, then swirled to facilitate contaminant extraction and let to stand for 60 min. Upon sample/control preparation, all experiments were immediately set-up for GC/MS analytical analysis.

- For dose confirmation samples, seven replicates were performed such that 2.0 uL CEPS was charged via glass syringe into an inert polypropylene bottle pre-filled with 20 mL extraction solvent. The extraction bottle was screw-capped, then swirled to facilitate contaminant extraction and let to stand for 60 min. Upon sample preparation, all experiments were immediately set-up for GC/MS analytical analysis.

TEST SPECIFIC:

Precondition Coupons:

- Coupons were conditioned for 24 h on the laboratory countertop under normal ambient air-conditioned laboratory RT (25 ^{o}C) and humidity (40-60% humidity), where no extreme temperatures or humidity fluctuations were noted.

Contamination:

- CEPS coupon contamination was performed (as previously mentioned) such that 2.0 uL total CEPS neat liquid at RT (2x 1.0 uL non-touching drops) was manually added via glass syringe. Contaminated coupons were immediately placed under an inverted Pyrex dessicator and let to stand for 60 min.

- Contamination density (CD) of coupons were optimized to utilize the entire surface area of coupons by applying two non-touching 1.0 uL contaminant drops, and by applying DeconGel decontaminant on top and evenly over the whole coupon surface area. Coupons utilized in the decontamination study of CEPS include Cadmium-plated Steel (12.75 cm^2 surface area), Carbon Steel (17.85 cm^2 surface area), Chemical Agent Resistant Coating (CARC) (19.63 cm^2 surface area), Black

Rubber (16.83 cm^2 surface area), and Silicone Rubber (19.63 cm^2 surface area).

Dose Confirmation Sample Preparation:

- For dose confirmation samples, seven replicates were performed such that 2.0 uL CEPS was charged via glass syringe into an inert polypropylene bottle pre-filled with 20 mL acetonitrile extraction solvent. The extraction bottle was screw-capped, then swirled to facilitate contaminant extraction and let to stand for 60 min. Upon sample preparation, all experiments were immediately set-up for GC/MS analytical analysis.

Post-Contamination Surface Contamination Observation:

- On non-porous inert coupon surfaces such as Cadmium-plated Steel and Carbon Steel, we observed that CEPS contaminant drops initially beaded on the surface. However, after about 20 min, drops applied to Carbon Steel slowly spread out to cover roughly 2-3x the initial surface area, while contaminant drops applied to Cadmium-plated Steel stayed beaded throughout the 60 min contaminant-coupon contact hold time. After the 60 min hold time, CEPS contaminant drops were evidenced on non-porous steel surfaces as wetted drops (Cd-plated Steel) or as wetted smears (Carbon Steel).
- On porous and/or chemically active coupon surfaces such as CARC and Silicone Rubber, CEPS contaminant drops were observed to rapidly spread out from the initial drop surface area over 10 min, becoming absorbed into coupon surfaces as evidenced by a wetted surface roughly 2-3x the initial surface area that persisted for the 60 min hold time. When CEPS drops were applied on top of Black Rubber, the contaminant did not roll or spread out, but instead absorbed within minutes into the coupon surface as evidenced by a dry surface bulging that persisted throughout the 24 h hold time employed for (+)-Control 2.

Aging:

- Coupons were conditioned for 24 h on the laboratory countertop under normal ambient air-conditioned laboratory room temperature (25 ^{o}C) and humidity (40-60% humidity), where no extreme temperature or humidity fluctuations were noted.

Post-Aging Surface Contamination Observation:

- No significant difference in appearance, size, or color of conditioned/aged coupons were noted, indicating that no significant or unexpected swelling, shrinking, deformation, decomposition, or surface drying or wetting had occurred.

Pre-Rinse:

- No pre-rinsing step of coupons was performed. Coupons were delivered to CBI Polymers directly from JPEO in a residue-free, clean state and used as received.
- Coupons were conditioned for 24 h on the laboratory countertop under normal ambient air-conditioned laboratory rt (25 ^{o}C) and humidity (40-60% humidity), where no extreme temperatures or humidity fluctuations were noted.

Decontamination:

- To decontaminate the contaminated coupons, after the 60 min contaminant-coupon surface hold time under an inverted Pyrex dessicator, 1.0-1.1 g of wet DeconGel 1101 at room temperature was poured evenly on top of the contaminated coupon throughout the whole surface area in an attempt to decontaminate all contaminant that might have spread throughout the coupon surface area.
- DeconGel was allowed to dry for 24 h, the dried gel was peeled from the contaminated surface, and the contaminated coupon was immediately placed in an inert polypropylene bottle pre-filled with 20 mL extraction solvent as to completely submerse the coupon in extraction solvent. The extraction bottle was screw-capped, then swirled to facilitate contaminant extraction and let to stand for 60 min. Upon sample preparation, all experiments were immediately set-up for GC/MS analytical analysis.

Post-Rinse:

- No post-rinsing step of DeconGel decontaminant was performed due to the chemical nature and intended utility of DeconGel; DeconGel is a viscous gel that upon surface contact first spreads out into a thin layer and then must air-dry for 12-24 h before it is peeled off of the contaminated surface. Rinsing either wet or dry DeconGel will result in partial to complete dissolution/emulsification of the decontaminant and most likely reduce its decontamination efficacy.

Drying:

- Upon addition of decontaminant to contaminated surfaces, DeconGel was allowed to dry for 24 h, the dried gel was peeled from the contaminated surface, and the contaminated coupon was immediately placed in an inert polypropylene bottle pre-filled with 20 mL extraction solvent as to completely submerse the coupon in the extraction solvent.

- Subsequent coupon post-washing or drying steps were not conducted or deemed necessary for this decontamination study.

Remaining Agent Measurement:

- Upon addition of 2.0 uL of CEPS (experimental samples and positive control) to coupon surfaces, contaminated coupons were placed under an inverted Pyrex dessicator for 60 min, and then either placed in extraction solvent as described above (for positive control) or 1.0-1.1 g of wet DeconGel 1101 was poured evenly throughout the whole coupon surface area. DeconGel was allowed to dry for 24 h, the dried gel was peeled from the contaminated surface, and the contaminated coupon was immediately placed in an inert polypropylene bottle pre-filled with 20 mL extraction solvent to completely submerse the coupon in extraction solvent. The extraction bottle was screw-capped, then swirled to facilitate contaminant extraction and let to stand for 60 min. Upon sample/control preparation, all experiments were immediately set-up for GC/MS analytical analysis in appropriate screw-cap GC vapor-tight vials (Fisher Scientific; Fair Lawn, NJ).

Chromatographic Analysis:

- A Thermo DSQ II capillary GC-double quadrupole MS with autosampler in positive mode was used to determine CEPS concentration (ppm, wt/wt) of all samples and controls using a 9-point calibration curve utilizing three independent stock solutions (Figure 2).

- A Restek (Bellefonte, PA) Rtx-5 polysiloxane capillary column was employed using Temperature Program: Initial at 120 °C, hold 1 min, Ramp at 30 °C/min to 320 °C, hold for 5 min.

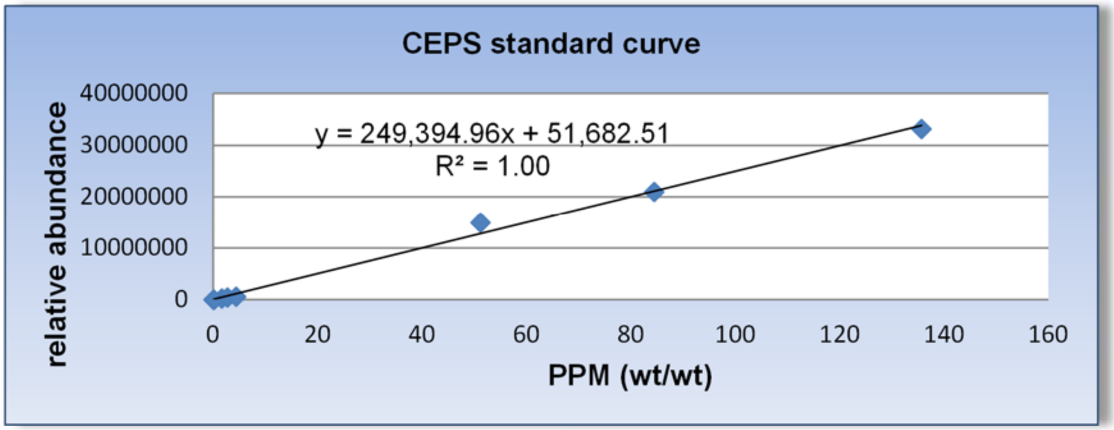
- The calibrated range for the standard calibration curve is 0.050-200 ppm (wt/wt) = 786-3144000 ng CEPS.

- The limits of detection (LOD) and quantitation (LOQ) normal MS scan mode, the MS method used for the present decontamination study, are $\leq$ 40 ppb and $\leq$ 51 ppb, respectively, however in using either SIM (single-ion mode) or CI (chemical ionization using methane) MS capabilities the lowest limits of detection/quantitation of CEPS approach $\leq$ 1 ppb (with

SIM), and with CI could potentially approach ≤ 1 ppt (parts per trillion) or lower.

- The calibration curve exhibits a curve fitting as approximated by the coefficient of determination of linear regression R^2, where $R^2 = 1.0$ (Figure 2).

Figure 2. CEPS Standard Calibration Curve



Reporting Statements/Summary:

- Tables 1 and 2 contain the Data Test Set, and Decontamination Efficacy and Reduction in Starting Challenge Percent Efficacy, respectively, for the sulfur mustard simulant contaminant CEPS decontamination study conducted using DeconGel 1101 as the decontaminant on a number of coupon surfaces including Cadmium-plated and Carbon Steels, CARC, and Silicone and Black Rubbers by solvent extraction.

- As seen in Table 2, when analyzing Decontamination Efficacy and reduction in starting challenge Percent Efficacy data it is noted: 1) on non-porous inert coupon surfaces such as Cadmium-plated and Carbon Steels, excellent Surface Decontamination of DeconGel 1101 on contaminant CEPS was achieved ranging from 96.3% (on Cd-plated Steel) to 97.2% (on Carbon Steel) as determined by residue analysis after solvent extraction; and 2) on porous and/or chemically active coupon surfaces DeconGel 1101 decontamination efficacy and reduction in starting challenge Percent Efficacy ranged from 51.2% (on CARC) to 51.8% (on Silicone Rubber) to 76.9% (on Black Rubber) as determined by residue analysis after solvent extraction. Overall, the decontamination study using decontaminant DeconGel 1101 on CEPS contaminant reveals DeconGel's ability to encapsulate/emulsify hydrophobic contaminants such as CEPS and other chlorinated organo-sulfur/ethyl sulfides. Additionally, due to its viscous, slow-drying, and emulisifying/encapsulating properties, DeconGel 1101 can serve as a partial chemical (vapor) barrier against agents such as sulfur mustards or other more toxic substances that might pose a significant contact risk or possess prolonged environmental persistence.

- For porous and/or chemically active coupon surfaces such as CARC and Rubbers, modest to good surface decontamination of the CEPS was realized; interestingly, CEPS was noted to appreciably absorb into such coupon surfaces, which seemed to sequester/dissolve CEPS within the coupon surfaces, hindering sufficient decontaminant-contaminant interaction and limiting DeconGel 1101's abilities to encapsulate/emulsify contaminant CEPS as reflected by low Decontamination and Percent Efficacies (Table 2).
- Since live agents may not exhibit the same bioactivity, volatility, or absorption characteristics as CEPS, live agent testing is recommended to accurately measure DeconGel effectiveness on test surfaces.

APPLICATION INSTRUCTIONS FOR END-USERS

Use product directly as is from container. DO NOT DILUTE. Masking or painters tape can be applied along one edge of the area that is to be decontaminated to aid creating a peeled edge to grip for peeling the dried film. Apply DeconGel using a paint brush, a trowel, a handheld sprayer, or an industrial grade sprayer (use DeconGel 1120 or 1121 for spray application).

The thickness of the gel and the number of coats is dictated by the surface to be decontaminated. Coating thickness required for good peel characteristics varies with substrate and generally increases with substrate porosity. It is recommended that first time customers test DeconGel on a small sample area to

confirm the required film thickness and dry time for their specific application. If the film is difficult to peel, please apply an additional coat. A razor blade is useful to start the peel. Lay the blade nearly flat and fillet the edge of the film to create a tab that can be pulled. For surfaces that the gel adheres to well, such as concrete, 12” – 24” strips can be cut in the film resulting in less force being required to peel the film.

- Let DeconGel dry for 24 hours

Dry time will vary depending on humidity, temperature, air flow and thickness of the DeconGel. This can take from as little time as an hour for thin coats in a dry environment with plenty of airflow, to overnight or longer if thicker coats are applied in humid environments. Dry times exceeding 24 hours may sometimes be required for good peel performance on bare concrete, wood and other highly porous substrates and substrates with deep cracks or grooves. However, 18-24 hrs is often sufficient dry time on good quality concrete. It is recommended that users test a small area to determine drying time prior to applying DeconGel for an entire job. Supplemental heat or air circulation will accelerate DeconGel’s drying time for any job.

- Peel DeconGel off the surface by starting from one of the edges



When dry, the product locks the contaminants into a polymer matrix. The film containing the encapsulated contamination can then be peeled. DeconGel peels from most non-porous and porous hard surfaces if the dried film is thick enough. If the film is difficult to peel, add another coat, let dry, and peel. In most cases the DeconGel will come off in a single sheet but for odd shaped surfaces you may be required to score DeconGel in order to be able to peel it off.

- Dispose of the dried DeconGel in accordance with the local, state and Federal disposal regulations of the contaminant/substance you are removing. DeconGel itself has no special disposal restrictions.

For more information on application techniques visit our website:
www.decongel.com

7.21. End-User Report for CWA VX-Simulant (Methyl Benzoate)

TITLE: Surface Decontamination of the VX Simulant Methyl Benzoate (MBz) by DeconGel 1101

ANALYST: Geoff Nadolski PhD, Senior Polymer Scientist, CBI Polymers, Inc.

SUPERVISOR: Garry J. Edgington PhD, Chief Scientist, CBI Polymers, Inc.

OBJECTIVE: Evaluate the decontamination efficacy of DeconGel 1101 on coupon types:

1) Cadmium-plated Steel,

2) Carbon Steel,

3) Chemical Agent Resistant Coating (CARC),

4) Silicone Rubber, and

5) Black Rubber.

