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ECBC-TR-766

DEVELOPMENT OF A PORTABLE SENSITIVE EQUIPMENT DECONTAMINATION SYSTEM

VOLUME II – ACTIVATED CARBON FIBER WIPE

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14. ABSTRACT

The objective of the effort was to develop a portable decontaminant that would be used for the decontamination of gross levels of chemical warfare agents from military surfaces including sensitive equipment, as defined within the Technology Transition Agreement. An activated carbon fiber cloth provides the absorptive capacity that is intrinsic to decontamination capability. Two carbon layers are sandwiched between a protective layer of nylon and a barrier layer of Tyvek®, to prevent carbon fiber shedding and contaminant permeation through to the user following and is compatible with vehicle/aircraft interior material decontamination, respectively. The carbon is saturated with a hydrofluoroether liquid, a nontoxic and environmentally friendly solvent that is compatible with vehicle/aircraft interior material, to aid in the decontamination efficacy efficiency. This report describes the development of the portable decontaminant (Wipe) and the measurements taken to determine its performance.

15. SUBJECT TERMS

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GD
DAAMS
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HD
Carbon Fiber
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PREFACE

The work described in this report was authorized under Contract No. 04-098-D-0014-022-01. The work was started in April 2001 and completed in December 2004.

This report was published through the Technical Releases Office; however, it was edited and prepared by the Decontamination Sciences Branch, Research and Technology Directorate, U.S. Army Edgewood Chemical Biological Center (ECBC).

The work described in this report was performed prior to the development of the 2007 Source Document. Therefore, different test methodology and calculation procedures were used that do not necessarily agree with the current procedures.

The use of either trade or manufacturers' names in this report does not constitute an official endorsement of any commercial products. This technical report may not be cited for purposes of advertisement.

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- David Hesselroth (3M™ Corporation) for technical discussions of the solvents
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DEVELOPMENT OF A PORTABLE SENSITIVE EQUIPMENT DECONTAMINATION SYSTEM

VOLUME II – ACTIVATED CARBON FIBER WIPE

1. EXECUTIVE SUMMARY

Activated carbon fiber (ACF) fabrics recently developed and tested with both gas and/or liquid phase adsorption of contaminants exhibit numerous properties that make them candidates for chemical agent decontamination. Based on their high surface areas, large pore volumes, and textile features, these fabrics are under evaluation by the U.S. Army Edgewood Chemical and Biological Center (ECBC) as portable decontaminants. They may be capable of gross level decontamination for a variety of surface types, including sensitive materials.

This is the second of two volumes reporting on the evaluation of activated carbon fiber (ACF) fabrics for the chemical agent decontamination of sensitive equipment and vehicle interiors. The ECBC is currently developing an ACF fabric mitt to provide a portable immediate and operational decontaminate that will also support a thorough decontamination process by providing contamination reduction for sensitive equipment and vehicle interiors. ACF fabrics recent industrial development, involving both gas and/or liquid phase adsorption of contaminants, exhibits numerous properties that make them candidates for chemical agent decontamination, based on their high surface areas, large pore volumes and textile features.

The work described was an effort to develop a contamination removal technology in support of the Joint Material Decontamination System (JMDS) program an effective chemical agent decontaminant to meet concentration exposure limit thresholds that is compatible with system interior materials.

The goals of the program were, therefore, to identify a wiper design that meets the following requirements:

- Effectively remove at least 90% of the liquid contaminants of interest to DoD, namely chemical agents (CA) and appropriate toxic industrial chemicals (TICs), from surfaces. These surfaces include vehicle interiors and sensitive equipment, which may be contaminated at a level of up to 10 g/m^2.
- Absorb/adsorb the contaminants removed from the treated surfaces into the wiper without any shedding, in order to leave no residues behind on the wiped surface.
- Demonstrate compatibility with the range of materials found in vehicle interiors and on sensitive equipment.
- Supply a system that is man-portable, comfortable to carry, and easy to use.
- Will not create undue safety hazards, and will provide a means to re-package the spent system for storage and transport.
- Provide cost effective use.

Based upon the results on the ACF fabric laboratory tests, the following wiper design was recommended.

1.1 Fabrics and Construction

The fabric combination that results in a decontamination wiper that best meets these requirements is:

1. A facing layer that is a laminate of either PFG 39278 nylon or PFG 66387 bonded to Zorflex 50K ACF with Spun Fab PA-1541C/1-025 web adhesive.
2. A second layer of Zorflex ACF based on HD and GD results, Zorflex 100 Meso suppresses off-gassing and will break down HD, GD, and VX, better than 50K.
3. A backing layer of TychemQC or TychemSL polyolefin to eliminate transfer hazard to the operator.

The three layers are edge-bonded to maintain flexibility.

1.1.1 Size and Configuration

The size and configuration were specified by ECBC, and will be similar in size and shape to the M-295 mitt—approximately 8.5 in. wide and 11 in. high. Like the M-295 mitt, there will be a Velcro strap at the bottom of the wiper to better secure it to wrist of the operator. See Figure 1 for a picture of the wiper.

1.1.2 Transfer Liquid

For all development tests, HFE 7200 was used as the transfer liquid and it was shown to be essential to transfer contaminant that had been removed from a surface, through the non-adsorbent contact layer, into the ACF layer(s) of a wiper. For a wiper of the size described above, approximately 35 mL of HFE 7200 will be required to saturate it.

1.1.3 Packaging

The mitt and a frangible bag containing 35 mL of HFE 7200 are placed in a larger resealable bag. If the mitt is folded in half over the HFE containing pouch, the larger bag will be approximately 6 x 8 in.

2. INTRODUCTION

The Edgewood Chemical and Biological Center (ECBC) worked on the development of a portable wipe from activated carbon fiber (ACF) fabric to remove gross levels of chemical agent contamination, specifically from sensitive equipment and material surfaces (those surfaces that cannot withstand caustic decontamination practices). These efforts are an attempt to support the current Joint Service Sensitive Equipment Decontamination (JSSED) and Joint Service Platform Interior Decontamination (JPID) programs and their related requirements (Joint Operational Requirement Documents). To date there is no decontamination system or process acceptable to the Joint Program Executive Office (JPEO) for sensitive equipment or vehicle interiors.

Extensive research has been conducted in the area of adsorptive processes for decontamination of chemical agents (CA) through the Defense Threat Reduction Agency (DTRA). Traditionally, solid substrates, in the form of particles and powders, such as granular carbon, metallic oxides (magnesium oxide and titanium dioxide), zeolites, etc. have been investigated for the decontamination removal of CA. Recently activated carbon fibers, in the form of felts and fabrics, have gained notice in the area of water and air purification. Although, these materials have gained wide industrial application, little research and development has been done in CA decontamination application.

In November 2006 a user evaluation was conducted through the Joint Material Decontamination System (JMDS) team by Battelle with support from the 20th Support Command, Tech Escort Unit (TEU), Aberdeen Proving Ground, Edgewood, Maryland. The objective of the field demonstration was to evaluate the durability and suitability of a new decontamination wipe.[1] Generally, the mitt concept and use was well received. The exception was the creation of carbon fiber fragments from the abrasion of the bare ACF on the sharp equipment surfaces. To eliminate the shedding of the carbon fibers, a layer of a thin nylon fabric was inserted over the ACF fabric to protect it from abrasion.

This is the second of two reports written for the development of a portable sensitive equipment decontamination system. The first report (Volume I) addressed the evaluation of commercial off the shelf (COTS) wiper material fabrics and fielded Government decontaminants considered suitable as a portable system. This volume (Volume II) addresses activated carbon fiber fabric and the development of a wipe to meet the appropriate requirements for the Joint Service Sensitive Equipment Decontamination (JSSED) and Joint Service Platform Interior Decontamination (JSPID) programs.

This report addresses the limited objectives development for a multi-layer ACF fabric wipe or mitt. The multi-layered design was derived from a series of tests and measures described in this report, and consists of the following four layers:

1. Facing layer – for protection of inner carbon layers against shedding
2. ACF layer 1 – a micro-porous knitted fabric
3. ACF Layer 2 – a meso-porous woven fabric
4. Backing Layer – Tyvec® protection against personnel-transfer hazard

To enhance decontamination efficacy and assist in the mass transfer of contaminant into the ACF pores, a benign solvent (hydrofluoroether) was included.

From the test data cover in this report and Volume I, a breadboard wipe mitt, fashioned after the M295 Kit, is shown in Figure 1.

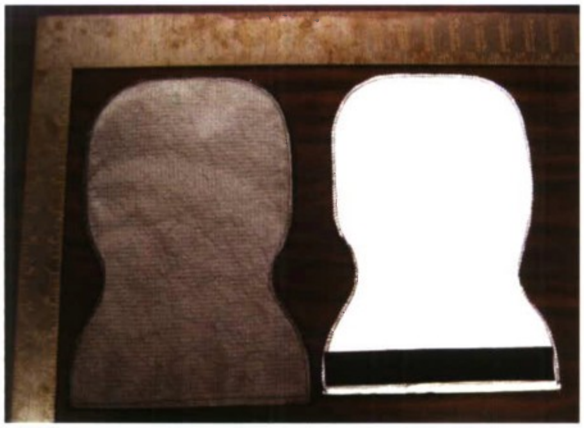

Figure 1. Breadboard mitt design.

2.1 Background

The initial development work on activated carbon fiber (ACF) fabrics, for use as a portable decontaminant, was carried out by Dr. Ralph Spafford and Dr. Robert Kaiser, under contract DAAD13-98-D-0014, Deliver Order 20 (Development of a Portable Sensitive Equipment Decontamination System, Volume I) and Deliver Order 22 (Development of a Portable Sensitive Equipment Decontamination System, Volume II). This work led to the further development of these fabrics to support the operational requirements for a portable broad-spectrum decontamination system, under the Joint Service Sensitive Equipment Decontamination (JSSED) program. The two main conclusions from this effort were:

- A carbon-based adsorptive wipe removed greater than 90 percent of the chemical agents, from a variety of sensitive material surfaces, and performed better than other commercial off the shelf fabrics, and
- Enhanced decontamination efficacy was achieved by: (a) adding a solvent to moisten the fabric, and (b) the application of multiple wipes.

The portable wipe concept originated from the initial JSSED program and the Analysis of Alternatives (AoA). At the request of the Business Area Manager (BAM) for decontamination, the AoA was released in July 2000[2] by a Joint Service Technology Assessment Review Panel.[*] The objective of the AoA was to evaluate technologies appropriate for decontaminating or precision cleaning of sensitive equipment and associated materials, which had some utility in industry or Government. Thirty-two candidate technologies from industry and other defense programs were evaluated, with the conclusion that none provided a comprehensive approach to meeting the JSSED ORD. However, the technology panel did agree that by dividing the requirement into three technology capability segments (three blocks), a comprehensive solution could be achieved. The third block (Block III) identified the need for a highly portable device to be used during operations, which is capable of resulting in Mission-Oriented Protective Posture (MOPP) reduction. The technology panel considered a wiper material made from or incorporating an adsorbent matrix to be a viable approach to meet the objectives for the operational requirement for Block III. A second AoA (AoA II) developed under the direction of the Commodity Area Manager (CAM) for Decontamination, released in July of 2005[3], came to a similar conclusion as its predecessor—none of the technologies investigated offer a comprehensive solution, and a portable device made from adsorptive or absorptive fabric had the potential to achieve the Immediate and Operational requirement objectives.

2.2 Technical Approach

The technical approach of this effort was to determine, through a series of laboratory tests in controlled environments, whether or not an activated carbon fiber fabric wipe could achieve the limited objectives stated in the JPID ORD[4] and JSSED ORD[5], for immediate and operational decontamination. In addition, a third objectives document, titled: "Technology Transition Agreement for the Wipe Technology to Meet the Joint Material Decontamination System (TTA #08-JMDS-06-001T)" (TTA)[6] was issued in an attempt to coordinate a Milestone B transition of a decontamination system.