- Experimentation and Reporting conducted following JPEO-CBD Source Document:
    - 2007 Chemical Decontamination Performance Evaluation Testing,
    - Section 6: Panel Contact Test to Determine Contact Hazard,
    - Section 6-E: Panel (Coupon) Extraction Method to Determine Remaining Agent;
- Sensitive (low ppb-level) GC/MS (gas chromatography/mass spectrometry) analytical methods developed.

HAZARDOUS MATERIALS RELEVANCE: VX, an organo-phosphonothioate, is a toxic nerve agent classified as a weapon of mass destruction by the United Nations. VX can be distributed as a liquid or aerosol, both pure and as a mixture, and due to its high viscosity and low volatility is considered an environmentally persistent biohazard. VX and its simulant methyl benzoate (MBz) have near identical Water:Octanol Coefficients (2.09 and 2.12, respectively), and as such behave similarly in both environmental settings and biological systems.

SUMMARY RESULTS:

- On the non-porous steel surfaces tested, excellent surface decontamination was achieved by applying DeconGel 1101 onto contaminated surfaces facilitating encapsulation/emulsification of contaminants by DeconGel's active components.
    - DeconGel 1101 reduction in starting challenge Percent Efficacies, a measure comparing samples to the Dose Confirmation Control, ranged from 97.2% (on Cd-plated steel) to 99.7% (on Carbon steel) as determined by solvent extraction;
    - DeconGel 1101 Decontamination Efficacies, a measure comparing samples to Positive Control 1 (adjusted accordingly regarding particular coupon surface area), ranged from 96.7% (on Cd-plated steel) to 99.2% (on carbon steel) as determined by solvent extraction.
- On porous and/or chemically active coupon surfaces such as CARC and Black and Silicone Rubbers, modest Surface Decontamination of MBz (a volatile plasticizer) was realized; even on delicate or difficult-to-clean customized rubber, epoxy, or polyester surfaces, DeconGel is able to effectively remove hydrophobic contaminants through encapsulation/emulsification.
    - Reduction in starting challenge (Percent Efficacies of DeconGel 1101) ranged from 37.9% (on Black Rubber) to 67.0% (on Silicone Rubber) to 91.8% (on CARC) as determined by solvent extraction.
    - DeconGel 1101 Decontamination Efficacies ranged from 26.5% (on Black Rubber) to 62.3% (on Silicone Rubber) to 67.3% (on CARC) as determined by solvent extraction.
- DeconGel 1101 can serve as a partial chemical (vapor) barrier. The vapor barrier effect demonstrates the reduction in the rate of release of agent to the surrounding atmosphere as well as indicating a prolonged residence-time inside the gel layer. An extended residence-time in the gel is predicted to enhance the efficacy of neutralizing agents that are proposed to be employed in future versions of the gel. This reduction in rate of release of agents is proposed to be especially valuable in enhancing neutralization efficacies of CWAs with high reactivity and volatility.

  To support the hypothesis that DeconGel 1101 can serve as an partial chemical (vapor) barrier, additional experimentation ((+)-Control 2) was performed to demonstrate the Chemical Barrier Efficiency of DeconGel 1101.

- On CARC and Black and Silicone Rubber surfaces, DeconGel 1101 Chemical Barrier Efficiencies ranged from 99.5% (on CARC) to 78.6% (on Silicone Rubber) to 99.0% (on Black Rubber) as determined by solvent extraction, indicating a significant chemical surface barrier capability for DeconGel 1101 when applied to porous, chemically active rubber/ epoxy/polyester surfaces able to absorb and entrain contaminants;
- On non-porous, chemically inert Cd- and Carbon Steel surfaces, due to MBz's significant volatility, contaminant applied to the steel coupons was not detectable after undergoing a 24-hour coupon-contaminant incubation as implemented for positive controls ((+)-Control 2). As such, samples and controls could not be compared, and therefore Chemical Barrier Efficiencies for Cd- and Carbon Steel surfaces could not be determined.

RESULTS: Tables 1 and 2 contain the Data Test Set, and Decontamination Efficacy, Reduction in Starting Challenge Percent Efficacy, and Chemical Barrier Efficiency, respectively, for the VX simulant contaminant MBz decontamination study conducted using DeconGel 1101 as the decontaminant on a number of coupon surfaces including Cadmium-plated and Carbon Steels, CARC, Silicone Rubber and Black Rubbers by solvent extraction.

Table 1. DeconGel decontaminant Data Test Set for MBz decontamination study on multiple surfaces as determined by residue analysis after solvent extraction

Coupon Type		RA_M (ng)	CD (g/m^2)
Cd-plated Steel	(-)-Control[1]	ND[2]	ND
	Remaining Agent	60302 ± 3593	0.05 ± 0.003
	(+)-Control 1[3]	1864078 ± 11034	1.46 ± 0.01
	(+)-Control 2[4]	ND	ND
	Dose Confirmation	2161275 ± 5981	N/A[5]
Carbon Steel	(-)-Control	ND	ND
	Remaining Agent	6266 ± 305.1	0.004 ± 0.001
	(+)-Control 1	792602 ± 25573	0.44 ± 0.02
	(+)-Control 2	ND	ND
	Dose Confirmation	2161275 ± 5981	N/A
CARC	(-)-Control	ND	ND
	Remaining Agent	176724 ± 8643	0.09 ± 0.004
	(+)-Control 1	543472 ± 10586	0.28 ± 0.01
	(+)-Control 2	885.77 ± 8.36	0.0005 ± 0.00006
	Dose Confirmation	2161275 ± 5981	N/A
Silicone Rubber	(-)-Control	ND	ND
	Remaining Agent	712525 ± 13315	0.36 ± 0.01
	(+)-Control 1	1890487 ± 15744	0.96 ± 0.01
	(+)-Control 2	126980.4 ± 463.5	0.06 ± 0.0006
	Dose Confirmation	2161275 ± 5981	N/A
Black Rubber	(-)-Control	ND	ND
	Remaining Agent	1341331 ± 8618	0.80 ± 0.01
	(+)-Control 1	1827953 ± 7127	1.08 ± 0.01
	(+)-Control 2	12989.8 ± 70.59	0.008 ± 0.00006
	Dose Confirmation	2161275 ± 5981	N/A

[1] (-)-Control: no contaminant applied, then decontaminant applied

[2] ND: not detected, See Test Specific section, lower limits of detection (LOD) for MBz using GC/MS ≤ 40 ppb

[3] (+)-Control 1: contaminant applied for 1 h, no decontaminant applied

[4] (+)-Control 2: contaminant applied for 24 h, no decontaminant applied

[5] N/A: Not Applicable

RA_M: Remaining Agent (mass); CD: Contamination Density

Table 2. Decontamination Efficacy, Percent Efficacy, and Chemical Barrier Efficiency evaluation for the VX simulant MBz decontamination study on multiple surfaces as determined by residue analysis after solvent extraction

Coupon Type	Decontamination Efficacy[1] (%)	Percent Efficacy[2] (%)	Chemical Barrier Efficiency[3] (%)
Cd-plated Steel	96.7 ± 0.19	97.2 ± 0.19	ND[4]
Carbon Steel	99.2 ± 0.11	99.7 ± 0.00	ND
CARC	67.3 ± 2.34	91.8 ± 0.42	99.5 ± 0.00
Silicone Rubber	62.3 ± 1.17	67 ± 0.96	78.6 ± 0.48
Black Rubber	26.5 ± 1.88	37.9 ± 0.98	99.0 ± 0.00

[1] See Decontamination Efficacy equation (Eqn 1) in Experimental section

[2] See Percent Efficacy equation (Eqn 2) in Experimental section

[3] See Chemical Barrier Efficiency equation (Eqn 3) in Experimental section

[4] ND: not determined (no contaminant detected for samples and/or controls)

NOTES:

- Due to MBz's low boiling point/volatility, significant loss of contaminant MBz was noted when storing contaminated coupons in a chemical hood for the required 60 min contact hold time. Utilizing an inverted Pyrex dessicator greatly improved both volatile surface retention and contaminant-coupon interaction, as evidenced after the 60 min hold time as either a significant surface retention of wetted drops, or as a raised surface.

- In applying a small amount (2.0 uL) of neat MBz contaminant as the starting challenge, there was no need to brush the volatile liquid contaminant throughout coupon surfaces to ensure a thin layer of MBz was loaded onto coupon surfaces; using a minute amount of contaminant facilitated a favorable interaction between DeconGel and MBz without overloading the relatively small amount of applied DeconGel decontaminant (1.0-1.1 g).

- Acetonitrile solvent was used to dissolve MBz contaminant and to generate a standard calibration curve possessing a significant concentration range and favorable accuracy and precision. Acetonitrile was used as the extraction solvent due to its ability to readily solvate methyl benzoate, compatibility with GC/MS analysis, relatively low volatility, and low toxicity profile. In accordance with the JPEO guidelines, 20 mL of acetonitrile extraction solvent was effective in generating precise data as evidenced by the low standard deviation of the modestly concentrated Dose Confirmation Samples ((DCS); DCS avg= 2161275 ± 5981 ng)

- VX and its simulant methyl benzoate (MBz) have near identical Water: Octanol Coefficients (2.09 and 2.12, respectively), and as such behave similarly in both environmental settings and biological system, qualifying MBz as a suitable VX chemical warfare simulant.

- GC/MS instrumentation is a sensitive and accurate analytical tool routinely used for the qualitative and quantitative determination of small- to medium-sized, low- to medium-polarity organic compounds. A greater than ten-point calibration curve utilizing three independent stock solutions was generated, exhibiting a calibrated range for the standard calibration curve of 0.040-360 ppm (wt/wt) = 629-5659200 ng MBz. The calibration curve exhibits excellent fit as demonstrated by the coefficient of determination of linear regression R^2, where $R^2 = 0.99$ (Figure 2). The limits of detection (LOD) and quantitation (LOQ) normal MS scan mode, the MS method used for the present decontamination study, are ≤ 40 ppb and ≤ 80 ppb, respectively, however in using either SIM (single-ion mode) or CI (chemical ionization, using methane) MS capabilities, the lowest limits of detection/quantitation of MBz approach ≤ 1 ppb (with SIM), and with CI could potentially approach ≤ 1 ppt (parts per trillion) or lower.

- As seen in Table 2, for Decontamination Efficacy and reduction in starting challenge Percent Efficacy determinations, on non-porous inert coupon Steel surfaces excellent surface decontamination was achieved by applying DeconGel 1101 onto contaminated surfaces facilitating encapsulation/emulsification of contaminants by DeconGel's active components.

- On porous and/or chemically active coupon surfaces such as CARC and Rubbers, modest surface decontamination of the volatile plasticizer MBz is noted; specifically, when reviewing the Percent Efficacy achieved in comparing remaining contaminant agent versus contaminant confirmation dosage, modest (for Black and Silicone Rubbers) to good (for CARC) surface decontamination of MBz was achieved. DeconGel is able to effectively encapsulate/emulsify hydrophobic contaminants such as MBz or other phosphate/carbonate esters from such porous, delicate, or difficult-to-clean customized surfaces as rubbers and epoxy/polyesters.

- To gauge DeconGel 1101's capabilities as an effective chemical (vapor) barrier, additional experimentation ((+)-Control 2) and data compilation provided a measure of DeconGel 1101's chemical barrier capacity - termed Chemical Barrier Efficiency (see Table 2): 1) On CARC and Black and Silicone Rubber surfaces, calculated DeconGel 1101 Chemical Barrier Efficiencies indicate a significant chemical surface barrier capability for DeconGel 1101 when applied to porous, chemically active rubber/ epoxy/polyester surfaces able to absorb and entrain contaminants; and 2) On non-porous, chemically inert Cd- and Carbon Steel surfaces, due to MBz's significant volatility, contaminant applied to the steel coupons was not detectable after undergoing a 24-hour coupon-contaminant incubation as implemented for positive controls ((+)-Control 2). As such, samples and controls could not be compared, and therefore Chemical Barrier Efficiencies for Cd- and Carbon Steel surfaces could not be determined.

EXPERIMENTAL SECTION:

Reagents:

- Reagent grade methyl benzoate (Fisher Scientific; Fair Lawn, NJ) was utilized as the starting challenge (2.0 uL) contaminant in the decontamination study.

- Proprietarily formulated DeconGel 1101 was used as the decontaminant such that 1.0-1.1 g wet DeconGel was applied to contaminated surfaces, then allowed to dry for 24 h, and finally peeled off the surface. Immediately following gel peeling, the decontaminated coupon was extracted with solvent as elaborated below.

- Acetonitrile was used as the extraction solvent due to its ability to readily solvate methyl benzoate, compatibility with GC/MS analysis, relatively low volatility, and low toxicity profile. In addition, generated analytical calibration curves using acetonitrile exhibited favorable accuracy and precision.

Equipment (see Figure 1):

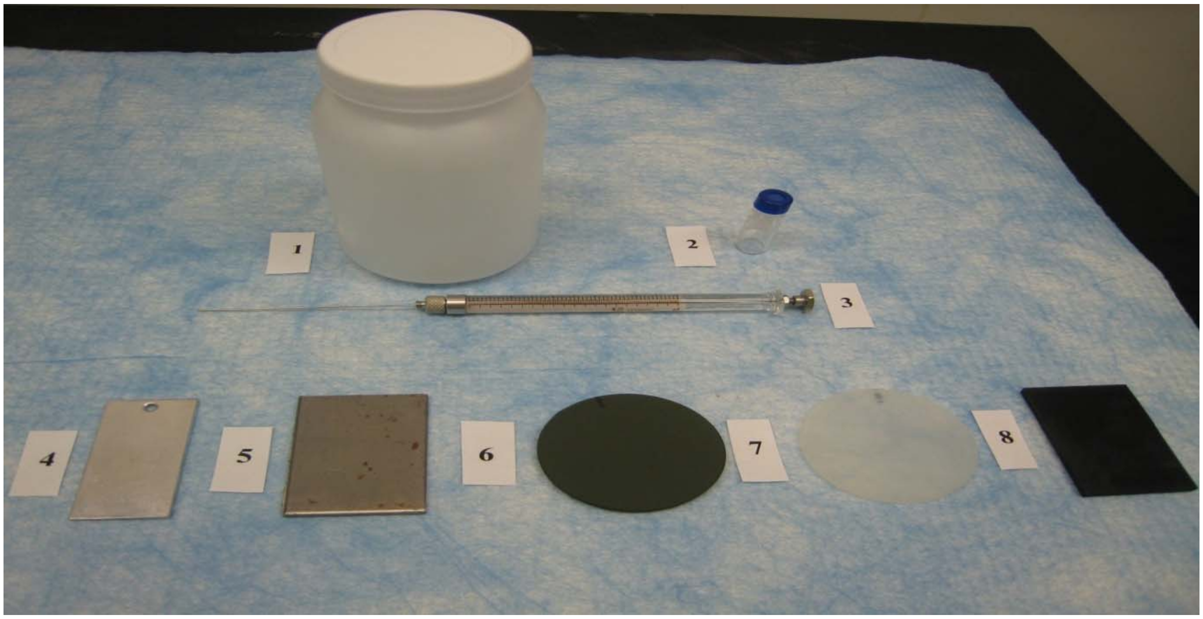
- A manual 2.0 uL glass syringe (National Scientific; Rockwood, TN) with a volume dispensing range of 0.0 to 2.0 uL, 0.01 uL grading, was used to dispense MBz contaminant, such that two non-touching 1.0 uL drops were dispensed on coupon surfaces per test.
- Calibrated manual pipets (Fisher Scientific; Fair Lawn, NJ) with disposable tips were used to generate the MBz calibration curve used to analytically determine amounts of MBz.
- A plastic squeezable bottle with coned dispenser was supplied with DeconGel 1101 and used to dispense 1.0-1.1 g wet DeconGel upon contaminated surfaces.
- Extraction solvent acetonitrile (20 mL) was dispensed into a 100 mL polypropylene bottle with screwcap prior to introduction of contaminated coupons using a 50 mL graduated cylinder.
- Samples and controls were prepared without further dilution/manipulation and immediately analyzed via GC/MS upon sample preparation.
- Due to the low boiling point of MBz, the 60 min contaminant contact time with coupon surfaces was conducted under an inverted Pyrex dessicator in attempts to minimize evaporative loss of MBz contaminant.
- A coupon area measurement in inches was performed using a standard ruler. Unit conversion and calculation was then conducted to achieve a coupon area in cm^2. Wet DeconGel was dispensed on coupons bordering the coupon edges and then the surface void filled in evenly as to maximize the spreading of DeconGel throughout the entire coupon surface area.
- A Thermo DSQ II capillary GC-double quadrupole MS with autosampler in positive mode was used to determine MBz concentration (ppm, wt/wt) of all samples and controls using a > 10 point calibration curve utilizing three independent stock solutions.

Materials (see Figure 1):

- Coupons utilized in the decontamination study of MBz include Cadmium-plated Steel (12.75 cm^2 surface area), Carbon Steel (17.85 cm^2 surface area), Chemical Agent Resistant Coating (CARC) (19.63 cm^2 surface area), Black Rubber (16.83 cm^2 surface area), and Silicone Rubber (19.63 cm^2 surface area). All coupons were supplied by JPEO to

Dr. Garry Edgington (CBI Polymers, Inc.; Honolulu, HI) and were conditioned on a laboratory countertop overnight.

Figure 1. Equipment and Materials utilized for the VX simulant MBz decontamination study on multiple surfaces



Legend: **1.** 100 mL wide mouth extraction bottle with screw cap, **2.** GC vapor-tight sampling vial, **3.** 2 uL syringe for contaminant dispensing, **4.** Cd-plated Steel coupon, **5.** Carbon Steel coupon, **6.** CARC coupon, **7.** Silicone Rubber coupon, **8.** Black Rubber coupon.

Calculations:

- **Eqn 1.** Decontamination Efficacy

  = [(Contamination Density (CD) (g/m^2) of (+)-Control 1 – CD (g/m^2) of Remaining Agent) / CD (g/m^2) of (+)-Control 1] x 100%

- **Eqn 2.** Percent Efficacy
-

  = [(RA$_M$ (ng) of Dose Confirmation Mass – RA$_M$ (ng) of Remaining Agent Mass) / RA$_M$ (ng) of Dose Confirmation Mass] x 100%

- **Eqn 3.** Chemical Barrier Efficiency

  = [1 – (RA$_M$ (ng) of (+)-Control 2 / (RA$_M$ (ng) of Remaining Agent Mass – (RA$_M$ (ng) of (+)-Control 2))] x 100%

Detailed Test Summary:

- Experimental decontamination sampling and controls (positive and negative) were conducted on five different coupon types (as previously mentioned) in five replicates.
- Upon addition of 2.0 uL of MBz (experimental samples and positive control) to coupon surfaces, contaminated coupons were placed under an inverted Pyrex dessicator for 60 min, and then either a) placed in extraction solvent via extraction bottle (for positive controls), or b) 1.0-1.1 g of wet DeconGel 1101 was poured evenly and thoroughly throughout the coupon surface area, DeconGel was allowed to dry for 24 h, the dried gel was peeled from the contaminated surface, and the contaminated coupon was immediately placed in an inert polypropylene bottle pre-filled with 20 mL extraction solvent as to completely submerse the coupon in extraction solvent. The extraction bottle was screw-capped, then swirled to facilitate contaminant extraction and let to stand for 60 min. Upon sample/control preparation, all experiments were immediately set-up for GC/MS analytical analysis in appropriate screw-cap GC vapor-tight vials (Fisher Scientific; Fair Lawn, NJ) (Figure 1).
- For negative controls, no contaminant was applied to coupon surfaces, and instead 1.0-1.1 g of wet DeconGel 1101 was poured evenly and thoroughly throughout the coupon surface area. DeconGel was allowed to dry for 24 h, the dried gel was peeled from coupon surfaces, and the coupon placed in an inert polypropylene bottle pre-filled with 20 mL extraction solvent as to completely submerse the coupon in extraction solvent. The extraction bottle was screw-capped, then swirled and let to stand for 60 min. Upon sample/control preparation, all experiments were immediately set-up for GC/MS analytical analysis.
- For positive controls, 2 uL of contaminant was applied to coupon surfaces and applied for either a) 1 h ((+)-Control 1), or b) 24 h ((+)-Control 2) coupon-contaminant incubation time, and then treated as described above for sample preparation. Positive Control 1 was implemented to provide an experimental control addressing the typical 60 min coupon-contaminant contact time; Positive Control 2 was implemented to provide an experimental control addressing the 24 h surface residence time employed for both contaminant and decontaminant, the timeframe needed to afford both adequate drying, and optimized surface decontamination by DeconGel 1101. After the appropriate incubation time, no decontaminant was applied, and contaminated coupons were immediately placed in an inert polypropylene bottle pre-filled with 20 mL extraction solvent. The extraction bottle was screw-capped, then swirled to facilitate

contaminant extraction and let to stand for 60 min. Upon sample/control preparation, all experiments were immediately set-up for GC/MS analytical analysis.

- For dose confirmation samples, seven replicates were performed such that 2.0 uL MBz was charged via glass syringe into an inert polypropylene bottle pre-filled with 20 mL extraction solvent. The extraction bottle was screw-capped, then swirled to facilitate contaminant extraction and let to stand for 60 min. Upon sample preparation, all experiments were immediately set-up for GC/MS analytical analysis.

TEST SPECIFIC:

Precondition Coupons:

- Coupons were conditioned for 24 h on the laboratory countertop under normal ambient air-conditioned laboratory RT (25 ^{o}C) and humidity (40-60% humidity), where no extreme temperatures or humidity fluctuations were noted.

Contamination:

- MBz coupon contamination was performed (as previously mentioned) such that 2.0 uL total MBz neat liquid at room temperature (2x 1.0 uL non-touching drops) was manually added via glass syringe. Contaminated coupons were immediately placed under an inverted Pyrex dessicator and let to stand for 60 min.

- Contamination density (CD) of coupons were optimized to utilize the entire surface area of coupons by applying two non-touching 1.0 uL contaminant drops, and by applying DeconGel decontaminant on top and evenly over the whole coupon surface area. Coupons utilized in the decontamination study of MBz include Cadmium-plated Steel (12.75 cm^2 surface area), Carbon Steel (17.85 cm^2 surface area), Chemical Agent Resistant Coating (CARC) (19.63 cm^2 surface area), Black Rubber (16.83 cm^2 surface area), and Silicone Rubber (19.63 cm^2 surface area).

Dose Confirmation Sample Preparation:

- For dose confirmation samples, seven replicates were performed such that 2.0 uL MBz was charged via glass syringe into an inert polypropylene bottle pre-filled with 20 mL acetonitrile extraction solvent. The extraction bottle was screw-capped, then swirled to facilitate contaminant extraction and let to stand for 60 min. Upon sample preparation, all experiments were immediately set-up for GC/MS analytical analysis.

Post-Contamination Surface Contamination Observation:

- On non-porous inert coupon surfaces such as Cadmium-plated Steel and Carbon Steel, we observed that MBz contaminant drops initially beaded on the surface. However, after about 20 min, drops applied to Carbon Steel slowly spread out to cover roughly 2-3x the initial surface area, while contaminant drops applied to Cadmium-plated Steel stayed beaded throughout the 60 min contaminant-coupon contact hold time. After the 60 min hold time, MBz contaminant drops were evidenced on non-porous steel surfaces as wetted drops (Cd-plated Steel) or as wetted smears (Carbon Steel); after the 24 h hold time employed for (+)-Control 2, no MBz contaminant drops were evidenced on non-porous steel surfaces as either drops or smears.
- On porous and/or chemically active coupon surfaces such as CARC and Silicone Rubber, MBz contaminant drops were observed to rapidly spread out from the initial drop surface area over 10 min, becoming absorbed into coupon surfaces as evidenced by a wetted surface roughly 2-3x the initial surface area that persisted for the 60 min hold time; after the 24 h hold time employed for (+)-Control 2, no MBz contaminant drops were evidenced on these surfaces. When MBz drops were applied on top of Black Rubber, the contaminant did not roll or spread out, but instead absorbed within minutes into the coupon surface as evidenced by a dry surface bulging that persisted throughout the 24 h hold time employed for (+)-Control 2.

Aging:

- Coupons were conditioned for 24 h on the laboratory countertop under normal ambient air-conditioned laboratory room temperature (25 ^{o}C) and humidity (40-60% humidity), where no extreme temperature or humidity fluctuations were noted.

Post-Aging Surface Contamination Observation:

- No significant difference in appearance, size, or color of conditioned/aged coupons were noted, indicating that no significant or unexpected swelling, shrinking, deformation, decomposition, or surface drying or wetting had occurred.

Pre-Rinse:

- No pre-rinsing step of coupons was performed. Coupons were delivered to CBI Polymers directly from JPEO in a residue-free, clean state and used as received.

- Coupons were conditioned for 24 h on the laboratory countertop under normal ambient air-conditioned laboratory RT (25 ºC) and humidity (40-60% humidity), where no extreme temperatures or humidity fluctuations were noted.

Decontamination:

- To decontaminate the contaminated coupons, after the 60 min contaminant-coupon surface hold time under an inverted Pyrex dessicator, 1.0-1.1 g of wet DeconGel 1101 at room temperature was poured evenly on top of the contaminated coupon throughout the whole surface area in an attempt to decontaminate all contaminant that might have spread throughout the coupon surface area.

- DeconGel was allowed to dry for 24 h, the dried gel was peeled from the contaminated surface, and the contaminated coupon was immediately placed in an inert polypropylene bottle pre-filled with 20 mL extraction solvent as to completely submerse the coupon in extraction solvent. The extraction bottle was screw-capped, then swirled to facilitate contaminant extraction and let to stand for 60 min. Upon sample preparation, all experiments were immediately set-up for GC/MS analytical analysis.

Post-Rinse:

- No post-rinsing step of DeconGel decontaminant was performed due to the chemical nature and intended utility of DeconGel; DeconGel is a viscous gel that upon surface contact first spreads out into a thin layer and then must air-dry for 12-24 h before it is peeled off of the contaminated surface. Rinsing either wet or dry DeconGel will result in partial to complete dissolution/emulsification of the decontaminant and most likely reduce its decontamination efficacy.

Drying:

- Upon addition of decontaminant to contaminated surfaces, DeconGel was allowed to dry for 24 h, the dried gel was peeled from the contaminated surface, and the contaminated coupon was immediately placed in an inert polypropylene bottle pre-filled with 20 mL extraction solvent as to completely submerse the coupon in the extraction solvent.

- Subsequent coupon post-washing or drying steps were not conducted or deemed necessary for this decontamination study.

Remaining Agent Measurement:

- Upon addition of 2.0 uL of MBz (experimental samples and positive control) to coupon surfaces, contaminated coupons were placed under an inverted Pyrex dessicator for 60 min, and then either placed in extraction solvent as described above (for positive control) or 1.0-1.1 g of wet DeconGel 1101 was poured evenly throughout the whole coupon surface area. DeconGel was allowed to dry for 24 h, the dried gel was peeled from the contaminated surface, and the contaminated coupon was immediately placed in an inert polypropylene bottle pre-filled with 20 mL extraction solvent to completely submerse the coupon in extraction solvent. The extraction bottle was screw-capped, then swirled to facilitate contaminant extraction and let to stand for 60 min. Upon sample/control preparation, all experiments were immediately set-up for GC/MS analytical analysis in appropriate screw-cap GC vapor-tight vials (Fisher Scientific; Fair Lawn, NJ).

Chromatographic Analysis:

- A Thermo DSQ II capillary GC-double quadrupole MS with autosampler in positive mode was used to determine MBz concentration (ppm, wt/wt) of all samples and controls using a 15 point calibration curve utilizing three independent stock solutions (Figure 2).

- A Restek (Bellefonte, PA) Rtx-5 polysiloxane capillary column was employed using Temperature Program: Initial at 45 ^{o}C, hold 1 min, Ramp at 30 ^{o}C/min to 300 ^{o}C, hold for 5 min.