The aforementioned ORD's do not specifically call for the development of an ACF fabric wipe in support of the overall decontamination system development, but indicate the possibility for two

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[*] The Technology Assessment Review Panel (TARP) included Joint Service participation from relevant technology experts involved in the development and/or evaluation of decontamination processes and including process effects; this panel was convened March 2000 in Tampa, Florida.

decontamination systems to achieve the requirements, including immediate and operational decontamination, and a portable system. The third document, the TTA, specifically defines the level of CA decontamination as a 90% reduction in agent, from a gross contamination level of $10 gm/m^2$ surface area, on a sensitive equipment material surface.

Decontamination levels, *immediate* and *operational*, as defined in Joint Publication 3-11[7], are as follows:

- Immediate – minimize casualties, save lives, limit spread by personal wipe-down, and operator spray-down of frequently touched surfaces, and

- Operational – reduce contact hazard and limit spread to eliminate or reduce the need for protective equipment.

Another important definition is that for the term "decontamination." Taken from the Joint Science and Technology Office (JSTO), Chemical and Biological Defense, CA *decontamination* is defined as the reduction in the harmful quantity of material from a surface. Decontamination does not mean the reduction is complete or the contaminant has to be neutralized of its toxic effects. This is not to say that complete removal or chemical detoxification is not desirable.

2.3 Test Objectives

The test objectives were derived from interpretation of the Key Performance Parameters found in the JSSED ORD, JPID ORD, and TTA. The test objectives were set of tests with measurements designed to provide data for the evaluation of the ACF wipe against user requirements, as they were interpreted from the appropriate documents.

- Solvent Extraction and GC Analysis: Rationale—the objective for this test segment was to measure the amount of chemical agent removed from a surface by the wiper process, usually measured by weight. This gross level removal was a required effect, identified as a minimum-acceptable performance threshold for the JMDS decontamination wipe system as described in TTA #08-JMDS-06-001T.

- Contact Test Using M-8 Indicator Paper: Rationale—the JPID ORD requires Immediate/Operational efficacy from a starting liquid challenge of $1 gm/m^2$ to below detection limit of M8 Paper.

- Off-Gassing from Spent Activated carbon fiber fabric: Rationale—the JSSED and JPID ORD require that the process or system must not permit any residual health hazards to personnel. This test segment evaluated the spent wipe fabric for off-gassing hazard associated with HD and GD decontamination process.

- Contact Hazard Test in Accordance with TOP 8-2-061, Single and Multiple Wipe Process: Rationale—the JPID ORD defines safe exposure levels for thorough decontamination as contact exposure levels.[8] The Test Operations Procedure (TOP) 8-2-061 for Decontamination Systems Laboratory/Field Testing was used to define the procedure for the contact exposure test. Single and multiple wipe decontamination processes were employed.

- Equipment Degradation: Rationale—the JPID and JSSED ORD require that no equipment/material degradation will occur beyond tactical mission capability. The two issues associated with the wipe process include the solvent and its materials compatibility, and shedding or residual fibers from the ACF fabric.

- Additional Development Testing: Measurements, such as solubility and performance tests with chemical agent simulants, were also included to provide insight into the development of the multi-layered actviated carbon fiber fabric wiper. In addition, the intent of the adsorptive wipe would be for dual-use on field non-chemical agent waste (i.e., motor oil, grease, lubricants).

The term *dissolution* is used here in a broader sense, to signify the complex interaction between mass transfer, solubility, and adsorption. Decontamination efficacy for a non-reactive system is only going to be as good as the surface-contaminant relationship, in other words, if the contaminant is absorbed below the substrate surface, effective decontamination will be difficult.

3. EXPERIMENTAL PROCEDURE

3.1 Wipe Materials

The materials in the following sections were used as wipe materials in this program testing.

3.1.1 Activated Carbon Fabrics (ACFs)

- Zorflex Activated Carbon Fabric (Calgon Carbon Corp, Chemiviron Carbon Ltd, Essex, England)
    - 50K
    - 10 micro
    - 100 Micro
    - 10 Meso
    - 100 Meso
    - 10 Meso Experimental Fabrics ST-1 to ST-5
- Kothmex Activated Carbon Fabric (Taiwan Carbon Technology Company, Nantuen Chiu, Taiwan)
    - Kothmex AM-1131

3.1.2 Facing Fabrics/Films

- PFG Polyester Fabrics (Performance Fabrics Group, Inc., Greensboro, NC, 27401)
    - 54717
    - 60171
    - 64918

  - 66290
  - S/E 1122
- PFG Nylon Fabrics:
  - 32978
  - 66190
  - 66165
- Delstar Polyethylene Films (Delstar Technologies, Inc., Middletown, DE 19709)
  - P520NAT-A
  - PQ218NAT-E
- Other Commercial Off-the-Shelf (COTS) Fabrics
  - Perfect Clean Microfiber Wipes
  - 3M Scotchbrite Cleaning Cloth
  - Polypropylene – Sock Fabric
- "Wicking Fabrics" From Running T-Shirts
  - Nike Dri-Fit – 100% Polyester
  - Under Armour Heatgear – 95% Polyester, 5% Lycra
  - Under Armour Heatgear – 80% Polyester, 20% Lycra

3.1.3 Backing Fabric (E.I. DuPont de Nemours & Co., Inc.)

- Tyvek® 1443R
- Tychem SL – Style 56591, Mix 1
- Tychem QC

3.1.4 Web-Bond Adhesives (Spunfab, Ltd.,Cuyahoga Falls, OH)

- Spunfab PA 1541C/1-0.25Spunfab PO 4401-0175
- Spunfab PO 4401-025
- Spunfab PA 1008-0.5
- Spunfab PE 2900-0.5
- Spunfab SL 7001-0.7

- Bostik SH 2410 (polyamide)

3.2 Coupon Test Materials

The test materials were selected by their common use on sensitive equipment or vehicle interiors. These materials have been used in other sensitive equipment decontaminant evaluation testing and offer a variety of functional and structural uses. The test materials used are listed below. The test coupons were cut (or prepared) from sheets of the test materials as 5.08 cm diameter circles, with an area of 0.002026 m^2, except for the polycarbonate and glass coupons. The polycarbonate coupons were cut into 2 in. diameter squares and the glass coupons (also circles) were purchased from McMaster-Carr and used as received. All of the coupons were washed in 2-propanol and either air or oven (100 °C) dried for at least 24 h. The coupons were then stored in separate plastic sealed containers until use.

- Aluminum (AL) – AL7075
- CARC – Iridite wash per MIL-C-5541 (Class 1/A), primer per MIL-P-53022 topcoat per MIL-C-53039A polyurethane (color #383 green)
- Air Force Top Coat – (AF Top Coat) MIL-PRF-85285C (color 36320)
- Polycarbonate – the polycarbonate (PC) was purchased through ECBC Experimental Fabrication Shop from EJ Enterprises, Glen Burnie, MD. The manufacturer of the PC was Sheffield Plastics, Inc., Sheffield, MA. The test coupons are cut from 4 x 8 ft sheets, 0.25 in. thick. The PC is purchased as clear polycarbonate; no other designation data was available.
- Kapton – Polyimide (Kapton HN), purchased from Goodfellow, Berwyn, PA, lot # LS293981, with a 0.125 mm thickness.
- Viton – Viton® fluoroelastomer, hexafluoropropylene vinylidenefluoride copolymer, purchased from Goodfellow, Berwyn, PA, lot # LS219873, with a 3.0 mm thickness
- Nylon Cloth – the nylon cloth was purchased from Franklin Fabrics and was prepared per MIL-C-7219F

3.3 Chemical Agents

All of the chemical agents were purchased from the ECBC Chemical Agent Transfer Facility and used as received:

- HD, bis-(2-chloroethyl) sulfide, Lot # HD-U-6060-CTF-N-2, was a CASARM agent measured to be 97.3 mole% pure.
- VX, O-ethyl-S-(2-diisopropylaminoethyl) methyl phosphonothiolate, Lot # VX-U-9348-CTF-N, was a munitions grade material and measured to be 94.3 weight% pure.
- GD, pinacolyl methyl phosphonofluridate, Lot # GD-U-2323-CTF-N, was another CASARM-grade chemical agent and was measured to be 98.8 mole% pure. No additional detail was given about the impurities.

- TGD, polymer thickened GD, was prepared from the stock GD, Lot # GD-U-2323-CTF-N, in-house by Seok Hong, PhD. The viscosity was measured via a rheometer from Advanced Rheometer, model AR 2000, to be 125 cps at room temperature.

3.4 Chemical Agent Simulants

- Chloroethyl ethylsulfide (CEES), 98%, Aldrich Chemical Cat. No. 242640.
- Diethyl phthalate (DEP), 99.5%,Aldrich Chemical Cat. No. 524972.
- Motor oils: SAE 40, SAE 10W-30, and SAE 80W-85W-90 (NAPA).
- Citroflex 4(tri-n-butyl citrate), Morflex, Inc.
- Fruit Tree Spray, Bonide Products, Inc., Oriskany, NY 13424
- Krytox AZ Oil (a fluorinated polyether), E.I. DuPont de Nemours & Co., Inc.
- Polydimethyl silicone (PDMS) oil, 10 cs, Dow Corning Corp.
- Barrierta L55/2 Fluorinated Grease, Kluber Lubrication.

3.5 Wiper Solvents/Transfer Liquids

Several solvents were used as transfer liquids. The solvents used are provided in the bulleted list. The following Table 1 lists the chemical and physical properties for the wiper solvents.

- HFE 7200 is ethoxyperfluorobutane ($C_4F_9OC_2H_5$), a hydrofluoroether manufactured by the 3M™ Company as a non-ozone-depleting solvent under the trade name Novec™ Engineered Fluid HFE 7200. The HFE 7200 solvent is a clear, colorless, low-odor, volatile liquid that is nonflammable, essentially nontoxic, generally non-hazardous to personnel, and compatible with a wide range of metals, plastics, and elastomers. It has a low environmental impact, and, while highly volatile, evaporates slowly enough to be useful as a solvent in an adsorptive wipe. Additional information on this or any of the 3M™ Novec™ engineered fluids can be found using the following link, http://solutions.3m.com.
- HFE 7100 is methoxyperfluorobutane ether.
- HFE 71IPA is an azeotropic mixture consisting of 95.5% (by weight) HFE 7100 and 4.5% (by weight) isopropanol.
- HFE 7300 (1,1,1,2,2,3,4,5,5,5-decafluoro-3-methoxy-4- (trifluoromethyl) pentane.
- HFE 7500 (3-ethoxy-1,1,1,2,3,4,4,5,5,6,6,6-dodecafluoro-2-trifluoromethyl-hexane.

Table 1. Chemical and physical properties for Novec™ solvents.

Solvent	HFE – 7100	HFE – 7200	HFE – 7300	HFE – 7500
Formula	C4F9OCH3	C4F9OC2H5	C7H3OF13	C9H5OF15
Average Molecular Weight, Dalton	250	264	350	414
Boiling Point @ 760 mm Hg °C	61	76	98	128
Freezing/Pour Point °C	-135	-138	-38	-100
Liquid Density, g/mL	1.52	1.43	1.66	1.61
Surface Tension dynes/cm	13.6	13.6	15	16.2
Viscosity, cSt	0.40	0.43	0.71	0.77
Solubility of Solvent in Water, ppm	12	< 20	0.6	<0.003
Solubility of water in Solvent, ppm	95	92	67	
Vapor Pressure, mm Hg	202	109	45	8
Heat of Vaporization @ BP, cal/g	30	30	24.3	22
Specific Heat, cal/°C-g	0.28	0.29		
Properties at 25°C unless otherwise specified				

The 3M™ Company's Novec™ solvents are considered by the EPA to be included into the Significant New Alternatives Policy (SNAP) program having 0 ozone-depletion potential, or class II substances as defined in section 612(c) of the Clean Air Act.

The Novec™ solvents were also evaluated in the development of the XM25 program: the Joint Service Sensitive Equipment Decontamination Apparatus. A final report was issued from Battelle[9] addressing materials compatibility during the optimization effort.

- Other chemicals
    - Isopropyl alcohol (IPA) has been a common solvent with good solubility properties for CA agents. IPA was purchased from Fisher Scientific and was HPLC grade.
- Chloroform is used as an extraction solvent, purchased from Sigma and was Capillary GC grade.