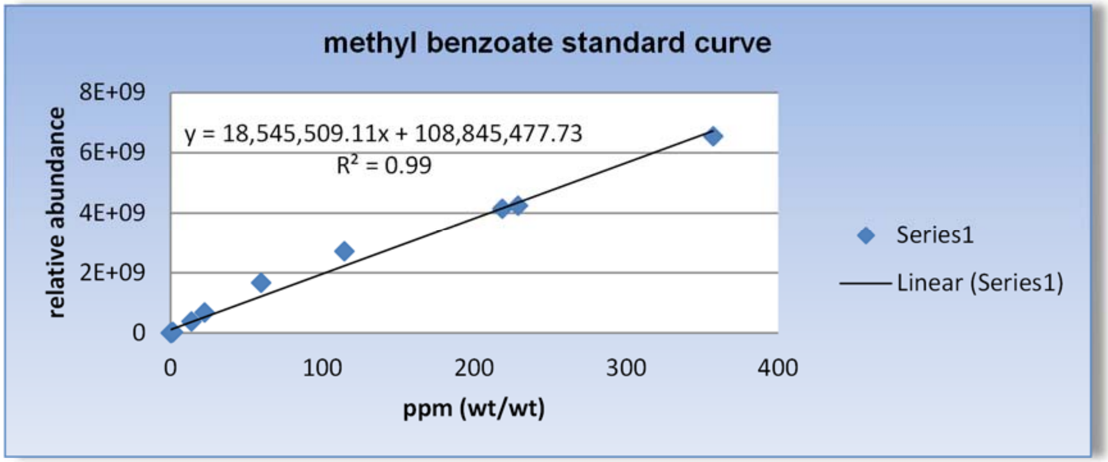
- The calibrated range for the standard calibration curve is 0.040-360 ppm (wt/wt) = 629-5659200 ng MBz.

- The limits of detection (LOD) and quantitation (LOQ) normal MS scan mode, the MS method used for the present decontamination study, are $\leq$ 40 ppb and $\leq$ 80 ppb, respectively, however in using either SIM (single-ion mode) or CI (chemical ionization using methane) MS capabilities the lowest limits of detection/quantitation of MBz approach $\leq$ 1 ppb (with

SIM), and with CI could potentially approach ≤ 1 ppt (parts per trillion) or lower.

- The calibration curve exhibits a curve fitting as approximated by the coefficient of determination of linear regression R^2, where $R^2 = 0.99$ (Figure 2).

Figure 2. MBz Standard Calibration Curve



Reporting Statements/Summary:

- Tables 1 and 2 contain the Data Test Set, and Decontamination Efficacy, Reduction in Starting Challenge Percent Efficacy, and Chemical Barrier Efficacy, respectively, for the VX simulant contaminant MBz decontamination study conducted using DeconGel 1101 as the decontaminant on a number of coupon surfaces including Cadmium-plated and Carbon Steels, CARC, and Silicone and Black Rubbers by solvent extraction.

- As seen in Table 2, when analyzing Decontamination Efficacy and reduction in starting challenge Percent Efficacy data it is noted: 1) on non-porous inert coupon surfaces such as Cadmium-plated and Carbon Steels, excellent Surface Decontamination of DeconGel 1101 on contaminant MBz was achieved ranging from 96.7 and 97.2% (on Cd-plated Steel) to 99.2 and 99.7% (on Carbon Steel) as determined by residue analysis after solvent extraction; and 2) on porous and/or chemically active coupon surfaces DeconGel 1101 decontamination efficacy and reduction in starting challenge Percent Efficacy ranged from 26.5 and 37.9% (on Black Rubber) to 62.3 and 67% (on Silicone Rubber) to 67.3 and 91.8% (on CARC) as determined by residue analysis after solvent extraction. Overall, the decontamination study using decontaminant DeconGel 1101 on MBz contaminant reveals

DeconGel's ability to both 1) encapsulate/emulsify hydrophobic contaminants such as MBz and other phosphate/carbonate esters, and 2) act as a chemical barrier against volatile agents such as MBz or other more toxic volatiles/substances that might pose a significant contact risk or possess prolonged environmental persistence.

- For porous and/or chemically active coupon surfaces such as CARC and Rubbers, modest surface decontamination of the volatile plasticizer MBz was realized; interestingly, MBz was noted to appreciably absorb into such coupon surfaces, particularly in regard to both Silicone and Black Rubber coupons, which seemed to sequester/dissolve MBz within the coupon surfaces, hindering sufficient decontaminant-contaminant interaction and limiting DeconGel 1101's abilities to encapsulate/emulsify contaminant MBz as reflected by low Decontamination and Percent Efficacies (Table 2).

- To gauge DeconGel 1101's capabilities as an effective chemical (vapor) barrier, additional experimentation ((+)-Control 2) and data compilation provided a measure of DeconGel 1101's chemical barrier capacity - termed Chemical Barrier Efficiency (see Table 2): 1) On CARC and Black and Silicone Rubber surfaces, calculated DeconGel 1101 Chemical Barrier Efficiencies indicates a significant chemical surface barrier capability for DeconGel 1101 when applied to porous, chemically active rubber/ epoxy/polyester surfaces able to absorb and entrain contaminants; and 2) On non-porous, chemically inert Cd- and Carbon Steel surfaces, due to MBz's significant volatility, contaminant applied to the steel coupons was not detectable after undergoing a 24-hour coupon-contaminant incubation as implemented for positive controls ((+)-Control 2). As such, Chemical Barrier Efficacies for Cd- and Carbon Steel surfaces could not be determined.

- The 60 min contact time between MBz contaminant and all coupons tested was deemed necessary, however adoption of a shorter contact time holds promise to allow the required interaction between contaminant and coupon surface while minimizing loss of volatile contaminant MBz to evaporation. Utilizing an inverted Pyrex dessicator to store contaminated coupons immediately after contamination and during the required 60 min contact time aided in retaining significant amounts of contaminant on/within the coupon surfaces.

- Since live agents may not exhibit the same bioactivity, volatility, or absorption characteristics as MBz, live agent testing is recommended to accurately measure DeconGel effectiveness on test surfaces.

APPLICATION INSTRUCTIONS FOR END-USERS

Use product directly as is from container. DO NOT DILUTE. Masking or painters tape can be applied along one edge of the area that is to be decontaminated to aid creating a peeled edge to grip for peeling the dried film. Apply DeconGel using a paint brush, a trowel, a handheld sprayer, or an industrial grade sprayer (use DeconGel 1120 or 1121 for spray application).

The thickness of the gel and the number of coats is dictated by the surface to be decontaminated. Coating thickness required for good peel characteristics varies with substrate and generally increases with substrate porosity. It is recommended that first time customers test DeconGel on a small sample area to confirm the required film thickness and dry time for their specific application. If the film is difficult to peel, please apply an additional coat. A razor blade is useful to start the peel. Lay the blade nearly flat and fillet the edge of the film to create a tab that can be pulled. For surfaces that the gel adheres to well, such as concrete, 12” – 24” strips can be cut in the film resulting in less force being required to peel the film.

- Let DeconGel dry for 24 hours

Dry time will vary depending on humidity, temperature, air flow and thickness of the DeconGel. This can take from as little time as an hour for thin coats in a dry environment with plenty of airflow, to overnight or longer if thicker coats are applied in humid environments. Dry times exceeding 24 hours may sometimes be required for good peel performance on bare concrete, wood and other highly porous substrates and substrates with deep cracks or grooves. However, 18-24 hrs is often sufficient dry time on good quality concrete. It is recommended that users test a small area to determine drying time prior to applying DeconGel for an entire job. Supplemental heat or air circulation will accelerate DeconGel's drying time for any job.

- Peel DeconGel off the surface by starting from one of the edges



When dry, the product locks the contaminants into a polymer matrix. The film containing the encapsulated contamination can then be peeled. DeconGel peels from most non-porous and porous hard surfaces if the dried film is thick enough. If the film is difficult to peel, add another coat, let dry, and peel. In most cases the DeconGel will come off in a single sheet but for odd shaped surfaces you may be required to score DeconGel in order to be able to peel it off.

- Dispose of the dried DeconGel in accordance with the local, state and Federal disposal regulations of the contaminant/substance you are removing. DeconGel itself has no special disposal restrictions.

For more information on application techniques visit our website: www.decongel.com

7.22. End-User Report for Jet Fuel

TITLE: Surface Decontamination of Jet Fuel (Jet A) by DeconGel 1101 and 1102

ANALYST: Geoff Nadolski PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVES: Surface decontamination determination of DeconGel formulations 1101 and 1102 on stainless steel, aluminum, and concrete surfaces contaminated with Jet Fuel using GC/MS (Gas Chromatography/Mass Spectrometry) following Environmental Protection Agency (EPA) SW-846 Methods 3500C (Sampling) and 8270C (Analysis).

HAZARDOUS MATERIALS RELEVANCE: Jet Fuel (Jet A) is a C8-C16 kerosene-type flammable fuel. Jet Fuel vapors and liquid are harmful, ingestion of sufficient quantities can be fatal, therefore, Jet Fuel use requires adopting safe and proper handling, storage, and disposal procedures.

SUMMARY RESULTS:

- As seen in Table 1, excellent surface decontamination was achieved by applying DeconGel 1101 and 1102 both via brushing or pouring (non-brushed) onto contaminated surfaces, resulting in encapsulation of Jet Fuel contaminant by DeconGel's active components. Decontamination efficacies of poured DeconGel 1101 ranged from 90.3% (on concrete) to 94.4% (on stainless steel) to 94.5% (on aluminum), brushed DeconGel 1101 ranged from 98.7% (on concrete) to 99.2% (on stainless steel) to 99.4% (on aluminum), as determined by residual swipe analysis. Decontamination efficacies of poured DeconGel 1102 ranged from 89.2% (on concrete) to 92.7% (on stainless steel) to 92.9% (on aluminum), brushed DeconGel 1102 ranged from 98.9% (on concrete) to 99.4% (on stainless steel) to 99.4% (on aluminum), as determined by residual swipe analysis.
- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of organics in polar solvent solvated samples. When necessary, the digestion methods were customized to result in the complete dissolution of the organic contaminants and to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Table 1 shows the decontamination efficacies of DeconGel 1101 on stainless steel, aluminum, and concrete surfaces contaminated with Jet Fuel as determined by residual swipe testing.

Table 1. Decontamination efficacies of DeconGel 1101 and 1102 on Jet Fuel (Jet A) contaminated stainless steel, aluminum, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		Formulation	Formulation
		DeconGel 1101	**DeconGel 1102**
Stainless Steel	Control	221.65 ± 1.23	221.65 ± 1.23
	Residual (non-brushed)	12.42 ± 0.47	16.19 ± 0.54
	Residual (brushed)	1.70 ± 0.02	1.30 ± 0.02
	Decon. Efficacy (non-brushed) (%)	**94.4 ± 0.22**	**92.7 ± 0.43**
	Decon. Efficacy (brushed) (%)	**99.2 ± 0.12**	**99.4 ± 0.15**
Aluminum	Control	219.92 ± 1.40	219.92 ± 1.40
	Residual (non-brushed)	12.09 ± 0.74	15.56 ± 0.55
	Residual (brushed)	1.70 ± 0.01	1.32 ± 0.04
	Decon. Efficacy (non-brushed) (%)	**94.5 ± 0.26**	**92.9 ± 0.16**
	Decon. Efficacy (brushed) (%)	**99.4 ± 0.15**	**99.4 ± 0.15**
Concrete	Control	165.20 ± 1.22	165.20 ± 1.22
	Residual (non-brushed)	16.06 ± 0.45	17.85 ± 0.59
	Residual (brushed)	2.18 ± 0.10	1.83 ± 0.10
	Decon. Efficacy (non-brushed) (%)	**90.3 ± 0.29**	**89.2 ± 0.29**
	Decon. Efficacy (brushed) (%)	**98.7 ± 0.10**	**98.9 ± 0.17**

236x dilution factor for samples and controls

NOTES:

- Application of a homogenous, thin layer of Jet Fuel contaminant on the respective substrate facilitated an optimized interaction between contaminant and DeconGel, and provided an accurate measure of DeconGel's decontamination efficacy in a scaled-down yet real-world setting. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel.
- ASTM method E1728-03, a standardized swipe testing method used for sampling of contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. Air-dried GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes wetted with methanol/DMSO (1:1) solvent were utilized in this swipe testing method.
- Standardized EPA SW-846 Sampling Method 3500C "Organic Extraction and Sample Preparation" was followed as a guideline to prepare all samples and controls. All samples, controls, and standards were prepared using the same solvent and appropriate experimental conditions to ensure accurate and GC/MS instrumental analysis.

- GC/MS instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of organic compounds. Standardized EPA SW-846 Analytical Method 8270C "Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)" was followed as a guideline to prepare all samples and controls.
- To ensure accurate determination of DeconGel decontamination efficacy, a standard curve of the analyte of interest was prepared using sufficiently pure analyte; the respective standards were diluted to a known concentration (ppm) using the same solvent as used for samples and controls.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.20 g Jet Fuel contaminant was evenly applied via brushing on 1) aluminum (surface area: 56.3 cm^2), 2) stainless steel (surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 or 1102 was either poured or brushed (brushed first in a top-bottom, then in a left-right fashion) onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC) wetted with methanol:DMSO (1:1) solvent (2 mL). Swipe and dried film samples were suspended in 50 mL methanol:DMSO (1:1) for 24 h. Samples were then analyzed via GC/MS (see below).

Control Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied via brushing on 1) aluminum (surface area: 56.3 cm^2), 2) stainless steel (surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC) wetted with methanol:DMSO (1:1) solvent (2 mL). Swipe and dried film samples were suspended in 50 mL methanol:DMSO (1:1) for 24 h. Samples were then analyzed via GC/MS (see below).

Reagents and Standards

Jet Fuel (Jet A; sourced Oahu, Hawaii) was used as received.

Analytical Instrumentation

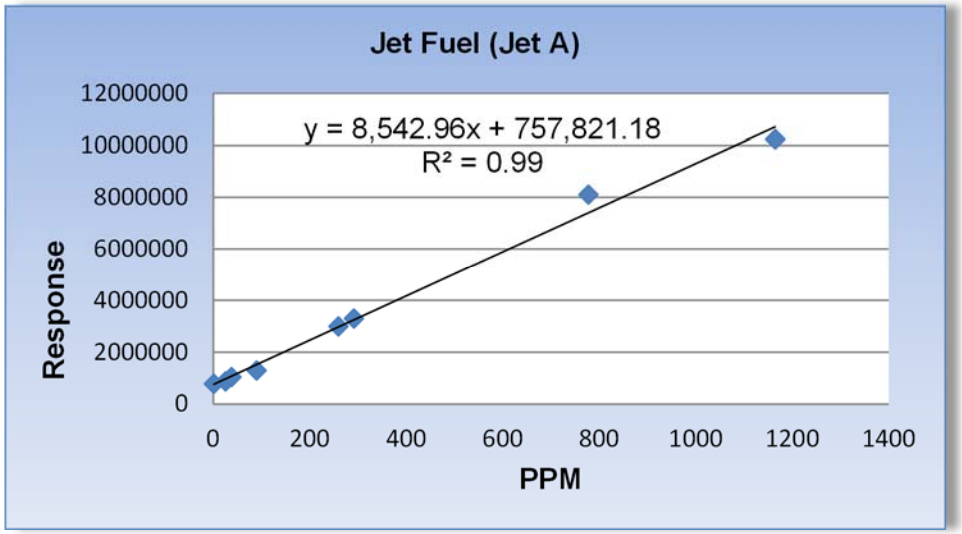
A Thermo DSQII GC/MS with autosampler in positive mode was used to determine Jet Fuel concentration (ppm, wt/wt) of all samples and controls, using a Restek (Bellefonte, PA) Rtx-5 capillary column (30 m x 0.25 mm, 0.5 um).

A 8-point standard curve derived from three independently prepared stock solutions was prepared using methanol:DMSO (1:1) as the working solvent. The calibration curve exhibits a curve fitting as approximated by the coefficient of determination of linear regression R^2, where $R^2 = 0.99$ (see Figure 1).

GC method: start at 45 °C, hold for 7 min, ramp at 30 °C/min to 320 °C, hold 10 min.

Jet Fuel (Jet A) GC/MS data: 11.0 min; $M^+ = 100$ (C_7H_{16}), 114 (C_8H_{18})

Figure 1. Jet Fuel (Jet A) Standard Calibration Curve



APPLICATION INSTRUCTIONS FOR END-USERS

Use product directly as is from container. DO NOT DILUTE. Masking or painters tape can be applied along one edge of the area that is to be decontaminated to aid creating a peeled edge to grip for peeling the dried film. Apply DeconGel using a paint brush, a trowel, a handheld sprayer, or an industrial grade sprayer (use DeconGel 1120 or 1121 for spray application).

The thickness of the gel and the number of coats is dictated by the surface to be decontaminated. Coating thickness required for good peel characteristics varies with substrate and generally increases with substrate porosity. It is recommended that first time customers test DeconGel on a small sample area to confirm the required film thickness and dry time for their specific application. If the film is difficult to peel, please apply an additional coat. A razor blade is useful to start the peel. Lay the blade nearly flat and fillet the edge of the film to create a tab that can be pulled. For surfaces that the gel adheres to well, such as concrete, 12" – 24" strips can be cut in the film resulting in less force being required to peel the film.

- Let DeconGel dry for 24 hours

Dry time will vary depending on humidity, temperature, air flow and thickness of the DeconGel. This can take from as little time as an hour for thin coats in a dry environment with plenty of airflow, to overnight or longer if thicker coats are applied in humid environments. Dry times exceeding 24 hours may sometimes be required for good peel performance on bare concrete, wood and other highly porous substrates and substrates with deep cracks or grooves. However, 18-24 hrs is often sufficient dry time on good quality concrete. It is recommended that users test a small area to determine drying time prior to applying DeconGel for an entire job. Supplemental heat or air circulation will accelerate DeconGel's drying time for any job.

- Peel DeconGel off the surface by starting from one of the edges

When dry, the product locks the contaminants into a polymer matrix. The film containing the encapsulated contamination can then be peeled. DeconGel peels from most non-porous and porous hard surfaces if the dried film is thick enough. If the film is difficult to peel, add another coat, let dry, and peel. In most cases the DeconGel will come off in a single sheet but for odd shaped surfaces you may be required to score DeconGel in order to be able to peel it off.

- Dispose of the dried DeconGel in accordance with the local, state and Federal disposal regulations of the contaminant/substance you are removing. DeconGel itself has no special disposal restrictions.

For more information on application techniques visit our website: www.decongel.com

7.23. End-User Report for Lead Compounds (Lead (II) Oxide, Lead (II) Sulfate)

TITLE: Surface Decontamination of Lead Compounds (Lead (II) Oxide (PbO), Lead (II) Sulfate ($PbSO_4$)) by DeconGel 1101

ANALYSTS: Geoff Nadolski PhD, Senior Scientist and Andreas Mylonakis PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel formulation 1101 on stainless steel, aluminum, and concrete surfaces contaminated with Lead (PbO, $PbSO_4$) using ICP-OES (Inductively Coupled Plasma-Optical Emission Spectroscopy) following Environmental Protection Agency (EPA) SW-846 Methods 3005A (sampling) and 6010C (analysis).

HAZARDOUS MATERIALS RELEVANCE: Lead compounds are used in batteries, bullets, as part of solders and alloys, and as a radiation shields. Specifically, lead (II) oxide is used in the production of ceramic glazes and leaded glass, and vulcanized rubber; lead (II) sulfate is commonly used as a component of battery electrodes. Lead and its compounds accumulate in soft tissue and bone, and are potent neurotoxins. The lead compounds PbO and $PbSO_4$ were chosen as representative lead compounds for evaluating DeconGel's efficacy; DeconGel is expected to have similar efficacy towards the wide range of lead compounds.

SUMMARY RESULTS:

- As seen in Tables 1 and 2, excellent to acceptable surface decontamination was achieved by applying DeconGel 1101 onto contaminated surfaces, resulting in encapsulation of Lead contaminants by DeconGel's active components. Decontamination efficacies of DeconGel 1101 on lead (II) oxide ranged from 81.2% (on aluminum) to 91.2% (on concrete) to 91.5% (on stainless steel) as determined by residual swipe analysis; decontamination efficacies of DeconGel 1101 on lead (II) sulfate ranged from 99.4% (on stainless steel) to 99.8% (on concrete) to 99.8% (on aluminum) as determined by residual swipe analysis

- Lead (II) oxide is very hygroscopic and is evidenced to react with trace amounts of water (on or within both non-porous and porous test surfaces), forming a fixed residue that was not able to be completely removed by DeconGel. Nevertheless, DeconGel showed acceptable decontamination efficacy of loose lead (II) oxide contamination from such surfaces.

- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of inorganic compounds in aqueous samples. When deemed necessary, experimental methods were customized to afford complete dissolution of inorganic contaminants, and to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Tables 1 and 2 show the decontamination efficacies of DeconGel 1101 on stainless steel, aluminum, and concrete surfaces contaminated with lead compounds (PbO, $PbSO_4$) as determined by residual swipe testing.

Table 1. Decontamination efficacies of DeconGel 1101 on lead (II) oxide contaminated stainless steel, aluminum, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		**Formulation**
		DeconGel 1101
Stainless Steel*	Control	456.7 ± 15.6
	Residual	39.0 ± 17.3
	Decon. Efficacy (%)	**91.5 ± 5.0**
Aluminum*	Control	433.9 ± 39.0
	Residual	81.4 ± 7.4
	Decon. Efficacy (%)	**81.2 ± 7.4**
Concrete*	Control	405.3 ± 35.6
	Residual	35.5 ± 13.5
	Decon. Efficacy (%)	**91.2 ± 8.7**

2000x dilution factor for samples and controls

* Lead (II) oxide is very hygroscopic and reacts with trace amounts of water (on or within both non-porous and porous test surfaces), resulting in a fixed residue on the contaminated surface that could not be completely removed by DeconGel.

Table 2. Decontamination efficacies of DeconGel 1101 on lead (II) sulfate contaminated stainless steel, aluminum, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		**Formulation**
		DeconGel 1101
Stainless Steel	Control	532.4 ± 14.7
	Residual	3.32 ± 4.2
	Decon. Efficacy (%)	**99.4 ± 3.5**
Aluminum	Control	539.0 ± 4.6
	Residual	0.984 ± 0.15
	Decon. Efficacy (%)	**99.8 ± 8.9**
Concrete	Control	560.4 ± 3.9
	Residual	1.13 ± 0.22
	Decon. Efficacy (%)	**99.8 ± 7.6**

2000x dilution factor for samples and controls

NOTES:

- Application of a homogenous, thin layer of Lead contaminants on the respective substrate facilitated an optimized interaction between contaminant and DeconGel, and provided an accurate measure of DeconGel's decontamination efficacy in a scaled-down yet real-world setting. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel. Lead (II) oxide and sulfate readily dissolve in aqueous acidic solutions used to prepare all samples and controls (15% HCl, 15% HNO_3, 70% DI H_2O).

- Lead (II) oxide undergoes a chemical reaction with trace amounts of water on or within both non-porous and porous test surfaces which prevents some contamination from being sampled (swipe testing) and encapsulated into dry DeconGel (direct gel testing). Nevertheless DeconGel showed acceptable decontamination efficacy against loose lead (II) oxide contamination.

- ASTM method E1728-03, a standardized swipe testing method used for sampling of inorganic contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes (pre-wetted with DI H_2O) were utilized in this swipe testing method.

- Standardized EPA SW-846 Sampling Method 3005A "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy" was followed as a guideline to prepare all samples and controls. When deemed necessary, digestion methods were customized by increasing hydrochloric and nitric acid concentrations from 15% to 25% wt, and/or by heating samples to higher temperatures using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC) to afford the complete digestion of the inorganic contaminants. All samples, controls, and standards were prepared using the same dissolution solution and experimental conditions to ensure both correct instrument calibration and accurate analytical results.

- ICP-OES instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of inorganic compounds. Standardized EPA SW-846 Analytical Method 6010C "Inductively Coupled Plasma-Atomic Emission Spectrometry" was followed as a guideline to prepare all samples and controls.
- To ensure accurate determination of DeconGel decontamination efficacy, calibration standards of the analyte of interest were prepared using either a sufficiently pure analyte or an appropriate ICP-MS Standard (Ricca Chemical Company; Arlington, TX); the respective standards were diluted to a known concentration (ppm) using the same digestion method as the one used for samples and controls. Instrument blank controls were DI H_2O ($\geq$17 M-Ohm).

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.050 g lead (II) oxide or lead (II) sulfate contaminants were evenly applied on 1) stainless steel (surface area: 56.3 cm^2), 2) aluminum (commercial grade, surface area: 100 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe and gel samples were suspended in 100 mL aqueous acidic solution (15% HCl, 15% HNO_3, 70% deionized (DI) H_2O) for 24 h. When deemed necessary, samples were heated to 94^{o}C for 4-24 h to effectively complete dissolution of inorganics using a HotBlock™ Sample Heater (Environmental Express; Mt. Pleasant, SC). Samples were allowed to cool to room temperature and were then analyzed via ICP-OES (see below).

Controls Methods

For Swipe Controls samples, a respectable amount of contaminant was evenly applied on 1) stainless steel (56.3 cm^2), 2) aluminum (commercial grade, 100 cm^2), or 3) concrete (industrial grade, 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using a GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC). Swipe samples were suspended in 100 mL aqueous acidic solution (15% HCl, 15% HNO_3, 70% DI H_2O) for 24 h and analyzed via ICP-OES (see below).

Reagents and Standards

Reagent grade Lead (II) Oxide, PbO, (CAS# 1317-36-8, Fisher Scientific; Fair Lawn, NJ) and Lead (II) Sulfate, $PbSO_4$, (CAS# 7446-14-2, Fisher Scientific; Fair Lawn, NJ) were used as received.

1000 ppm calibration standards were prepared using reagent grade PbO and $PbSO_4$ in freshly prepared aqueous acidic solution (15% HCl, 15% HNO_3, 70% DI H_2O). DI H_2O was used as the blank sample.

Analytical Instrumentation

A Thermo ICP-OES instrument model radial iCap 6300 was used to determine lead compound concentration (ppm) of all samples and controls using a freshly prepared 1000 ppm calibration standard.

Analyte (lead) analyzed at 220.4 nm

Pump Speed: 0.5 mL/min

APPLICATION INSTRUCTIONS FOR END-USERS

Use product directly as is from container. DO NOT DILUTE. Masking or painters tape can be applied along one edge of the area that is to be decontaminated to aid creating a peeled edge to grip for peeling the dried film. Apply DeconGel using a paint brush, a trowel, a handheld sprayer, or an industrial grade sprayer (use DeconGel 1120 or 1121 for spray application).

The thickness of the gel and the number of coats is dictated by the surface to be decontaminated. Coating thickness required for good peel characteristics varies with substrate and generally increases with substrate porosity. It is recommended that first time customers test DeconGel on a small sample area to confirm the required film thickness and dry time for their specific application. If the film is difficult to peel, please apply an additional coat. A razor blade is useful to start the peel. Lay the blade nearly flat and fillet the edge of the film to create a tab that can be pulled. For surfaces that the gel adheres to well, such as concrete, 12" – 24" strips can be cut in the film resulting in less force being required to peel the film.

- Let DeconGel dry for 24 hours

Dry time will vary depending on humidity, temperature, air flow and thickness of the DeconGel. This can take from as little time as an hour for thin coats in a dry environment with plenty of airflow, to overnight or longer if thicker coats are applied in humid environments. Dry times exceeding 24 hours may sometimes be required for good peel performance on bare concrete, wood and other highly porous substrates and substrates with deep cracks or grooves. However, 18-24 hrs is often sufficient dry time on good quality concrete. It is recommended that users test a small area to determine drying time prior to applying DeconGel for an entire job. Supplemental heat or air circulation will accelerate DeconGel's drying time for any job.

- Peel DeconGel off the surface by starting from one of the edges

When dry, the product locks the contaminants into a polymer matrix. The film containing the encapsulated contamination can then be peeled. DeconGel peels from most non-porous and porous hard surfaces if the dried film is thick enough. If the film is difficult to peel, add another coat, let dry, and peel. In most cases the DeconGel will come off in a single sheet but for odd shaped surfaces you may be required to score DeconGel in order to be able to peel it off.

- ➢ Dispose of the dried DeconGel in accordance with the local, state and Federal disposal regulations of the contaminant/substance you are removing. DeconGel itself has no special disposal restrictions.

For more information on application techniques visit our website: www.decongel.com

7.24. End-User Report for Mercury (Elemental)

TITLE: Surface Decontamination of Mercury (elemental) by DeconGel 1101

ANALYSTS: Geoff Nadolski PhD, Senior Scientist and Andreas Mylonakis PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVE: Surface decontamination determination of DeconGel formulation 1101 on stainless steel, porcelain tile, composite tile (unwaxed), linoleum tile, and concrete surfaces contaminated with mercury (elemental) using Mercury Vapor Analysis Sensor (Mercury Tracker 3000) following Environmental Protection Agency (EPA) SW-846 Method 7471B: "Mercury in Solid or Semisolid Waste (Manual Cold-Vapor Technique)".