3.6 M8 Paper

The M8 Paper was purchased from Research Development and Engineering Command, Rock Island, and was used as received. The item description was Paper, Chemical Agent Detector, VGH, ABC-M8, and the lot used during the test was CCR05A310-001. Accordingly, M8 Paper detects and identifies liquid chemical agent with a claimed response detection sensitivity[†] of a drop greater than 0.02 mL (liquid). There is an indicator dye that will cause a color change, depending on the CA contacted, which results in a change in pH triggering the release of the appropriate dye. A blister agent (HD) turns the M8 Paper red, VX should turn the paper dark green, and GD should turn the paper yellow. The color change is stated to occur within 30 s.

[†] M8 Paper detector technical information taken from the Worldwide Chemical Detection Equipment Handbook, Section 20.1 Chemical Agent Detectors.

4. METHODS AND PROCEDURES

4.1 Characterization of Wipe Components

4.1.1 Shedding Tests

The results of the field-testing of prototype activated carbon fabric (ACF) wipers and mitts, performed at ECBC during November of 2006[1], indicated that the decontamination wipers provided did an excellent job of removing the applied organic contaminants from the test pieces being cleaned, but left carbon particles on the test pieces due to abrasion of the activated carbon fibers (ACFs) as they rubbed against the surfaces of these test pieces. The lesson learned from this field test is that the wiper needs to have a protective layer to prevent shedding of activated carbon particles. The protective layer cannot result in a barrier that prevents the transport of contaminants into the adsorbent ACF.

The purpose of the shedding tests was to establish the shedding potential of different candidate facing fabric (CFF)/ACF combinations, and to identify the combination that would result in minimal shedding on the pieces being decontaminated during actual use.

All shedding tests were performed using the rotary-wiping device, the rotary-wiping mandrel, and the four-button keypad shown below in Figure 2.

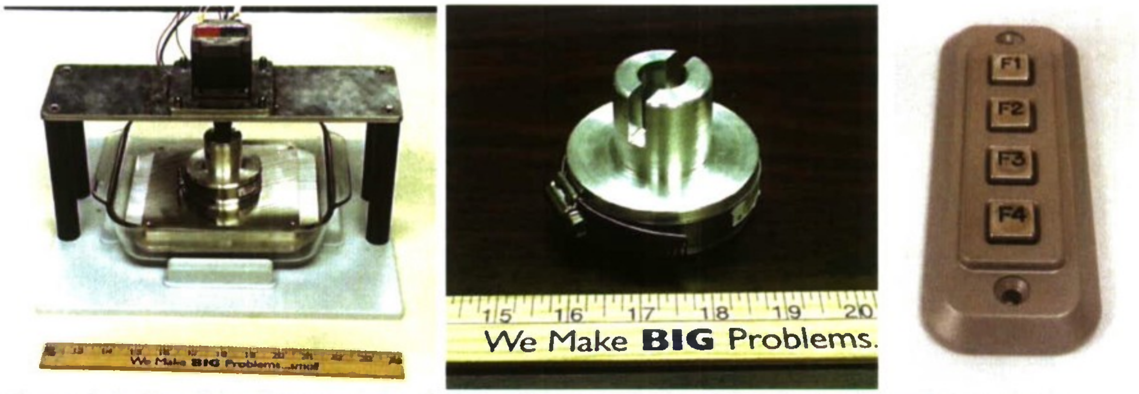

Figure 2. Left to right: Rotary-wiping device, wiping mandrel, and four-button keypad (test piece).

In each test, a swatch of CFF was backed with a swatch of ACF and fastened to the wiping mandrel. In the case of Delstar films, the materials were supplied as a pre-made laminate of the Delstar film to 50K. A circle of the bonded laminate was die-cut and secured to the mandrel using double-back sticky tape. A stack of concentric washers was added to the 3.0 in. OD mandrel to increase the total weight on the fabric from 370 g, which is the weight of the mandrel, to 2570 g so that the amount of pressure exerted by the mandrel (about 0.8 psi) would be similar to the amount of pressure exerted by a person during an actual wiping operation. With the mandrel in place and the four-button keypad positioned directly under the mandrel, rotary wiping program G210 was executed. This program consists of eight cycles, each cycle containing one complete clockwise revolution followed by one complete counterclockwise revolution, at a speed of 0.3 rev/s. A picture was taken of the mandrel and test piece both before and after wiping to establish whether shedding had occurred. The keypad was fully cleaned after testing, with solvent if necessary, to ensure a clean test piece for the next test. Unless otherwise noted, all tests were performed dry as a worst-case shedding scenario, and performed in duplicate.

Rotary shedding tests were performed for the candidate-facing fabric (CFF) and activated carbon-fabric (ACF) combinations marked with an "X" in Table 2. All tests were performed dry, except for the nylon PFG/50K combinations, which have become the leading candidate material combinations. The nylon PFG/50K combinations were tested both dry and wetted with HFE 7200.

Table 2. Rotary shedding test matrix.

Rotary Shedding Test Matrix		Activated Carbon Fabrics			
		Dry			HFE-7200 Wetted
		50K	100 Micro	100 Meso	50K
Control	None	X	N/A	N/A	N/A
PFG Polyester Fabrics	54717	X	X	X	N/A
	64918	X	X	X	N/A
PFG Nylon Fabrics	39278	X	X	X	X
	66190	X	X	X	X
Delstar Polyethylene Films	P520NAT-A	X	N/A	N/A	N/A
	P520NAT-A	X	N/A	N/A	N/A

4.1.2 Flexibility Tests

In the design of a decontamination wiper, one has to take into account the mechanical properties of the candidate fabrics. The fabrics must be strong and sufficiently resistant to abrasion not to fall apart during the wiping operation. In addition, the fabric must be flexible enough to conform to the shape of the surfaces to obtain good contact for surface cleaning.

In theory, a piece of fabric can be viewed as a beam of low mechanical rigidity, whose flexural properties are governed by the standard laws of mechanics and strength of materials.

The curvature of the deflection curve of a rectangular beam in bending can be expressed by the following equation:

$$1/\rho = M/EI \qquad \text{Equation 1}$$

where

$1/\rho$ = radius of curvature

E = modulus of elasticity

M = bending moment

I = moment of inertia of the cross section with respect to the neutral axis

The moment of inertia of a rectangular beam through its central axis is expressed by the following equation:

$$I = bh^3/12 \qquad \text{Equation 2}$$

where

b = width of beam

h = thickness of beam

I = moment of inertia of the cross section with respect to the neutral axis

Everything else being equal, as the thickness of a beam or of a fabric increases, the rigidity increases as the third power of the thickness. Different material layers in a wipe can be attached to each other either uniformly across the area of the wiper, or along the periphery of the wiper. With area bonding, the resulting composite behaves mechanically as a single entity whose thickness is the sum of the thicknesses of the individual layers. With peripheral bonding, each layer is mechanically independent of the other layers. Because of this third power relationship, the composite obtained by area bonding of two or more layers of fabric is much stiffer than the composite obtained by peripheral bonding.

A number of standard test methods exist for the measurement of the stiffness of fabrics. These include:

- ASTM D 1388-96 (Re-approved 2002): Standard Test Method for Stiffness of Fabrics
- ASTM D 4032-94 (Re-approved 2001): Standard Test Method for Stiffness of Fabric by the Circular Bend Procedure.
- ASTM D 5732-95 (Re-approved 2001): Standard Test Method for Stiffness of Nonwoven Fabrics Using the Cantilever Test
- ASTM D 6829-02: Standard Test Method for Stiffness of Fabric by the Blade Slot Procedure
- ASTM D 747-02: Standard Test Method for Apparent Bending Modulus of Plastics by Means of a Cantilever Beam
- Tappi T-451 Flexural Properties of Paper (Clark Stiffness)
- ASTM Methods D 1388-96 and D 5732-95 are basically the same method applied to different types of fabrics.

The ASTM D1388-96 method is by far the simplest to implement, and the only one of the above test methods that does not require the purchase of specialized test equipment.[10] This test method could be performed using an apparatus that could be built in-house from purchased materials, making it the method of choice.

This test method covers determination of the stiffness properties of fabrics by measuring a bending length and calculating the flexural rigidity. ASTM D1388-96, Option A was used for the measurements in this report. This method employs the principle of cantilever bending of the fabric under its own mass. In practice, a 1 x 8 in. specimen is slid at a specified rate in a direction parallel (about 120 mm/min) to its long dimension, until its leading edge projects from the edge of the horizontal surface. The length of the overhang is measured when the tip of the specimen is depressed under its own mass to a point along a line from the top to the edge of the platform, which makes a 0.724 rad (41.5°) angle with the horizontal. From this measured length, the bending length and flexural rigidity are calculated according to the following equations (3 and 4):

$$c = o/2 \qquad \text{Equation 3}$$

and

$$G = W \times c^3 \qquad \text{Equation 4}$$

where

c = bending length, cm

o = length of overhand, cm

G = flexural rigidity, mg cm

W = fabric mass per unit area, mg/cm2

The test apparatus was fabricated by cutting a 2 x 6 in. block of wood to a length of 16 in. with a miter saw so that one side of the length of wood formed a 41.5° with the top surface of the piece of wood. The top and the angled side faces were covered with 2 in. wide by 0.080 in. thick adhesive-backed strips of ultra-high molecular weight (UHMW) polyethylene to minimize the frictional resistance of these surfaces. Adhesive-backed measuring tape was then placed along the side edge of each polyethylene strip to provide a way to measure the length of overhanging fabric. A moveable slide was fabricated by gluing a 5/8 in. diameter machine nut to a 1.5 in. wide x 8 in. long x 0.125 in. thick bar of 304 stainless steel. This bar weighs 250 g.

Figure 3 is a photograph of a piece of fabric on the top platform at the start of the test. Figure 4 is a picture of the same piece of fabric at a point during the test where it has bent less than 41.5°, and Figure 5 and Figure 6 are pictures of the fabric at the end of the test, once a 41.5° bend was attained.

A variety of materials were tested. These included various activated carbon fabrics and facing materials of interest, as well as laminates received from different sources, as listed in Table 3.

Table 3. Fabrics tested.

Activated Carbon Fabrics	
Calgon Zorflex Fabrics:	FM 10 meso, FM 50K, FM 70, FM 100 micro, FM 100 meso
CTK (Taiwan):	CT 1001
Facing Materials	
PFG Fabrics:	54717 HSS, 64918, 39278
Delstar Films	PQ 218 NAT-E, P520 Nat-A

In addition, in order to assess the effect of the adhesive in a laminate on its flexibility, a number of PFG 39278 facing fabric/Zorflex 50K laminates were prepared at Entropic Systems, Inc. (ESI) with different web adhesives and then tested. Attempts to contact two U.S. manufacturers of web adhesives, Bostik and Spunfab Ltd. were initiated. Only Spunfab responded and provided test laminations and samples of some of their different products. The samples of the various products received are listed in Table 4. Laminates with all these products were tested, except SL-7001. SL-7001 was not tested because of its relatively high basis weight and high glue line temperature.

Table 4. Properties of adhesive web materials received from Spunfab Ltd.

Product Name	PA 1541C	PO 4401	PO 4401	PA 1008	PE 2900	SL 7001
Description	Copolyamide	Polyolefin	Polyolefin	Copolyamide	Copolyester	ternary
Basis Weight, osy	0.25	0.175	0.25	0.60	0.60	0.70
Koffle Stick Point, °C	85			102	100	166
Recommended Glue Line Temperature, °C	105 - 125	135 - 150	135 - 150	115 - 130	125- 150	168 - 183

The laminates were prepared by hot pressing a layup of PFG 39278, adhesive web material, and Zorflex 50K, placed between two thin Teflon sheets on a JetPress 14 hot press (manufactured by Geo. Knight & Co., Inc., Brockton, MA). The plate size of 12 x 14 in. dictated the maximum size samples that could be prepared. The sheets were laminated at the recommended glue line temperatures indicated in Table 4, using a standard dwell time of 30 s.

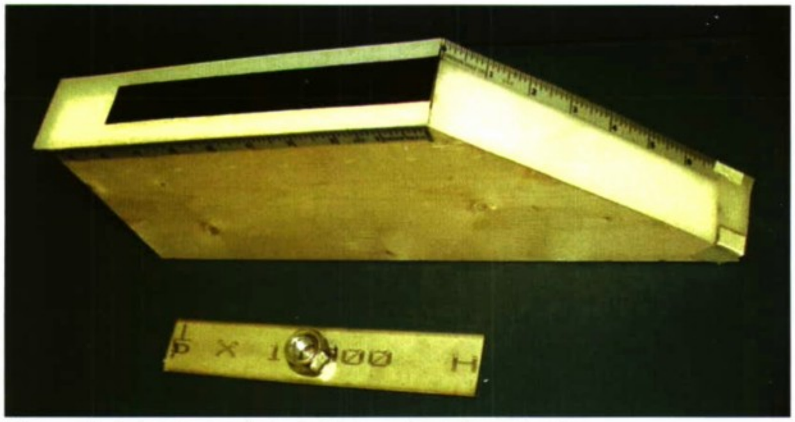

Figure 3. Fabric sample in initial position (top view).

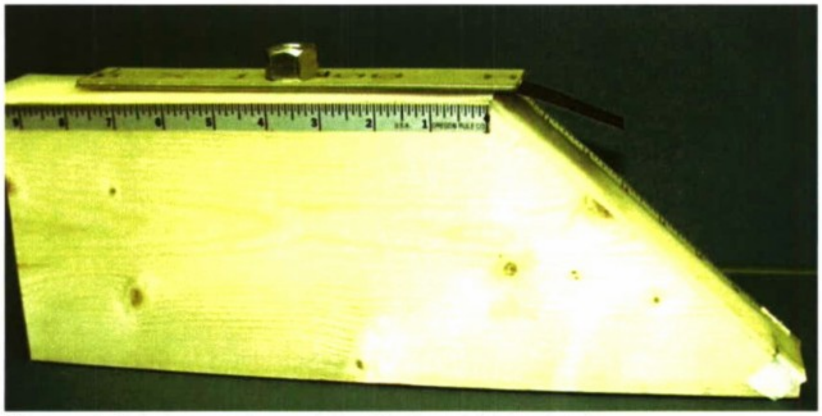

Figure 4. Fabric sample partially displaced (side view).

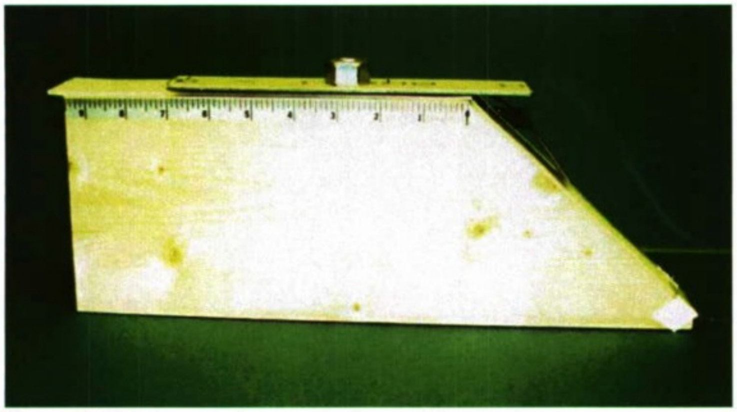

Figure 5. Fabric sample at end of test (side view).

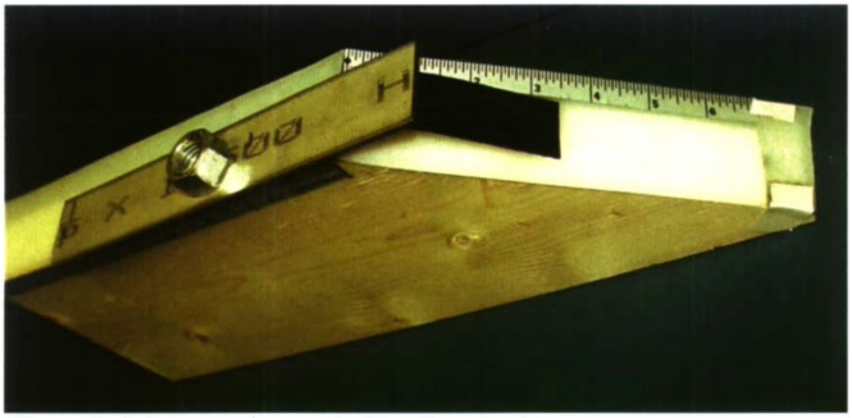

Figure 6. Fabric sample at end of test (top view).

4.2 Contaminant Adsorption Tests

The test objective was to establish a correlation between the pore structure of an activated carbon fabric and the ability of this fabric to remove a CA or a CA simulant from solution in a carrier liquid by adsorption. In these tests, comparable measurements were performed with a military grade vesicant, dichloroethyl sulfide (chemical warfare agent HD), and with chloroethyl ethylsulfide (CEES), a well-established HD simulant. Additional tests were performed with CEES solutions, but not HD solutions, with fabrics with a wider pore size distribution.

Estimates of the molecular dimensions of the liquids of interest, contaminants and carrier liquids, are presented in Table 5. Because of solvation, adsorption from the liquid phase may involve larger molecules than those found in the gas phase. Consequently, it was expected that the size of the pores, which would effectively capture contaminants from a liquid, could also be larger than the micropores that control adsorption from the gas phase. It was anticipated that the adsorption capacity of a sorbent could be enhanced by the presence of mesopores.

Table 5. Estimated diameter of molecules of interest.

	Molecular Weight	Density @ B.P.	Molecular Volume	Molecular Diameter	Est. Solvated Diameter in HFE-7100
	Dalton	g/cm^3	nm^3	nm	nm
GASES					
Helium	4	0.936	0.007	0.19	
Nitrogen	28	0.807	0.058	0.39	
SOLVENTS					
Dichloromethane	84.93	1.325	0.106	0.47	
HFE-7100	250	1.52	0.272	0.65	
HFE-7200	264	1.43	0.307	0.68	
SIMULANTS					
Chloroethyl ethyl sulfide	124.63	1.07	0.193	0.58	1.87
Chloroethyl phenyl sulfide	172.68	1.174	0.244	0.63	1.92
Diethyl methyl phosphonate	152.13	1.041	0.243	0.62	1.92
CWA					
Agent HD	159	1.27	0.208	0.59	1.89
Agent GD	182	1.025	0.296	0.67	1.96

Four different activated carbon fabrics, from two different suppliers, were examined in the tests presented in this section. The suppliers were: Zorflex from Charcoal Cloth International, a subsidiary of the Calgon Corporation, and Kothmex, from Taiwan Carbon Company. Material properties data abstracted for these materials are presented in Table 6.

Table 6. Properties of commercial activated carbon fabrics examined.

Supplier	Kothmex	Calgon	Calgon	Calgon
Material	1131	FM 100 Micro	FM-10 Meso	FM-100 Meso
Form	Felt	1/1 Double Weave	1/1 Plain Weave	1/1 Double Weave
Precursor	PAN	Rayon	Rayon	Rayon
Carbon Content	>90%	>90%	>90%	>90%
Weight, g/m^2	150	240	120	240
Thickness, mm	2	1	0.5	1
BET Surface Area, g/m^2	1230	1360	995	655
Volume Percent Mesopores	13%	18%	60%	84%
Volume Mean Pore Diameter, AU	2	6	22	29

Adsorption tests were performed with 3M's Novec HFE 7100 as the carrier liquid. These tests were also performed with dilute (70 ppm) solutions CEES and of Agent HD in HFE 7100.

A dilute solution of contaminant in HFE 7100 of known concentration was pumped through a column of adsorbent at a constant flow rate, while monitoring the concentration of contaminant in the effluent as a function of time. The operation was stopped when the presence of contaminant was observed in the effluent. Column capacity was equal to the weight of contaminant removed from solution until breakthrough, divided by the weight of adsorbent in the bed.

Circular coupons, 3/8 in. diameter of the fabric being tested, are cut with an arch punch, weighed then packed into a ½ in. OD Swagelok™ connector tube. The apparent volume of the adsorbent tested was 1.4 ± 0.14 (10%) cm^3. The column was inserted into a liquid flow system, powered by a magnetic gear pump and a variable output DC power supply. Piping was 1/16 in. O.D. tubing. Figure 7 is a sketch of the adsorption column. Figure 8 is an experimental flow sheet and Figure 9 is a photograph of the experimental setup.

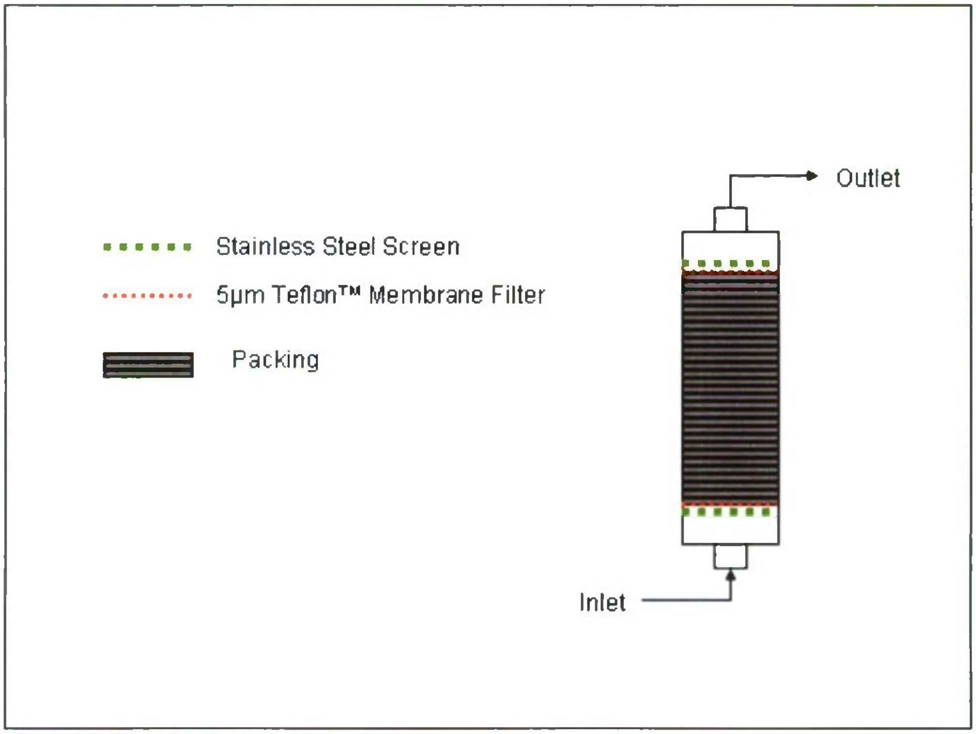

Figure 7. Sketch of adsorption column.