HAZARDOUS MATERIALS RELEVANCE: Mercury is used in thermometers, electronics, lighting, gold and silver refinery, and combined with other metals to form useful amalgams. Mercury and most of its compounds are extremely toxic, causing neurological damage due to inhalation of vapors/dust or ingestion.

SUMMARY RESULTS:

- As seen in Table 1, excellent to acceptable surface decontamination was achieved by applying DeconGel 1101 onto contaminated surfaces, resulting in encapsulation of mercury (elemental) by DeconGel's active components. Decontamination efficacies of poured DeconGel 1101 ranged from 66.0% (composite tile, using Zn-controlled contaminant loading) to 85.9% (porcelain tile, using Zn-controlled contaminant loading) to 90.8% (linoleum tile, using Zn-controlled contaminant loading) to 91.7% (composite tile, using pipet-controlled contaminant loading) to 95.4% (linoleum tile, using pipet-controlled contaminant loading) to 97.8% (concrete, using pipet-controlled contaminant loading) to 98.4% (stainless steel, using pipet-controlled contaminant loading) to 99.2% (porcelain tile, using pipet-controlled contaminant loading); decontamination efficacy of brushed DeconGel 1101 was 99.0% (linoleum tile, using Zn-controlled contaminant loading).

- Mercury (elemental) was evidenced to interact with most plastic surfaces such as un-waxed composite and linoleum tiles, adhering to the plastic surfaces and forming a fixed residue that was not able to be completely removed by DeconGel. Nevertheless, DeconGel showed excellent to acceptable decontamination efficacy of loose mercury (small beads <1 mm diameter) contamination from such surfaces (see Table 1).

- Optimized experimental and analytical methods were successfully developed following a standardized EPA analysis method as a guideline for determination of elemental mercury vapor on solid surfaces. When deemed necessary, experimental methods were customized to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Table 1 shows the decontamination efficacies of DeconGel 1101 on stainless steel, concrete, porcelain tile, composite tile, and linoleum tile surfaces as determined by mercury vapor analysis.

Table 1. Decontamination efficacies of DeconGel 1101 on Mercury (elemental) contaminated stainless steel, concrete, porcelain tile, composite tile, and linoleum tile surfaces as determined by mercury vapor analysis.

Mercury Vapor Analysis Testing (ppt)		Formulation
		DeconGel 1101
Stainless Steel	Control [1]	18.6 ± 3.0
	Residual [1]	0.3 ± 0.12
	Decon. Efficacy (%) [1]	**98.4 ± 0.7**
Concrete	Control [1]	18.6 ± 8.9
	Residual [1]	0.4 ± 0.13
	Decon. Efficacy (%) [1]	**97.8 ± 0.7**
Porcelain Tile*	Control [1]	12.2 ± 1.5
	Control [2]	6.4 ± 1.2
	Residual [1]	0.1 ± 0.04
	Residual [2]	0.9 ± 0.6
	Decon. Efficacy (%) [1]	**99.2 ± 0.4**
	Decon. Efficacy (%) [2]	**85.9 ± 11.7**
Composite Tile*	Control [1]	72.1 ± 39.1
	Control [2]	5.0 ± 2.7
	Residual [1]	6.0 ± 1.4
	Residual [2]	1.7 ± 0.8
	Decon. Efficacy (%) [1]	**91.7 ± 2.1**
	Decon. Efficacy (%) [2]	**66.0 ± 11.7**
Linoleum Tile*	Control [1]	60.4 ± 6.6
	Control [2]	20.7 ± 10.0
	Control [3]	20.7 ± 10.0
	Residual [1]	2.8 ± 0.5
	Residual [2]	1.9 ± 1.1
	Residual [3]	0.2 ± 0.1
	Decon. Efficacy (%) [1]	**95.4 ± 0.9**
	Decon. Efficacy (%) [2]	**90.8 ± 5.8**
	Decon. Efficacy (%) [3]	**99.0 ± 0.2**

* Mercury (elemental) has an affinity for plastics, mercury in the form of small beads was noted to adhere to plastic test surfaces (composite tile and linoleum tile), resulting in a fixed residue on the contaminated surface that could not be completely removed by DeconGel.

[1] Contaminant administration controlled using pipet-aided removal of bulk mercury contamination to yield contaminant as very small liquid beads (<1 mm diameter); DeconGel was poured onto contaminated surface

[2] Contaminant administration controlled using zinc dust-assisted removal of bulk mercury contamination to yield contaminant as very small liquid beads (<1 mm diameter); DeconGel was poured onto contaminated surface

[3] Contaminant administration controlled using zinc dust-assisted removal of bulk mercury liquid to yield contaminant as very small liquid beads (<1 mm diameter); DeconGel was brushed onto contaminated surface

NOTES:

- Application of homogeneously dispersed mercury (elemental) contaminant in the form of very small beads (<1 mm diameter) on the respective substrates facilitated an optimized interaction between contaminant and

DeconGel, and provided an accurate measure of DeconGel's decontamination efficacy in a scaled-down yet real-world setting. Contaminant administration was controlled using either 1) pipet-aided test surface spreading and then removal of bulk mercury liquid (approximately 0.25 g) initially loaded onto test surfaces, or 2) zinc dust-assisted test surface spreading and then removal (using mercury removal kit, Lab Safety Supply Inc., Janesville, WI) of bulk mercury liquid (approximately 0.25 g) initially loaded onto test surfaces, to yield mercury contaminant in the form of very small liquid beads (<1 mm diameter) spread homogeneously throughout the test surfaces. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel. Mercury (elemental) vapor was analyzed using a mercury vapor analyzer (see below).

- Mercury (elemental) was evidenced to interact with plastic surfaces such as coated porcelain, composite, and linoleum tiles, adhering to the plastic surfaces and forming a fixed residue that was not able to be completely removed by DeconGel. Nevertheless, DeconGel showed excellent to acceptable decontamination efficacy of loose mercury (small beads <1 mm diameter) contamination from such surfaces, as determined using a mercury vapor analyzer (see below).
- Standardized EPA SW-846 Sampling Method 7471B: "Mercury in Solid or Semisolid Waste (Manual Cold-Vapor Technique)" was followed as a guideline to prepare all samples and controls, to ensure both accurate and precise analytical testing results.
- Mercury Vapor Analysis using the Mercury Tracker 3000 (rented from Mercury Instruments USA; Littleton, CO) allows for the continuous measurement of mercury (elemental) concentration (parts per trillion (ppt)) in ambient air, instrument determination of mercury concentration utilizes mercury resonance absorption of 253.7 nm. The Mercury Tracker 3000 contains an internalized mercury lamp used for instrument calibration, and during use the instrument routinely performs instrument calibration to provide accurate analytical determination.

CALCULATIONS:

Decontamination Efficacy (Mercury Vapor Testing) =

[(Contaminant (ppt) of Control) – (Contaminant (ppt) of Residual)/Contaminant (ppt) of Control] x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, approximately 0.25 g of mercury (elemental) was pipetted onto 1) stainless steel (surface area: 56.3 cm^2), 2) plastic-coated porcelain tile (surface area: 48.8 cm^2), 3) non-waxed composite tile (surface area: 48.8 cm^2), 4) linoleum tile (surface area: 48.8 cm^2), or 5) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Contaminant administration was controlled using either 1) pipet-aided test surface spreading and then removal of bulk mercury contamination initially loaded onto test surfaces, or 2) zinc dust-assisted test surface spreading and then removal (using mercury removal kit, Lab Safety Supply Inc., Janesville, WI) of bulk mercury liquid initially loaded onto test surfaces, to yield mercury contaminant in the form of very small liquid beads (<1 mm diameter) spread homogeneously throughout the test surfaces Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was analyzed for mercury vapor using the Mercury Tracker 3000 (Mercury Instruments USA; Littleton, CO) (see below).

Control Method

For Control samples, a respectable amount of contaminant (see above in Sample Method) was evenly applied on 1) stainless steel (56.3 cm^2), 2) plastic-coated porcelain tile (surface area: 48.8 cm^2), 3) non-waxed composite tile (surface area: 48.8 cm^2), 4) linoleum tile (surface area: 48.8 cm^2), or 5) concrete (industrial grade, surface area: 56.3 cm^2) coupons and the surface was analyzed for mercury vapor using the Mercury Tracker 3000 (Mercury Instruments USA; Littleton, CO) (see below).

Reagents and Standards

Mercury (elemental) (CAS# 7439-97-6, Fisher Scientific; Fair Lawn, NJ) was used as received.

Analytical Instrumentation

A Mercury Tracker 3000 (rented from Mercury Instruments USA; Littleton, CO) was used to determine mercury (elemental) surface vapor concentration (ppt) of all samples and controls. Precise vapor measurements were conducted using a hand-held open-ended wand connected to the instrument detector, such that the wand was passed just above (<0.5 cm) the contaminated surface at a 45 degree, passing the wand first in a top-bottom, then in a left-right fashion.

APPLICATION INSTRUCTIONS FOR END-USERS

Use product directly as is from container. DO NOT DILUTE. Masking or painters tape can be applied along one edge of the area that is to be decontaminated to aid creating a peeled edge to grip for peeling the dried film. Apply DeconGel using a paint brush, a trowel, a handheld sprayer, or an industrial grade sprayer (use DeconGel 1120 or 1121 for spray application).

The thickness of the gel and the number of coats is dictated by the surface to be decontaminated. Coating thickness required for good peel characteristics varies with substrate and generally increases with substrate porosity. It is recommended that first time customers test DeconGel on a small sample area to confirm the required film thickness and dry time for their specific application. If the film is difficult to peel, please apply an additional coat. A razor blade is useful to start the peel. Lay the blade nearly flat and fillet the edge of the film to create a tab that can be pulled. For surfaces that the gel adheres to well, such as concrete, 12" – 24" strips can be cut in the film resulting in less force being required to peel the film.

- Let DeconGel dry for 24 hours

Dry time will vary depending on humidity, temperature, air flow and thickness of the DeconGel. This can take from as little time as an hour for thin coats in a dry environment with plenty of airflow, to overnight or longer if thicker coats are applied in humid environments. Dry times exceeding 24 hours may sometimes be required for good peel performance on bare concrete, wood and other highly porous substrates and substrates with deep cracks or grooves. However, 18-24 hrs is often sufficient dry time on good quality concrete. It is recommended that users test a small area to determine drying time prior to applying DeconGel for an entire job. Supplemental heat or air circulation will accelerate DeconGel's drying time for any job.

- Peel DeconGel off the surface by starting from one of the edges

When dry, the product locks the contaminants into a polymer matrix. The film containing the encapsulated contamination can then be peeled. DeconGel peels from most non-porous and porous hard surfaces if the dried film is thick enough. If the film is difficult to peel, add another coat, let dry, and peel. In most cases the DeconGel will come off in a single sheet but for odd shaped surfaces you may be required to score DeconGel in order to be able to peel it off.

- Dispose of the dried DeconGel in accordance with the local, state and Federal disposal regulations of the contaminant/substance you are removing. DeconGel itself has no special disposal restrictions.

For more information on application techniques visit our website: www.decongel.com

7.25. End-User Report for Motor Oil

TITLE: Surface Decontamination of Motor Oil by DeconGel 1101 and 1102

ANALYST: Geoff Nadolski PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVES: Surface decontamination determination of DeconGel formulation 1101 and 1102 on aluminum, stainless steel, and concrete surfaces contaminated with Motor Oil (polyaromatic hydrocarbons fraction) using LC/MS (Liquid Chromatography/Mass Spectrometry) following Environmental Protection Agency (EPA) SW-846 Methods 3500C (Sampling), 8321B (Analysis), and 1654A (Analysis of PAH Content of Oil).

HAZARDOUS MATERIALS RELEVANCE: Motor oil is used throughout the world as a combustion engine lubricant. Motor oil is derived from petroleum-based materials including crude oil, and is composed of a variety of saturated and unsaturated hydrocarbons including polyaromatic hydrocarbons (PAHs) (up to 6%). PAHs are widespread organic pollutants known for their carcinogenic, mutagenic, and tetratogenic properties.

SUMMARY RESULTS:

- As seen in Table 1, excellent surface decontamination was achieved by applying DeconGel 1101 and 1102 both via brushing or pouring (non-brushed) onto contaminated surfaces, resulting in encapsulation of Motor oil contaminant by DeconGel's active components. Decontamination efficacies of poured DeconGel 1101 ranged from 87.2% (on concrete) to 96.2% (on aluminum) to 96.2% (on stainless steel), brushed DeconGel 1101 ranged from 98.0% (on concrete) to 99.4% (on stainless steel) to 99.4% (on aluminum), as determined by residual swipe analysis. Decontamination efficacies of poured DeconGel 1102 ranged from 85.6% (on concrete) to 95.3% (on stainless steel) to 95.1% (on aluminum), brushed DeconGel 1102 ranged from 98.5% (on concrete) to 99.5% (on stainless steel) to 99.5% (on aluminum), as determined by residual swipe analysis.
- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of organics in polar solvent solvated samples. When necessary, the digestion methods were customized to result in the complete dissolution of the organic contaminants and to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Table 1 shows the decontamination efficacies of DeconGel 1101 on stainless steel, aluminum, and concrete surfaces contaminated with Motor oil as determined by residual swipe testing.

Table 1. Decontamination efficacies of DeconGel 1101 and 1102 on Motor Oil contaminated stainless steel, aluminum, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		**Formulation**	**Formulation**
		DeconGel 1101	**DeconGel 1102**
Stainless Steel	Control	283.30 ± 2.38	283.30 ± 2.38
	Residual (non-brushed)	10.84 ± 0.49	13.26 ± 0.49
	Residual (brushed)	1.70 ± 0.10	1.33 ± 0.10
	Decon. Efficacy (non-brushed) (%)	**96.2 ± 0.16**	**95.3 ± 0.16**
	Decon. Efficacy (brushed) (%)	**99.4 ± 0.12**	**99.5 ± 0.12**
Aluminum	Control	282.06 ± 2.82	282.06 ± 2.82
	Residual (non-brushed)	10.67 ± 0.12	13.13 ± 0.53
	Residual (brushed)	1.71 ± 0.15	1.40 ± 0.04
	Decon. Efficacy (non-brushed) (%)	**96.2 ± 0.16**	**95.3 ± 0.10**
	Decon. Efficacy (brushed) (%)	**99.4 ± 0.16**	**99.5 ± 0.10**
Concrete	Control	204.91 ± 1.64	204.91 ± 1.64
	Residual (non-brushed)	26.23 ± 1.58	29.51 ± 1.11
	Residual (brushed)	4.11 ± 0.19	3.09 ± 0.28
	Decon. Efficacy (non-brushed) (%)	**87.2 ± 0.91**	**85.6 ± 0.64**
	Decon. Efficacy (brushed) (%)	**98.0 ± 0.15**	**98.5 ± 0.15**

11260x dilution factor for samples and controls

NOTES:

- Application of a homogenous, thin layer of Motor oil contaminant on the respective substrate facilitated an optimized interaction between contaminant and DeconGel, and provided an accurate measure of DeconGel's decontamination efficacy in a scaled-down yet real-world setting. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel.
- ASTM method E1728-03, a standardized swipe testing method used for sampling of contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. Air-dried GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes wetted with methanol/DMSO (1:1) solvent were utilized in this swipe testing method.