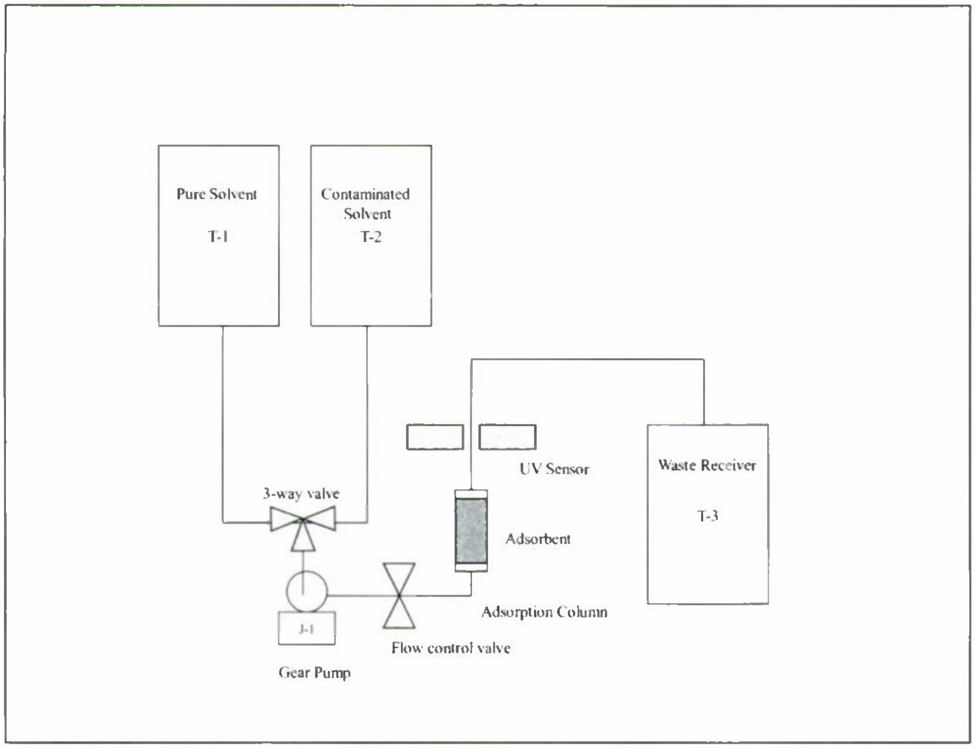

Figure 8. Flow sheet of adsorption system.

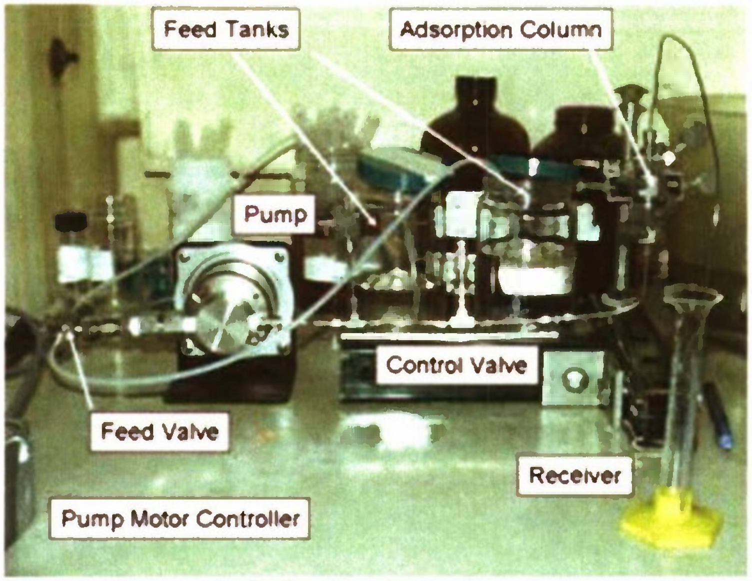

Figure 9. Photograph of adsorption test system.

Columns were initially flushed with clean HFE 7100 to establish a flow rate corresponding to a residence time of 1 ± 0.1 (10%) min. The inlet stream was switched to a contaminated HFE 7100 solution, which typically contains 70 ppm of either CEES or HD. The composition of outlet stream was monitored as a function of time to determine breakthrough time and adsorption capacity. This composition was determined by taking grab samples of this effluent stream and analyzing the composition of these samples by gas chromatography. The CEES samples were assayed on an HP 5890 Gas Chromatograph with a flame ionization detector (FID) and a HP-5 column. The HD samples were assayed using an Agilent 6890N Gas Chromatograph coupled with a Flame Ionization Detector, linked to a 7683 Series Auto-Injector and connected to a chemistry-station. The CEES concentration was determined by measuring the area of the CEES peak which was observed at an effluent time of 2.40 min. The detection limit of this method was 1 ppm. The concentration of HD was determined from an external standard curve, with a mass concentration range of 1 to 5000 ng HD.

4.3 Contaminant Transport (Wicking) Tests

The purpose of the wicking tests was to establish the contaminant transport or wicking ability of different (candidate facing fabrics) CFFs, and to identify those CFF that will most effectively transport the contaminant into the ACF layer of wiper. The results of the wicking tests also indicate the fate of the contaminant within the fabric stacks. This is information that is useful in the design of the wiper.

In each test, a single CFF swatch and a single 50K ACF swatch were punched out with a 1.875 in. or 2.0 in. circular die. The swatches were contaminated at a contamination density of 10 g/m^2. Contamination application volumes were 33.2 μl of DEP, 31.7 μl of CEES, or 20.0 μl of VX, was added to a non-porous surface (aluminum or glass). The CFF swatch, followed by the 50K swatch, followed by a 165 g conical weight, was then placed on top of the contaminant, as shown in Figure 10. After a measured amount of contact time had elapsed, the aluminum weighing tray, the CFF layer, and the 50K layer were placed into individual 2 oz glass jars, each containing 20 mL of chloroform. Each jar was sonicated for 10 min, and a sample was withdrawn for GC analysis. At a minimum, all tests were performed in duplicate. Tests were performed with both dry and HFE 7200 wetted fabric stacks to determine the effect HFE has on the transport of contaminants through a fabric stack. For tests in which the fabric stacks were wetted, the fabric swatches were saturated in HFE 7200 and drip-dried before being placed on top of the contaminant in the weighing tray.

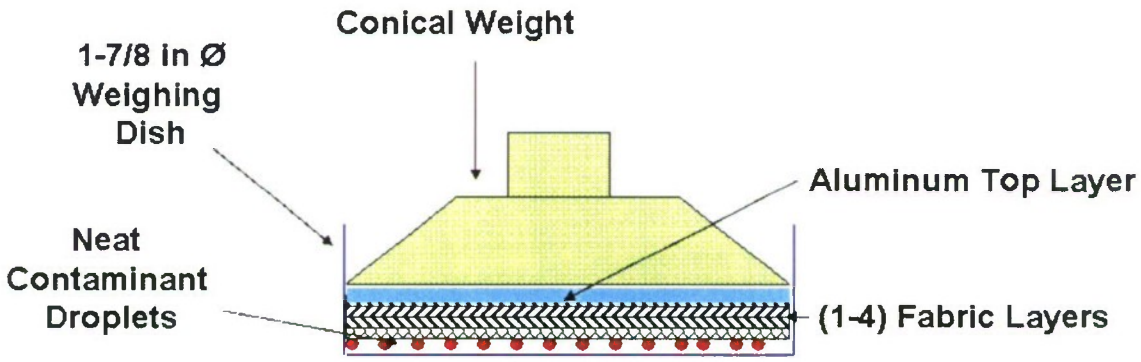

Figure 10. Contaminant transport (wicking) test setup.

The CFFs and simulant combinations that were evaluated are shown in Table 7.

Table 7. Static contaminant (CEES/DEP) transport test matrix.

Candidate Contact Layers		CEES		DEP	
		Dry	HFE-Wetted	Dry	HFE-Wetted
PFG Polyesters	54717	X	X	X	X
	60171	X	X	X	X
	66290	X	X	X	X
	1122	X	X	X	X
	64918	X	X		
PFG Nylons	39278				X
	66190				X
	66165				X
DelStar Polyethylene	P520NAT-A			X	X
	PQ218NAT-E			X	X
Other Fabrics	3M Scotchbrite	X	X	X	X
	Perfect Clean	X	X	X	X
	Polypropylene			X	X
Wicking T-Shirt Fabrics	Dri-Fit (100% Polyester)	X	X		
	Heatgear (80% Polyester, 20% Elastine)	X	X		
	Heatgear (95% Polyester, 5% Elastine)	X	X		

Other tests were conducted to determine if the CFF selected (PFG 39278) interfered with CA transport into the ACF fabric by absorption. Thermogravimetric Analysis (TGA) was employed to measure absorption of HD and GD into the nylon fabric. TGA is an analytical tool useful in determining minute weight changes in relation to temperature change. The TGA consists of a high-precision balance with an attached sample pan. The TGA was from TA Instruments–Waters, LLC, model SDT Q60. In theory, if liquid contaminant absorbs onto a polymer then it should be transferred from the polymer by heat, being careful not to disturb the chemical bonds of the polymer.

In each test, a small circle (approximately 0.4 cm diameter) was punch cut and soaked with the appropriate CA for 2 h at 28 °C. The CFF circles were removed from the CA, washed with isopropyl alcohol, and blotted dry. The contaminated circles were inserted into an alumina basket and positioned into the TGA furnace. For HD, the temperature ramp was from room temperature to 220 °C (HD bp= 217 °C) at 10 °C per min. The GD ramp went from room temperature to 200 °C (GD bp = 198 °C) at a rate of 10 °C.

4.4 Chemical Agent Decontamination Efficacy Tests

4.4.1 Coupon Contamination Procedure

The test coupons were contaminated at the appropriate density (1 gm/m^2 or 10 gm/m^2) by applying 1.0 μL drops of the appropriate chemical agent. Either a Rainin micro-syringe repeating pipette, coupled with a 10 μL glass syringe, or an Eppendorf Repeater® Plus pipette[‡], equipped with an adoptive tip capable of delivering 1.0 μL drops (shown in Figure 11) were used. The drops were placed uniformly

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[‡] The Rainin and Eppendorf pipette was calibrated by weighing 10 drops (10 μL) of distilled water periodically throughout the test and observing the weight; the Rainin measured to be more accurate then the Eppendorf, 10.0 ± 0.0002 gm to 10.0 ± 0.0006 gm, respectively.

over the entire surface of the test material coupon. Consistency in the application of the CA drops and the contamination procedure was maintained by using the same operator throughout the test. Fluctuations in the test material temperature were controlled by employing a slide warmer to maintain the temperature at 24 ± 3 °C. The number of drops per test material coupon was determined prior to the start of testing. The number of drops per coupon depended on the density of the CA and its purity. The following table represents the number of drops delivered, based on coupon size and CA contamination density:

Agent	Density [gm/mL]**	# Drops/Coupon	
		1 g/m^2	10 g/m^2
HD	1.268	2	16
VX	1.008	2	20
GD	1.022	2	20
TGD	~1.022		

** Liquid density is measured at ~25 °C.

The equation for determining the number of 1 L drops per test material coupon was determined from the following simple equation:

$$X = (A \times B/C \times 10) \div (D/100) \qquad \text{Equation 5}$$

where

X = # of drops

A = Contamination density (g/m2)

B = Area of coupon (cm2)

C = Density of the CA (g/mL)

D = Purity (%)

The number of drops was rounded to the nearest 1.0 µL. In the majority of the tests, the CA was allowed to contact the test material coupon surface for 60 min prior to any subsequent operation. During this 60 min dwell period the coupons were covered to reduce evaporation, using Pyrex® round glass cover dish with a 5.7 cm diameter and 0.7 cm height.

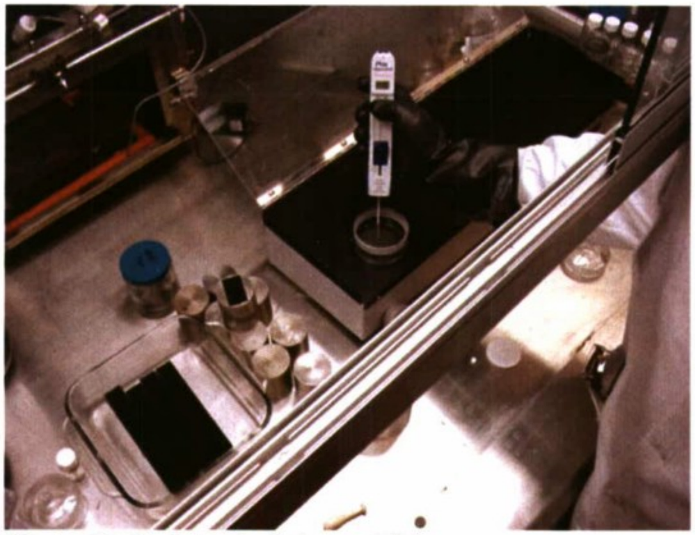

Figure 11. Applying 1 μL drops of CA.