- Standardized EPA SW-846 Sampling Method 3500C “Organic Extraction and Sample Preparation” and EPA Method 1654A “PAH Content of Oil by HPLC/UV) were followed as a guideline to prepare all samples and controls. All samples, controls, and standards were prepared using the same solvent and appropriate experimental conditions to ensure accurate and LC/MS instrumental analysis.
- LC/MS instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of organic compounds. Standardized EPA SW-846 Analytical Method 8321B “Solvent-Extractable Nonvolatile Compounds by High-Performance Liquid Chromatography/Thermospray/Mass Spectrometry (HPLC/TS/MS) or Ultraviolet (UV) Detection” was followed as a guideline to prepare all samples and controls.
- To ensure accurate determination of DeconGel decontamination efficacy, a standard curve of the analyte of interest was prepared using sufficiently pure analyte; the respective standards were diluted to a known concentration (ppm) using the same solvent as used for samples and controls.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.07 g (1.0 mL of 3.5 g/50 mL hexane) Motor oil contaminant was evenly applied via brushing on 1) aluminum (surface area: 56.3 cm^2), 2) stainless steel (surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and then the hexane carrier solvent allowed to evaporate for 20 min. Approximately 6.0 g of DeconGel 1101 or 1102 was either poured or brushed (brushed first in a top-bottom, then in a left-right fashion) onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC) wetted with methanol:DMSO (1:1) solvent (2 mL). Swipe and dried film samples were suspended in 50 mL methanol:DMSO (1:1) for 24 h. Samples were then analyzed via LC/MS (see below).

Control Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied via brushing on 1) aluminum (surface area: 56.3 cm^2), 2) stainless steel (surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC) wetted with methanol:DMSO (1:1) solvent (2 mL). Swipe samples were suspended in 50 mL methanol:DMSO (1:1) for 24 h. Samples were then analyzed via LC/MS (see below).

Reagents and Standards

Motor oil (SAE 5W-30, Valvoline) was suspended in methanol/DMSO (1:1) solvent mixture, sonicated for 10 min, let to stand for 24 h, and then the liquid decanted from undissolved material used to generate standard curves.

Analytical Instrumentation

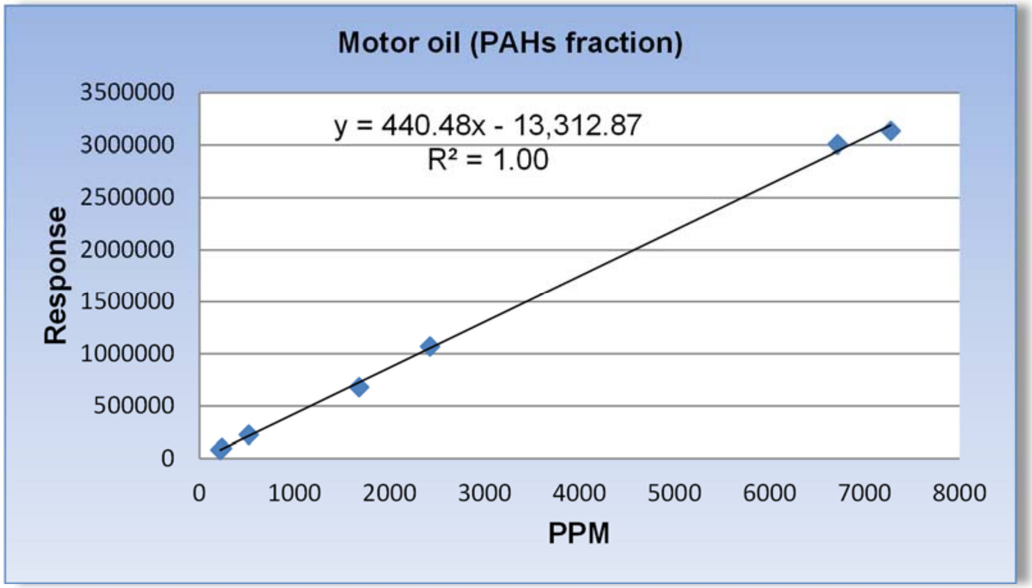
A Thermo LCQ LC/MS with autosampler in positive mode was used to determine PAHs (polyaromatic hydrocarbons) fraction concentration (ppm, wt/wt) of all samples and controls, using a Grace Davison (Deerfield, IL) C18-Select analytical column (150 x 4.6 mm, 5 um).

A 7-point standard curve derived from three independently prepared stock solutions (motor oil dissolved in methanol:DMSO (1:1) was prepared. The calibration curve exhibits a curve fitting as approximated by the coefficient of determination of linear regression R^2, where $R^2 = 1.0$ (see Figure 1).

LC method using A= water (0.1% formic acid), B=acetonitrile (0.1% formic acid); start at 95%A to 3 min at 70%A, to 6 min at 50%A, to 12 min at 30%A, to 15 min at 90%B, to 16 min at 100%B, hold until 22 min at 100% B, to 23 min at 95%A, hold until 24 min.

Motor oil (PAHs fraction) LC/MS data: 19.8 min; lambda max = 284 nm; no data acquired for MS.

Figure 1. Motor oil (PAHs fraction) Sstandard Calibration Ccurve



APPLICATION INSTRUCTIONS FOR END-USERS

Use product directly as is from container. DO NOT DILUTE. Masking or painters tape can be applied along one edge of the area that is to be decontaminated to aid creating a peeled edge to grip for peeling the dried film. Apply DeconGel using a paint brush, a trowel, a handheld sprayer, or an industrial grade sprayer (use DeconGel 1120 or 1121 for spray application).

The thickness of the gel and the number of coats is dictated by the surface to be decontaminated. Coating thickness required for good peel characteristics varies with substrate and generally increases with substrate porosity. It is recommended that first time customers test DeconGel on a small sample area to confirm the required film thickness and dry time for their specific application. If the film is difficult to peel, please apply an additional coat. A razor blade is useful to start the peel. Lay the blade nearly flat and fillet the edge of the film to create a tab that can be pulled. For surfaces that the gel adheres to well, such as concrete, 12" – 24" strips can be cut in the film resulting in less force being required to peel the film.

- Let DeconGel dry for 24 hours

Dry time will vary depending on humidity, temperature, air flow and thickness of the DeconGel. This can take from as little time as an hour for thin coats in a dry environment with plenty of airflow, to overnight or longer if thicker coats are applied in humid environments. Dry times exceeding 24 hours may sometimes be required for good peel performance on bare concrete, wood and other highly porous substrates and substrates with deep cracks or grooves. However, 18-24 hrs is often sufficient dry time on good quality concrete. It is recommended that users test a small area to determine drying time prior to applying DeconGel for an entire job. Supplemental heat or air circulation will accelerate DeconGel's drying time for any job.

- Peel DeconGel off the surface by starting from one of the edges

When dry, the product locks the contaminants into a polymer matrix. The film containing the encapsulated contamination can then be peeled. DeconGel peels from most non-porous and porous hard surfaces if the dried film is thick enough. If the film is difficult to peel, add another coat, let dry, and peel. In most cases the DeconGel will come off in a single sheet but for odd shaped

surfaces you may be required to score DeconGel in order to be able to peel it off.

- Dispose of the dried DeconGel in accordance with the local, state and Federal disposal regulations of the contaminant/substance you are removing. DeconGel itself has no special disposal restrictions.

For more information on application techniques visit our website: www.decongel.com

7.26. End-User Report for Navy Dye Marker

TITLE: Surface Decontamination of Navy Dye Marker by DeconGel 1101

ANALYST: Geoff Nadolski PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVES: Surface decontamination determination of DeconGel formulation 1101 on aluminum, stainless steel, and concrete surfaces contaminated with Navy Dye Marker using LC/MS (Liquid Chromatography/Mass Spectrometry) following Environmental Protection Agency (EPA) SW-846 Methods 3500C (Sampling) and 8321B (Analysis).

HAZARDOUS MATERIALS RELEVANCE: Navy Dye Marker is a water-soluble dye used as an ocean marker for a variety of applications and uses. Navy Dye Marker resists short-term environment-mediated degradation and possesses a strong chromophore, and as such even small amounts of dye are plainly visible on both porous and non-porous surfaces, complicating the complete and facile removal of Navy Dye Marker from commonly utilized surfaces.

SUMMARY RESULTS:

- As seen in Table 1, excellent surface decontamination was achieved by applying DeconGel 1101 onto contaminated surfaces, resulting in encapsulation of Navy Dye Marker contaminant by DeconGel's active components. Decontamination efficacies of poured DeconGel 1101 ranged from 99.1% (on concrete) to 99.3% (on aluminum) to 99.4% (on stainless steel) as determined by residual swipe/solvent extraction analysis. Additionally, for concrete substrate, no residual dye was detected visually when adding droplets of water to the concrete panels after DeconGel administration and film peel.
- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of organics in polar solvent solvated samples. When necessary, the digestion methods were customized to result in the complete dissolution of the organic contaminants and to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Table 1 shows the decontamination efficacies of DeconGel 1101 on stainless steel, aluminum, and concrete surfaces contaminated with Navy Dye Marker as determined by residual swipe/solvent extraction testing.

Table 1. Decontamination efficacies of DeconGel 1101 on Navy Dye Marker contaminated stainless steel, aluminum, and concrete surfaces as determined by residual swipe testing/solvent extraction.

Swipe/Extraction/Visual Testing (ppm)		Formulation
		DeconGel 1101
Stainless Steel*	Control	198.63 ± 0.12
	Residual	1.28 ± 0.10
	Decon. Efficacy (%)	**99.4 ± 0.10**
Aluminum*	Control	198.71 ± 0.14
	Residual	1.34 ± 0.10
	Decon. Efficacy (%)	**99.3 ± 0.10**
Concrete**	Control	199.38 ± 0.58
	Residual	1.87 ± 0.14
	Visual inspection	ND***
	Decon. Efficacy (%)	**99.1 ± 0.10**

10000x dilution factor for samples and controls

* Decontamination efficacy determined using residual swipe testing

** Decontamination efficacy determined using solvent extraction

*** ND: not detected (residual dye not detected visually after 5, 24 hours after adding droplets of water to decontaminated concrete panel)

NOTES:

- Application of a homogenous, thin layer of Navy Dye Marker contaminant on the respective substrate facilitated an optimized interaction between contaminant and DeconGel, and provided an accurate measure of DeconGel's decontamination efficacy in a scaled-down yet real-world setting. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel.

- ASTM method E1728-03, a standardized swipe testing method used for sampling of contaminants was the integral method used to accurately evaluate DeconGel's decontamination efficacy. Pre-wetted (with water) GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes were utilized in this swipe testing method.

- For concrete testing surface, both control and sample coupons contaminated with Navy Dye Marker were extracted with deionized water in a suitably-sized closed plastic bottle, such that concrete coupons were completely submerged in water (50 mL) for 24 h to afford the complete dissolution of analyte in such a porous substrate such as concrete.

- To evaluate leaching dynamics of the Dye in porous substrates such as concrete, after decontamination using DeconGel, water droplets were added to the decontaminated concrete panels to determine if any residual Dye remained on or within the substrate surfaces. Applied water droplets were inspected after 5 and 24 hours of administration, and gratifyingly, no residual dye/coloration was noted for any of the water droplets/concrete surfaces tested.

- Standardized EPA SW-846 Sampling Method 3500C "Organic Extraction and Sample Preparation" was followed as a guideline to prepare all samples and controls. All samples, controls, and standards were prepared using the same solvent and appropriate experimental conditions to ensure accurate and LC/MS instrumental analysis.
- LC/MS instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of organic compounds. Standardized EPA SW-846 Analytical Method 8321B "Solvent-Extractable Nonvolatile Compounds by High-Performance Liquid Chromatography/Thermospray/Mass Spectrometry (HPLC/TS/MS) or Ultraviolet (UV) Detection" was followed as a guideline to prepare all samples and controls.
- To ensure accurate determination of DeconGel decontamination efficacy, a standard curve of the analyte of interest was prepared using sufficiently pure analyte; the respective standards were diluted to a known concentration (ppm) using the same solvent as used for samples and controls.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

Decontamination Efficacy (Solvent Extraction Testing) =

[(Contaminant (ppm) of Solvent Extraction Control) – (Contaminant (ppm) of Residual Solvent Extraction)/Contaminant (ppm) of Solvent Extraction Control] x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.005 g (0.50 mL of 0.5 g/50 mL deoinized water) Navy Dye Marker contaminant was evenly applied via brushing on 1) aluminum (surface area: 56.3 cm^2), 2) stainless steel (surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was poured onto the contaminated surface and let to dry for

24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was either 1) swipe tested (ASTM method E1728-03) using pre-wetted (with water) GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes (for aluminum and stainless steel coupons); or 2) extracted with deionized water (50 mL) in a suitably-sized closed plastic bottle as to completely submerge contaminated coupon surfaces with solvent, and let to stand for 24 h to afford the complete dissolution of analyte (for concrete coupons). Swipe and dried film samples were suspended in 50 mL deionized water for 24 h. All samples were then analyzed via LC/MS (see below). To evaluate leaching dynamics of the Dye in concrete (porous substrate), after decontamination using DeconGel, small water droplets (deionized water, 2 mL) were added throughout the area of the decontaminated concrete panels. Applied water droplets were inspected visually after 5 and 24 hours of administration for any notable coloration on the surface or within water droplets.