4.4.2 Wiping Procedure (Decontamination)

Using Velcro®, the ACF fabric swatch (coupon) was fixed to the bottom of an aluminum block[§] that weighs approximately 370 gm and has a diameter of 7.62 cm. The ACF fabric coupon was removed from the solvent, and the excess solvent permitted to drip from the fabric for 10 s prior to fixing to the block. The block was then placed directly on the test material coupon, sandwiching the ACF fabric between the block and the test coupon. The block was moved by hand in a circular pattern—first clockwise then counterclockwise in three full rotations. In an attempt to maintain consistency throughout the test, the same operator performed this operation. At no time was force applied to the block by the operator during the rotation. Figure 12 shows the aluminum decontamination wipe block used in the wipe process.

Figure 12. Decontamination operation with aluminum block.

[§] The Rotary-wiping device was used in initial tests, but discontinued in favor of the more expedient aluminum block and the physical operation.

The ACF fabric coupons were soaked in the appropriate solvent prior to use. The fabric coupons were punch cut, normally yielding a surface area of 19.6 cm^2 (5 cm diameter). The multiple fabric layers were achieved by laying one over the other and stapling them together, using a common office stapler (the bonding adhesive described earlier was not included). The coupon was then inserted into a glass jar containing the solvent at least 30 min prior to use. To perform the test, the coupons were removed from the solvent and allowed to drip dry for approximately 30 s at room temperature prior to use. Several weight measurements were taken to determine the approximate weight of the solvent at the approximate point of use in the decontamination process. In addition, the weight loss from the multi-layered coupon was measured over time to determine the evaporation at room temperature and under ambient relative humidity. All of the tests were run at room temperature and ambient relative humidity. Prior to the start of a test, the identification number, material type, and dimensions of the test coupon to be used as a substrate in the test were recorded.

4.4.3 Solubility Measurements and Modified Solvent

The solubility of HD was measured for several neat HFE's and HFE blends. The purpose of these tests was to determine whether the solubility of HD could be increased into the solvent, while not compromising materials compatibility and environmental and personnel safety. Earlier measurements conducted on the solubility of CA, in a variety of commercially available solvents[11] considered material safe, concluded that HD showed the lowest solubility (1.6 v/v %) at room temperature, compared with VX and GD, which were determined to be completely soluble.

In a series of personal communications with Mr. David Hesselroth, from 3M's Performance Materials Division, a list of HFE blends either commercial-off-the-shelf or in development with similar application to simple hydrocarbon sulfides were developed.

This series of tests were performed using a 2 mL gas chromatography (GC) vial as the measurement vessel. One milliliter of the appropriate solvent was added to the GC vial. Then, using a micoliter pipette, HD was added to this volume of solvent. For each solvent, samples with a range of HD loads were created. These solvents ranged in concentration from zero volume percent (no HD addition) to enough added HD to result in the formation of two visible phases. Generally, the minimum incremental amount of HD added was 20 L, which corresponds to a concentration increase of two volume percent, based on the initial amount of solvent present. Following the addition of HD, the vials were capped and shaken and then allowed to settle for at least 18 h. The vials were placed on a surface to maintain a temperature of 24 ± 1 °C.

4.4.4 M8 Paper Test

The M8 paper test was performed to address one of the Key Performance Parameters (KPP) within the JSPID ORD, as sufficient to meet the decontamination objective for Immediate/Operational Decontamination. M8 Paper, in the presence of liquid CA, should produce a color indicating the presence of liquid CA greater than 0.02 mL drop.

In setting up the test, the only operational information obtained for the use of M8 Paper was located at www.ArmyStudyGuide.com (accessed between April 2001 and December 2004). This website indicated that the paper should be blotted and not rubbed over the surface to be studied. The material test coupons were contaminated at a starting density of 1 or 10 g/m^2, following the same procedure outlined in Section 4.4.1. Immediately following the 60 min incubation period, the ACFF wipe or M295 Kit decontamination procedure was started. Immediately following the wiping procedure a sheet of M8 Paper (8.7 x 6.4 cm) was applied directly to the test coupon material surface. This was immediately followed by the application of a piece of aluminum foil (5.1 cm diameter circle) then a 1 kg weight. The M8 paper contacted the material test coupon for 15 min and was then removed for observation. The recommended procedure for determining whether the M8 Paper detected CA was visual observation. To aid in this detection effort, a 3x magnifying glass and 125 W Halogen lamp were used. The M8 paper was observed initially following removal and then again 15 min later.

4.4.5 Solvent Extraction and Gas Chromatography Analysis

4.4.5.1 Mass Removed by Wiper at Room Temperature

In the majority of the agent wiping tests conducted during the study, the amount of post-wipe residual agent remaining on the surface of a test coupon was determined by solvent extraction and gas chromatography (GC) analysis.

The amount of chemical agent (CA) removed by the decontamination wipe was determined by the measurement of the mass of the CA initially applied to a surface (coupon) and compared to the residual mass remaining directly after decontamination. The mass of the CA removed or remaining was determined by solvent extraction using chloroform and CA mass analysis by GC.

After completion of the wiping procedure, the material coupon was inserted into a glass weighing bottle containing a pre-measured amount of solvent[**] for 60 min at room temperature. The ACF fabric coupon was removed to a 40 mL volatile organic analysis (AOA) vial, with a Teflon® seal cap, containing a pre-measured amount of chloroform. It was then immediately inserted into a sonicating[††] water bath for 60 min. The 20.26 cm^2 material coupon was inserted into a Kimble glass weigh bottle (70 x 33 mm) then extraction solvent was added. The solvent temperature was uniformly maintained during the extraction period by setting the glass bottles[‡‡] onto a Lab Line Slide Warmer set to 25 °C. The appropriate volume of extraction solvent was quantitatively added to each sampling bottle/vial using a variable-volume Brand Dispensette® Organic digital pipette, connected to the bottle of solvent. At the end of the extraction period an aliquot of the extraction solvent was removed, it was volumetrically diluted if required, and transferred to a glass gas chromatography autosampler vial for analysis. The chemical agent was qualitatively measured (comparative retention time, CA test vs. CA standard) and quantitatively measured (mass measurement versus external standard curve) using an Agilent 6890N Gas Chromatograph coupled with a Flame Ionization Detector, linked to a 7683 Series Auto-Injector and connected to a chemistry station. The column was a 0.32 mm x 30 m, HP-5 (5% phenyl methyl siloxane) wide-bore capillary with a film thickness of 0.25 µm. The GC was calibrated over a mass range of 5 ng to 5000 ng for HD, 4 ng to 4000 ng for VX, and 3 ng to 3000 ng for GD and TGD. The GC/FID parameters used in the analysis of the HD, VX, GD, and TGD are shown in Table 8 below.

[**] In most tests Optima®-grade chloroform was used; with polycarbonate HPLC-grade 2-propanol replaced the chloroform.

[††] The sonicating water bath was from Crest Ultrsonics and was equipped to deliver a constant 132 KHz. In a typical 60 min extraction, the water bath temperature was measured with a thermometer and calibrated by ECBC Calibration, starting at 24 ± 1 °C and ending at 380 ± 4 °C.

[‡‡] The seal made with the ground-glass lid was determined to be acceptable as the weight loss for 20 mL of chloroform at room temperature for 60 min. was measured at 0 g.

Table 8. GC test parameters.

GC/FID Parameters Used in the Analysis of the HD, VX, GD anf TGD Solvent Extracts			
Parameter	**HD**	**VX**	**GD/TGD**
Carrier Gas	Helium (He)	He	He
Injection Port Temp. [^{0}C]	230	230	225
Initial Temp. [^{0}C]	80	80	60
Initial Hold Time [min]	2	2	1
Ramping Rate [^{0}C /min]	20	20	20
Final Column Temp [^{0}C]	220	250	240
Final Hold Time [min]	1	2	1
Injection Volume [µL]	1	1	1.0
Detector Temp [^{0}C]	250	250	210
Split Ratio	2:1	2:1	2:1
Retention Time [min]	~ 5.8	~ 11.4	~ 8.6

Another set of tests was run using mass removal analysis (extraction), but this time comparing the removal efficiency for the ACF fabric wipe an initial contamination density of 1 g/m^2 to 10 g/m^2. Basically, these tests compared the percent of the initial mass removed from the two conditions in a side-by-side test.

4.4.6 Chemical Agent Mass Removed by Wiper at Elevated and Reduced Surface Temperature

In other tests the test material coupon temperature was either elevated or reduced to show the effect these conditions had on decontamination efficacy. The reduced temperature was achieved by supporting the test material coupons directly above a layer of ice. By resting the coupons on 0.25 in. rubber discs, the desired surface temperature (14 °C[§§]) could be maintained for the duration of the test, approximately 60 min. The test material coupons were spiked with HD at an initial contamination density of 10 g/m^2, placed into a covered glass weighing vial, and inserted onto the rubber discs over the ice. The coupon surface temperature was monitored using a Type-J surface thermocouple connected to a multi-channel input with digital display. The initial HD contact with the coupon was 60 min. The temperature of the surface was constant within ±2 °C for the duration of the initial contact time. At the conclusion of the 60 min initial contact period, the coupons were removed from the layer of ice and immediately decontaminated using the ACF fabric wiping procedure described in Section 4.4.2.

The elevated coupon surface temperature was achieved using a conduction oven and monitoring the coupon surface temperature with a Type-J surface thermocouple. By some trial and error, the oven was set to achieve the desired temperature (49 °C) for the duration of the test. The coupons were spiked with HD at an initial contamination density of 10 g/m^2, covered and inserted into the oven for 60 min. The temperature of the coupon was constant within ±1 °C. During the first attempt at the elevated temperature test, when the coupons were removed from the oven following the 60 min CA contact period, it was observed that HD condensate formed on the underside of the ground glass weighing vial cover (lid), confirmed by M8 paper. It was decided to reduce the initial contamination contact period to the time it took to achieve the desired temperature of 49 °C and hold it for a period of 5 min.

4.4.7 Vapor Monitoring

Time-resolved near real time monitoring of the spent ACF fabric wipe was conducted by solid-sorbent monitoring. Three analytical systems, connected to vapor cups capable of monitoring the effluent air stream for the CA in use, were employed. The first system used a single vapor cup, connected by a short length of heated Teflon® tubing to a Miniature Continuous Air-Monitoring System

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[§§] The freezing point for HD is 14.4 °C, as determined by Army Field Manual 3-9.

(MINICAMS) Flame Photometric Detector. The MINICAMS near real-time vapor system is shown in Figure 13. The vapor cup was machined from #305 stainless steel. Located at either end are the inlet and outlet, both circular 0.125 in. holes, connected by Swagelok® compression fittings. The cup was fashioned in two parts, a bed where the test sample (coupon) was inserted, and a lid that was locked in place using a rotational cam, which pinches the two halves together, making a seal with a Teflon® o-ring. The headspace volume of the cup is 60 cm^3, with a measured height above the coupon of 1.3 cm.

The effluent outlet stream was connected to the MINICAMS by 0.125 in OD Teflon TFE tubing, passing through Swagelok stainless steel needle valves (one for coarse adjustment and the other for fine adjustment). The needles valves were used to control the pulled air flow rate through the cup and monitored using a mass flow monitor from Fisher Scientific. The collected MINICAMS samples were analyzed directly by the MINICAMS. The MINICAMS collected the CA using a solid sorbent, which was then thermally desorbed periodically (cycle time) onto the detector. The mass of the CA was measured from an external standard curve and calculated by a linear regression analysis. The four-point calibration curve process occurred every 10–15 days from fresh standards, and a single mass point standard was measured daily to ensure the accuracy and consistency for the instrument quantitation.

Figure 13. MINICAMS near real-time monitoring system, single vapor cup.

The second system employed a bank of vapor cups (six), which connected the effluent to a solid sorbent tube. The same vapor cup as described for the MINICAMS system was used. The CA-contaminated effluent pulled from the cup passed through and was collected onto the solid sorbent tube. The tube(s) was manually changed at timed intervals, and analyzed by either a Perkin-Elmer model 7880 Thermal Desorption Analyzer, which was coupled to an Agilent 6890 Plus Gas Chromatograph and Flame Ionization Detector, or a Marks Thermal Desorption System, connected to an Agilent 6890N, which was coupled to a Mass Selected Detector. The mass of CA was determined by linear regression calculation to a four to six-point external standard curve, generally run immediately prior to the test coupon samples. The airflow pulled through the vapor cup was maintained at a constant rate and measured by a Brooks Instrument mass flow controller, model 5850, and coupled output readout device. The bank of vapor cups is shown in Figure 14.

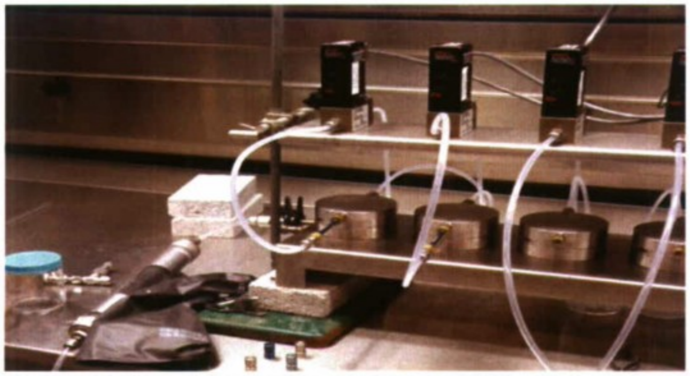


Figure 14. Vapor cup system connected to sorbent tubes.

The results obtained from the time-resolved, near real-time vapor monitors were recorded as a mass per volume concentration of air pulled across the solid sorbent. The final results are reported as milligrams per cubic meters. The air that was pulled through the vapor cup was conditioned at the inlet by a charcoal filter. All of the vapor monitoring was done a room temperature and ambient relative humidity (RH). The room temperature and RH were measured using a Fisher Scientific digital meter daily. The temperature was 24 ± 3 °C and ranged from 2 to 40 RH. The airflow through the cup was maintained at a rate of 300 mL/min.

4.4.8 Wiping Efficacy Tests

The purpose of the wiping efficacy tests was to establish the level of cleanliness that can be attained by wiping a contaminated substrate with wipers that contain actviated carbon fiber fabrics.

The developmental tests were exploratory to establish the effect of wiper construction on the removal of a contaminant from a flat stainless steel surface. Baseline validation tests were then performed with the wipers deemed to be the most effective in these developmental tests, and in the shedding and wicking tests. Chosen were the PFG 39278 nylon faced 50K wipes, as well as wipes faced with a supposedly equivalent nylon fabric, PFG 66378. Once it was established that the two wipers were equivalent, further wiping tests were performed to remove contaminant from a flat stainless steel plate in which the effects of the following were examined: (1) multiple wiping cycles, (2) contaminant composition, and (3) solvent composition. These tests were performed to establish an optimum surface-cleaning procedure, and to determine the contaminant removal capabilities of a decontamination wiper.

In a normal operating environment, it will be necessary to decontaminate objects that are geometrically more complex than a flat plate. These items will be more difficult to decontaminate than a flat plate because the wiper may not be able to come into contact with all of the contaminated areas. Additional testing was performed to determine the efficacy of removing a contaminant from a geometrically complex object. The testing was done using two methods: (1) by wiping with a multi-layered, HFE-wetted ACF laminate wiper, and (2) by first spraying the object with an aerosolized HFE then wiping with a dry ACF laminate wiper.

During each wiping efficacy test, a wiper or multiple wipers were used to remove DEP from three stainless steel sheets, which were initially contaminated at a level of 10 g/m^2. After wiping, the stainless steel sheets were extracted individually to determine the amount of residual DEP remaining on each sheet. Each layer of the used wiper or wipers was extracted to determine the fate of DEP within the wiper, and to obtain a mass balance. The following procedures were used in each test.

4.4.8.1 Wiping Procedure

1. Secure three (3) 6 in. square sheets of 2 mil stainless steel foil to a flat surface using duct tape.

2. Contaminate each sheet evenly with 208 µL (233 mg) of DEP, which is equivalent to a contaminant load of 10 g/m^2.

3. Before wiping, submerge the wiper in a jar of HFE 7200 and allow it to drip-dry.

4. Wipe each of the three sheets for 20 s using same wipe.

5. After wiping, separate the wiper into its constituent layers, place each layer individually into a 2 oz jar and seal the jar.

6. For tests in which multiple wipes were performed, repeat steps 3 through 5 for each additional wiping pass.

7. After the appropriate number of wiping operations has been completed, carry out the following extraction methods for both the wiper layers and the wiped sheet.

4.4.8.2 Extraction Procedure for Wiped Sheet

1. After wiping is complete, place each sheet into separate 500 mL screw cap jars and then add 160 mL of chloroform to each.

2. Sonicate each jar for 5 min in a 132 kHz ultrasonic bath.

3. Withdraw a sample from each jar and perform GC analysis to determine the amount of residual contaminant left on each sheet.

4.4.8.3 Extraction Procedure for Wiper Layers

1. At the conclusion of the wiping procedure, each layer of the wipers used in each test should be sealed in a 2 oz glass jar.

2. Add 40 mL of chloroform to each jar and then sonicate each jar for 5 min in a 132 kHz ultrasonic bath.

3. Prepare a 10:1 dilution of each sample and transfer an aliquot to a 2 mL GC vial.

4. Perform GC analysis to determine the amount of residual DEP in each wiper layer.

All wiping efficacy tests were performed using DEP rather than CEES for two primary reasons:

1. The CEES was much more volatile than DEP, and there were concerns that CEES evaporation could influence the wiping efficacy results.

2. Because of its higher viscosity, DEP wicks much less readily than CEES through the layers of a wiper and represents the tougher challenge.

Test Matrix: The wipers used and the number of wiping passes performed for the wiping efficacy tests are shown in Table 9. Note that 50K was used exclusively as the ACF, due to its low shedding potential.

Table 9. Wiping efficacy test matrix.

Test #	Wiper Configuration			Contact Layer Laminanted to ACF 1?	Number of Wipes		
	Contact Layer	ACF 1	ACF 2		Wiped Once	Wiped Twice	Wiped Three Times
1		50K		N/A	X		
2		50K	50K	N/A	X		
3	54717	50K		No	X		
4	39278	50K		No	X		
5	39278	50K	50K	No	X		
6	39278	50K		Yes	X		
7	39278	50K	50K	Yes	X	X	X

4.4.9 Validation Baseline Wiping Efficacy Tests

The test parameters, shown in the test matrix presented in Table 10, include the candidate facing fabric (CFF), lamination, whether the wipe was dry or HFE wetted, and the number of times the plates were wiped. In each test, the appropriate wipe materials were cut into 3 in. squares, layered on top of each other, and fastened to a wiping block. Each CFF was layered with two 50K layers, and each CFF laminate was layered with one 50K layer. Three 6 in. square stainless steel sheets were cut and taped to the table in a row. Each sheet was evenly contaminated with 208 μL of DEP. This was equivalent to 10 g/m^2, the standard load currently used for contact exposure level decontamination testing by the U.S. military. The decontamination wipe was then saturated with HFE 7200 or left dry, and each stainless steel sheet was wiped for 20 s, one after another. After wiping, each layer of the wipe was separated and placed in different 2 oz jars with 50 mL of chloroform. All wiping was done using a counterclockwise, circular motion over the entire area of the sheets. After the wiping was complete, each steel sheet was removed from the table, put into separate 16 oz jars with 100 mL of chloroform, and labeled according to the order in which they were wiped. The jars were then sonicated for 5 min in a 132 kHz ultrasonic bath. Once removed, a sample was taken from each jar and analyzed by gas chromatography.

Table 10. Test matrix for wiping test.

Test #	Candidate Facing Fabric (CFF)	Laminated (y/n)	Wetted (y/n)
1	39278	No	No
2	66387		
3	39278	Yes	
4	66387		
5	39278	No	Yes
6	66387		
7	39278	Yes	
8	66387		

4.4.9.1 Effect of Multiple Wipe Cycles

Additional testing was done with 39278/50K laminate wipers to determine the level of decontamination that could be achieved using multiple (three) wipe cycles. The procedure for these wiping tests was identical to the procedure for the baseline validation wiping tests except that:

- All tests were performed using 39278/50K laminate, transfer solvent HFE 7200, and DEP.
- Three wipe cycles were performed on the stainless steel sheets.

4.4.10 Wiping Efficacy Tests for Removal of Other Contaminants

Motor Oil: All wiping efficacy tests presented thus far in this report were performed with DEP as the contaminant. The decontamination should be able to remove other materials from surfaces, including those with different physical properties such as viscosity and HFE solubility. One contaminant that is likely to be found in an operational environment is motor oil, which is much more chemically complex and viscous than DEP. Motor oils are also only slightly soluble in HFEs. Wiping tests were performed with the following three motor oils: SAE 40, SAE 10W-30, and SAE 80W-85W-90.

The procedure was similar to that used for baseline validation wiping tests with the following exceptions:

- Two wipe cycles were performed.
- PFG 66387/50K laminate wipers wetted with HFE 7200 were used for all tests.
- All tests were performed using the same volume of contaminant. In all cases, the metal sheets were contaminated with 208 µL for a complete contaminant load of 624 µL. This volume was equivalent to the volume of DEP that would yield a 10 g/m^2 contamination load, the standard load currently used for contact exposure level decontamination testing by the US military.

Other Contaminants (Oils): The objective of these tests was to determine the efficacy of the decontamination wipe for contaminants with different chemical and physical properties than the DEP and motor oils. Citroflex (Butyl Citrate), Krytox AZ Oil (a fluorinated polyether), and PDMS (methylsilicone oil) were examples of contaminants with different viscosities and composition. The PDMS; Krytox oil; and Barrierta L55/2, a fluorinated grease, are usually very difficult to remove from a surface. Some limited testing was also performed with a fruit tree spray to demonstrate the removal of a material that contained toxic industrial chemicals. Because a different analytical procedure was used for the removal of Barrierta L55/2 oil and the fruit tree spray, these tests are discussed separately.

The procedure was identical to the validation baseline wiping test procedure except that:

- The tests were performed using PFG66387/50K laminate wipes wetted with HFE 7200.
- Three wipe cycles were used for all tests.
- All tests were performed using the same volume of contaminant. In all cases the metal sheets were contaminated with 208 µL for a complete contaminant load of 624 µL. This volume is equivalent to the volume of DEP that would yield a 10 g/m^2

contamination load, the standard load currently used for contact exposure level decontamination testing by the US military.

- Krytox-fluorinated oil was insoluble in chloroform so all GC analysis was performed using HFE 7200 as the solvent.

Other Contaminants (Barrierta L55/2 Grease Wiping Tests): Barrierta L55/2 is a highly fluorinated grease that is normally difficult to remove from a surface. The objective of these tests was to determine the efficacy of the decontamination wipe for another contaminant that is normally difficult to remove.

The procedure for these wiping tests was significantly from that used for the validation baseline wiping tests because of the unusual chemical and physical properties of the contaminant. The procedure was changed in several important ways:

- The tests were performed using PFG 66387/50K laminate, wetted with HFE 7200, using three wipe cycles.

- The plates were evenly contaminated with 0.2323g of the Barrierta L55/2 grease. This was equivalent to 10 g/m^2, the standard load currently used for contact exposure level decontamination testing by the U.S. military.

- The Barrierta grease is insoluble in chloroform so that extraction for analysis was performed with HFE 7200.

- The thickening agent in the grease is insoluble in HFE 7200, so after being dissolved in the HFE 7200, the solution was filtered using a 0.22 μm polypropylene syringe filter.

- This filtrate could not to be analyzed using GC analysis. UV/VIS analysis was used instead.

Other Contaminants (Fruit Tree Spray): Tests were performed with Bonide Complete Fruit Tree Spray, an insecticide commercially available from a local nursery (http://www.bonideproducts.com/). The contents of the spray are shown in Table 11. The toxic industrial chemicals include Captan, Malathion and Carbaryl. The purpose of this test was to show the effectiveness of the wipe on the toxic industrial chemicals, such as insecticides and pesticides.

Table 11. Contents of bonide complete fruit tree spray.