Control Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied via brushing on 1) aluminum (surface area: 56.3 cm^2) or 2) stainless steel (surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using pre-wetted (with water) GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes. Swipe samples were suspended in 50 mL deionized water for 24 h, and then analyzed via LC/MS (see below).

For Solvent Extraction Control samples, a respectable amount of contaminant was evenly applied via brushing on concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the coupons were extracted with deionized water (50 mL) in a suitably-sized closed plastic bottle as to completely submerge contaminated coupon surfaces with solvent, and let to stand for 24 h to afford the complete dissolution of analyte, and then analyzed via LC/MS (see below).

Reagents and Standards

Navy Dye Marker powder (sourced in Oahu, Hawaii) was dissolved in water and used to generate standard curves.

Analytical Instrumentation

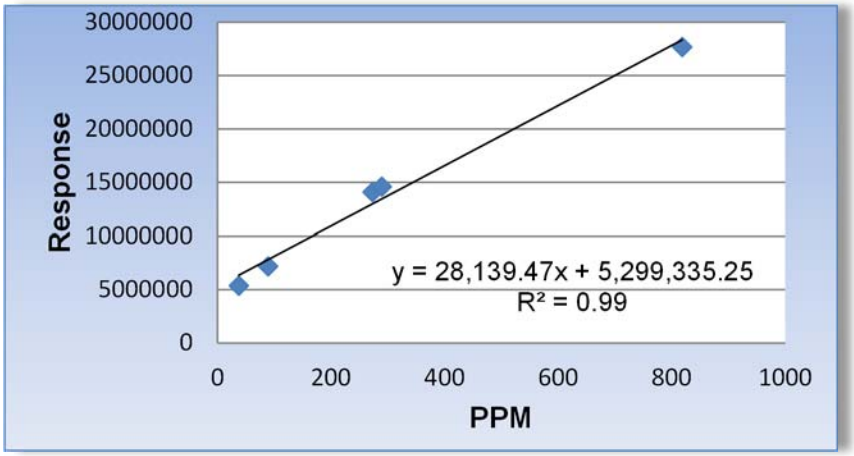
A Thermo LCQ LC/MS with autosampler in positive mode was used to determine Navy Dye Marker concentration (ppm, wt/wt) of all samples and controls, using a Grace Davison (Deerfield, IL) C18-Select analytical column (150 x 4.6 mm, 5 um).

A 5-point standard curve derived from three independently prepared stock solutions was prepared using deionized water as the working solvent. The calibration curve exhibits a curve fitting as approximated by the coefficient of determination of linear regression R^2, where $R^2 = 0.99$ (see Figure 1).

LC method using A= water (0.1% formic acid), B=acetonitrile (0.1% formic acid); start at 90%A to 2 min at 70%A, to 3 min at 50%A, to 5 min at 30%A, to 6 min at 100%B, hold until 16 min at 90% A hold until 17 min.

Navy Dye Marker LC/MS data: 7.58 min; lambda max = 266, 293 nm; M^+ = 333.

Figure 1. Navy Dye Marker Standard Calibration Curve



APPLICATION INSTRUCTIONS FOR END-USERS

Use product directly as is from container. DO NOT DILUTE. Masking or painters tape can be applied along one edge of the area that is to be decontaminated to aid creating a peeled edge to grip for peeling the dried film. Apply DeconGel using a paint brush, a trowel, a handheld sprayer, or an industrial grade sprayer (use DeconGel 1120 or 1121 for spray application).

The thickness of the gel and the number of coats is dictated by the surface to be decontaminated. Coating thickness required for good peel characteristics varies with substrate and generally increases with substrate porosity. It is recommended that first time customers test DeconGel on a small sample area to confirm the required film thickness and dry time for their specific application. If the film is difficult to peel, please apply an additional coat. A razor blade is useful to start the peel. Lay the blade nearly flat and fillet the edge of the film to create a tab that can be pulled. For surfaces that the gel adheres to well, such as concrete, 12" – 24" strips can be cut in the film resulting in less force being required to peel the film.

- Let DeconGel dry for 24 hours

Dry time will vary depending on humidity, temperature, air flow and thickness of the DeconGel. This can take from as little time as an hour for thin coats in a dry environment with plenty of airflow, to overnight or longer if thicker coats are applied in humid environments. Dry times exceeding 24 hours may sometimes be required for good peel performance on bare concrete, wood and other highly porous substrates and substrates with deep cracks or grooves. However, 18-24 hrs is often sufficient dry time on good quality concrete. It is recommended that users test a small area to determine drying time prior to applying DeconGel for an entire job. Supplemental heat or air circulation will accelerate DeconGel's drying time for any job.

- Peel DeconGel off the surface by starting from one of the edges

When dry, the product locks the contaminants into a polymer matrix. The film containing the encapsulated contamination can then be peeled. DeconGel peels from most non-porous and porous hard surfaces if the dried film is thick enough. If the film is difficult to peel, add another coat, let dry, and peel. In most cases the DeconGel will come off in a single sheet but for odd shaped surfaces you may be required to score DeconGel in order to be able to peel it off.

- Dispose of the dried DeconGel in accordance with the local, state and Federal disposal regulations of the contaminant/substance you are removing. DeconGel itself has no special disposal restrictions.

For more information on application techniques visit our website:
www.decongel.com

7.27. End-User Report for Tin Compounds

TITLE: Surface Decontamination of Tin compounds (tributyltin chloride) by DeconGel 1101

ANALYST: Geoff Nadolski PhD, Senior Polymer Scientist, CBI Polymers, Inc.

OBJECTIVES: Surface decontamination efficacy of DeconGel 1101 on stainless steel, aluminum, and concrete surfaces contaminated with organotin (tributyltin chloride) compound; experimentation and associated analyses using ICP-OES (Inductively Coupled Plasma-Optical Emission Spectroscopy) following Environmental Protection Agency (EPA) SW-846 Methods 3005A (sampling) and 6010C (analysis).

HAZARDOUS MATERIALS RELEVANCE: Tin compounds (organotin) have been extensively used as biocides, wood preservatives, and as anti-biofouling agents, however, concerns over potent toxicity to marine life have led to a worldwide ban by the International Maritime Organization. Organotin compounds are considered environmentally persistent pollutants.

SUMMARY RESULTS:

- As seen in Table 1, excellent surface decontamination was achieved by applying DeconGel 1101 via brushing onto contaminated surfaces, resulting in encapsulation of organotin contaminant by DeconGel's active components. Decontamination efficacies of brushed DeconGel 1101 ranged from 99.0% (on concrete) to 99.3% (on aluminum) to 99.4% (on stainless steel) as determined by residual swipe analysis; decontamination efficacies of poured DeconGel 1101 ranged from 86.9% (on concrete) to 96.2% (on stainless steel) to 96.3% (on aluminum) as determined by residual swipe analysis.

- Optimized experimental and analytical methods were successfully developed following standardized EPA sampling and analysis methods as guidelines for determination of inorganics/organometallics in aqueous/polar aprotic solvated samples. When necessary, the digestion methods were customized to result in the complete dissolution of the inorganic contaminants and to ensure accurate decontamination efficacy determination of DeconGel.

RESULTS: Table 1 shows the decontamination efficacies of DeconGel 1101 on stainless steel, aluminum, and concrete surfaces contaminated with organotin compound (tributyltin chloride), as determined by residual swipe testing.

Table 1. Decontamination efficacies of DeconGel 1101 on Organotin (tributyltin chloride) contaminated stainless steel, aluminum, and concrete surfaces as determined by residual swipe testing.

Swipe Testing (ppm)		**Formulation**
		DeconGel 1101
Stainless Steel	Control	465.03 ± 2.80
	Residual (non-brushed)	17.84 ± 0.88
	Residual (brushed)	2.82 ± 0.60
	Decon. Efficacy (non-brushed) (%)	**96.2 ± 0.21**
	Decon. Efficacy (brushed) (%)	**99.4 ± 0.10**
Aluminum	Control	468.37 ± 3.39
	Residual (non-brushed)	17.24 ± 0.83
	Residual (brushed)	3.34 ± 0.28
	Decon. Efficacy (non-brushed) (%)	**96.3 ± 0.21**
	Decon. Efficacy (brushed) (%)	**99.3 ± 0.10**
Concrete	Control	341.73 ± 3.79
	Residual (non-brushed)	44.82 ± 0.77
	Residual (brushed)	3.47 ± 0.45
	Decon. Efficacy (non-brushed) (%)	**86.9 ± 0.16**
	Decon. Efficacy (brushed) (%)	**99.0 ± 0.12**

764x dilution factor for samples and controls

NOTES:

- Application of a homogenous, thin layer of the organotin liquid contaminant on the respective substrate facilitated an optimized interaction between contaminant and DeconGel, and provided an accurate measure of DeconGel’s decontamination efficacy in a scaled-down yet real-world setting. No less than 6.0 g of DeconGel was used for each experiment to allow an optimized interaction between contaminant and DeconGel.

- ASTM method E1728-03, a standardized swipe testing method used for sampling of contaminants was the integral method used to accurately evaluate DeconGel’s decontamination efficacy. Air-dried GhostWipe™ (Environmental Express; Mt. Pleasant, SC) swipes wetted with DMSO solvent were utilized in this swipe testing method.

- Standardized EPA SW-846 Sampling Method 3005A “Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy” was followed as a guideline to prepare all samples and controls. All samples, controls, and standards were prepared using the same dissolution solution and experimental conditions to ensure both correct instrument calibration and accurate analytical results.

- ICP-OES instrumentation is a sensitive and accurate analytical tool for qualitative and quantitative determination of a large number of inorganic compounds. Standardized EPA SW-846 Analytical Method 6010C "Inductively Coupled Plasma-Atomic Emission Spectrometry" was followed as a guideline to prepare all samples and controls.
- To ensure accurate determination of DeconGel decontamination efficacy, calibration standards of the analyte of interest were prepared using either a sufficiently pure analyte or an appropriate ICP-MS Standard (Ricca Chemical Company; Arlington, TX); the respective standards were diluted to a known concentration (ppm) using the same digestion method as the one used for samples and controls. Instrument blank controls were DI H_2O ($\geq$17 M-Ohm) and DMSO solvent.

CALCULATIONS:

Decontamination Efficacy (Swipe Testing) =

[(Contaminant (ppm) of Swipe Control) – (Contaminant (ppm) of Residual Swipe)/Contaminant (ppm) of Swipe Control] x 100%

MATERIALS AND METHODS:

Sample Method

In a typical procedure, 0.080 g tributyltin chloride contaminant was evenly applied via brushing on 1) aluminum (surface area: 56.3 cm^2), 2) stainless steel (surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons. Approximately 6.0 g of DeconGel 1101 was either poured or brushed (brushed first in a top-bottom, then in a left-right fashion) onto the contaminated surface and let to dry for 24 h. Dried DeconGel samples were peeled off the contaminated surface, and the surface was swipe tested (ASTM method E1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC) wetted with DMSO solvent (2 mL). Swipe and dried film samples were suspended in 50 mL DMSO for 24 h. Samples were then analyzed using ICP-OES following EPA SW-846 Method 6010C (analysis).

Control Methods

For Swipe Control samples, a respectable amount of contaminant was evenly applied via brushing on 1) aluminum (surface area: 56.3 cm^2), 2) stainless steel (surface area: 56.3 cm^2), or 3) concrete (industrial grade, surface area: 56.3 cm^2) coupons, and the surface was swipe tested (ASTM method 1728-03) using an air-dried GhostWipe™ swipe (Environmental Express; Mt. Pleasant, SC) wetted with DMSO solvent (2 mL). Swipe samples were suspended in 50 mL DMSO for 24 h. Samples were then analyzed using ICP-OES following EPA SW-846 Method 6010C (analysis).

Reagents and Standards

Tributyltin chloride, liquid, (CAS# 1461-22-9, Fisher Scientific; Fair Lawn, NJ), was used as received.

A 1000 ppm calibration standard of contaminant was prepared using tributyltin chloride in DMSO solvent. DMSO was used as the blank sample.

Analytical Instrumentation

A Thermo ICP-OES instrument model radial iCap 6300 was used to determine tin concentration (ppm) of all samples and controls using a freshly prepared 1000 ppm calibration standard.

Analyte (aluminum) analyzed at 189.9 nm

Pump Speed: 0.5 mL/min

APPLICATION INSTRUCTIONS FOR END-USERS

Use product directly as is from container. DO NOT DILUTE. Masking or painters tape can be applied along one edge of the area that is to be decontaminated to aid creating a peeled edge to grip for peeling the dried film. Apply DeconGel using a paint brush, a trowel, a handheld sprayer, or an industrial grade sprayer (use DeconGel 1120 or 1121 for spray application).

The thickness of the gel and the number of coats is dictated by the surface to be decontaminated. Coating thickness required for good peel characteristics varies with substrate and generally increases with substrate porosity. It is recommended that first time customers test DeconGel on a small sample area to confirm the required film thickness and dry time for their specific application. If the film is difficult to peel, please apply an additional coat. A razor blade is useful to start the peel. Lay the blade nearly flat and fillet the edge of the film to create a tab that can be pulled. For surfaces that the gel adheres to well, such as concrete, 12" – 24" strips can be cut in the film resulting in less force being required to peel the film.

- Let DeconGel dry for 24 hours

Dry time will vary depending on humidity, temperature, air flow and thickness of the DeconGel. This can take from as little time as an hour for thin coats in a dry environment with plenty of airflow, to overnight or longer if thicker coats are applied in humid environments. Dry times exceeding 24 hours may sometimes be required for good peel performance on bare concrete, wood and other highly porous substrates and substrates with deep cracks or grooves. However, 18-24 hrs is often sufficient dry time on good quality concrete. It is recommended that users test a small area to determine drying time prior to applying DeconGel for an entire job. Supplemental heat or air circulation will accelerate DeconGel's drying time for any job.

- Peel DeconGel off the surface by starting from one of the edges

When dry, the product locks the contaminants into a polymer matrix. The film containing the encapsulated contamination can then be peeled. DeconGel peels from most non-porous and porous hard surfaces if the dried film is thick enough. If the film is difficult to peel, add another coat, let dry, and peel. In most cases the DeconGel will come off in a single sheet but for odd shaped surfaces you may be required to score DeconGel in order to be able to peel it off.

- Dispose of the dried DeconGel in accordance with the local, state and Federal disposal regulations of the contaminant/substance you are removing. DeconGel itself has no special disposal restrictions.

For more information on application techniques visit our website: www.decongel.com