Components of Bonide Complete Fruit Tree Spray					
Name	**CAS #**	**Melting Point (°C)**	**Boiling Point (°C)**	**Ambient Phase**	**Percentage**
Captan	133-06-2	178		Solid	**11.76%**
Related Derivatives					**0.24%**
Malathion	121-75-53	2.85	156	Liquid	**6.00%**
Carbaryl	63-25-2	142	315	Solid	**0.30%**
Other Ingredients					**81.70%**
Total					**100.00%**

The Fruit Tree Spray consists of mostly solvent. In order to show that the ACF can adsorb very large amounts of toxins, the spray was concentrated. A volume of Fruit Tree Spray was placed uncapped in a hood for four days, where approximately 57% of the initial weight evaporated. Based on the data taken from the container label (shown in the list below), the concentrate contains about 42% active ingredients, assuming no evaporation of these ingredients. The concentrate consisted of two layers that were easily remixed by shaking. After remixing before each use, this concentrated Fruit Tree Spray was used in all subsequent tests.

- Captan, Carbaryl, and Malathion, the three main ingredients of interest, all have relatively high boiling points.
- Due to the low volatility, residual contamination was assessed by rinsing the test object with a solvent, which was then analyzed for contaminant content.

Six inch square (6 x 6 in.) aluminum plates and two three inch square (3 x 3 in.) coupons of the ACF wiper were cut. The plates were washed with approximately 5 mL of chloroform, and the initial weights were taken. The clean side of a plate was contaminated with 174 μL of Concentrated Fruit Tree Spray, weighed, and allowed to passively dry in the hood. The ACF wipe coupon was wetted with HFE 7200 then used to wipe the freshly contaminated plate, using the corners and center of the wipe evenly.

The plates were extracted several times in approximately 31 mL of chloroform. All of the collected sample extracts were then analyzed with a Shimadzu 1201 UV/VIS spectrometer at 267 nm. A calibration curve, presented in Figure 15, was constructed using known concentrations of concentrated Fruit Tree Spray in Chloroform.

The equation given for the trend line in Figure 15 was used to determine the concentration of concentrated fruit tree spray in a test sample. If the concentration is well above 200 ppm, the solution must be diluted and rescanned. Using the weight of the sample and the density of Chloroform, it is possible to determine the volume of contaminant in the solution.

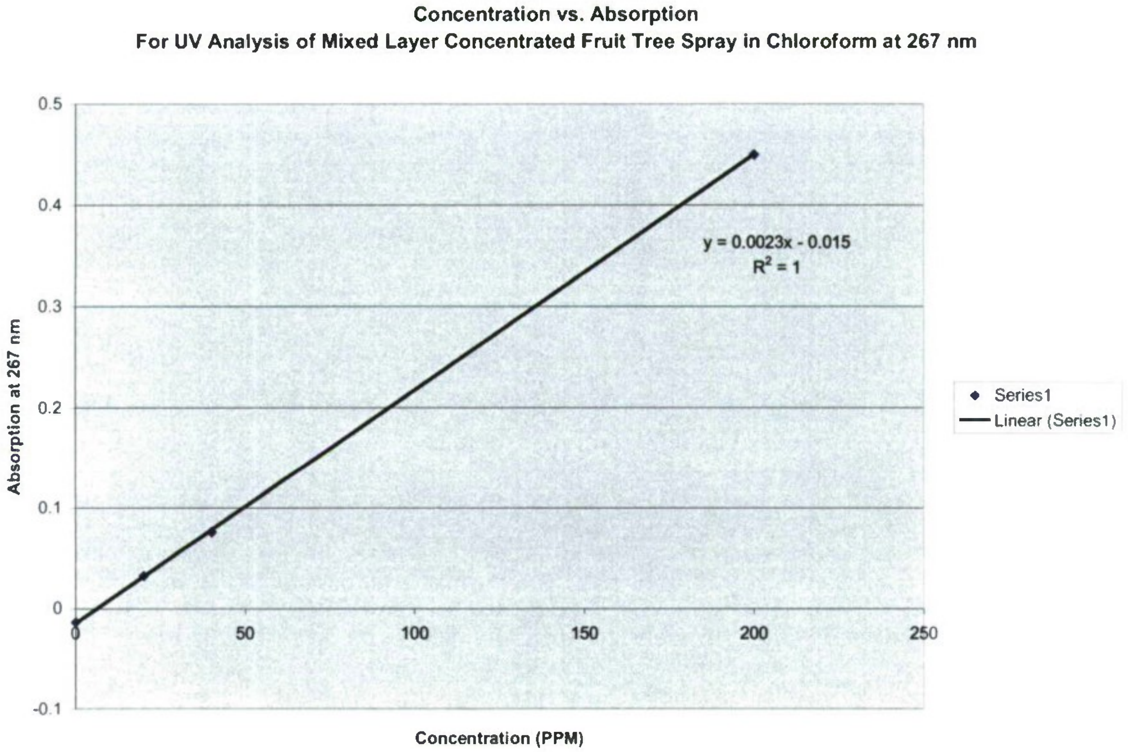


Figure 15. Calibration curve for concentrated Fruit Tree Spray in chloroform.

4.4.11 Wiping Efficacy Test for Objects with Complex Geometries

Decontaminating a geometrically complex object is inherently more difficult than decontaminating a flat plate because the wiper may not be able to come into contact with all of the contaminated areas. The objective of these tests was to determine the wiping efficacy of a multi-layered HFE-wetted ACF laminate wiper on a geometrically complex object such as the twelve (12) button keypad shown in Figure 16 (Part K3350NS, MGR Industries, Inc., Ft. Collins, CO). This keypad was used as a model complex object for testing because it has a highly complex geometry that incorporates ridges and sharp edges, as well as wells around the keys where a decontamination wipe would have a hard time reaching. It is also an object of convenient size for laboratory work (4.5 in. long x 3.75 in. wide x 0.5 in. deep). Such keypads are also often found on military radios.

Figure 16. Assembled keypad.

To show how the location of the contamination affects the decontamination results, wiping efficacy tests were performed in which different areas of the keypad were selectively contaminated. The following areas were contaminated in three tests:

1. 100% surface contamination
2. 50% well and 50% surface contamination
3. 100% well contamination

Before each test the keypad was disassembled, and a protective layer of polyethylene was placed between the keys and the rubber keypad to prevent absorption of the contaminant by the rubber key support as shown in Figure 17 through Figure 19.

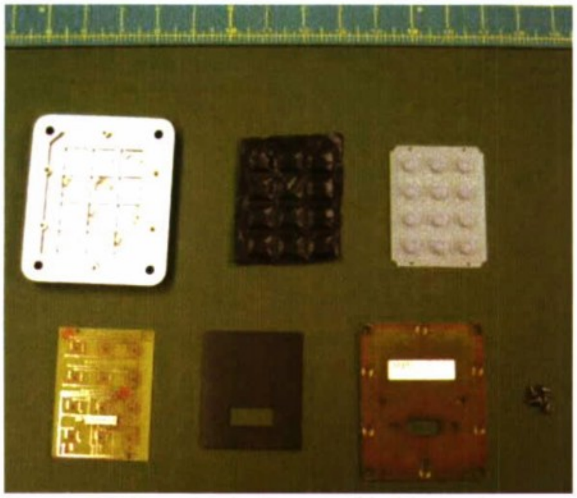


Figure 17. Completely disassembled keypad.

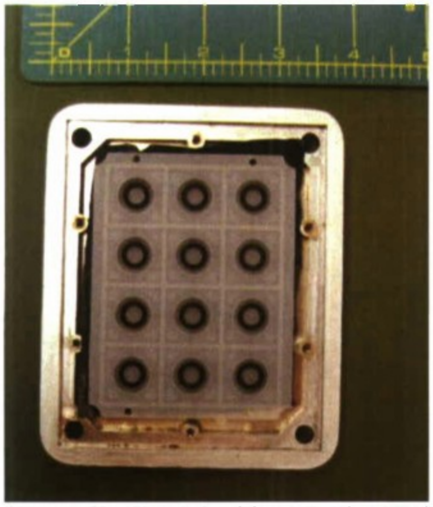


Figure 18. Keypad with protective PE layer and rubber key support in place.

Figure 19. Back of assembled keypad.

In each trial, 3.5 in. square pieces of PFG39278-50K laminate, an additional piece of 50K ACF, and Tyvek 1443R were fastened together with staples. The 12-button keypad was contaminated with 91 µL of DEP to attain $10 g/m^2$, which is the standard load currently used by the US military for contact exposure level decontamination testing. Different regions of the keypad were contaminated in three different sets of tests. In the first test, all the contaminant was placed on the surface of the keypad. In the second test, all the contaminant was put in the wells of the keypad. In the third test, 50% of the contaminant was put in the wells and 50% was placed on the surface. After contamination the keypad was wiped for exactly 30 s. The wipe was first saturated with HFE 7200 then wiped in a counterclockwise, circular motion across the surface of the keypad.

After the 30 s, the wipe was put into a 200 mL jar with 100 mL of chloroform. The keypad was disassembled, and the metal portions of the keypad (keys and frame) were placed in an 8 qt, stainless steel container with 230 mL of chloroform. All the metal pieces were completely submerged in solvent. The protective layer of polyethylene (PE) was put in a 100 mL screw-cap jar with 50 mL of chloroform. All containers were sonicated at room temperature for 3 min at 132 kHz. Liquid samples from each container were taken for GC analysis. The decontamination wipe sample was diluted 10:1

before GC analysis. In order to assure the keypad was clean for future tests, it was sonicated a second time in 230 mL of fresh chloroform. A sample of this was also taken for GC analysis to obtain a more accurate mass balance. A third chloroform rinse was found to be unnecessary because the keypad was sufficiently clean after two immersions. All tests were performed in duplicate.

4.4.12 Spray and Wipe Testing

The decontamination of complex objects can be problematic when considering the use of wipes. As discussed in the previous section, while a decontamination wipe can effectively remove contaminant from the surface of a complex object, when the contaminant is located in a hard to reach area, such as the well of a keypad, a wiper is ineffective. A different strategy was needed to clean complex objects. This strategy used a pressurized spray of Novec HFE 7100 to flush contaminants from deep crevices. Aerosol cans of this material were sold commercially by 3M as Novec Contact Cleaner. Theoretically, the spray should be able to get into the wells of the keypad and remove the contaminant through flooding and displacement.

Initial spraying tests were carried out to determine the effect of increasing spray cycles on the total contaminant removal from a keypad.

The keypad was disassembled and a protective layer of polyethylene was placed between the keys and the rubber keypad to prevent absorption of the contaminant by the rubber as shown in Figure 20, Figure 21, and Figure 22. With the protective layer in place, each key was contaminated with one drop of DEP, dispensed with a 50 μL syringe. The keypad was contaminated with a total of 50 μL of DEP, a simulant of VX nerve agent.

The contaminated keypad was then placed in a glass baking dish at approximately a 45° angle, as shown in Figure 23. The aerosol can of Novec Contact Cleaner was weighed before proceeding. The keypad was then sprayed with the cleaner. The keypad was subjected to varying numbers of spray cycles (1, 2, or 3). A cycle was defined as spraying each row of the keypad, going from the top of the keypad down to the bottom, without stopping. The directions on the HFE 7100 can were followed in all spraying procedures.

After spraying, the aerosol can was reweighed and the keypad was removed from its position to allow any remaining solvent to more efficiently drip out of the keypad before disassembly. Once the dripping stopped, the run-off was collected in a 2 oz jar and its volume was measured. A sample of this run-off was diluted 10:1 for analysis by GC. The keypad was then disassembled. The polyethylene layer was placed in a 100 mL jar with 50 mL of chloroform. The metal pieces of the keypad were placed in a metal container with 230 mL of chloroform. Both vessels were sonicated for 3 min, and liquid samples were taken for GC analysis. The metal pieces were subsequently put into 230 mL of clean chloroform and sonicated a second time in order to ensure cleanliness for the next experiment. A sample of this chloroform was also taken for GC analysis. Before starting the next trial, the baking dish was wiped with chloroform to remove any DEP residue that may have remained in the dish from the previous trial. Each trial was done in duplicate.

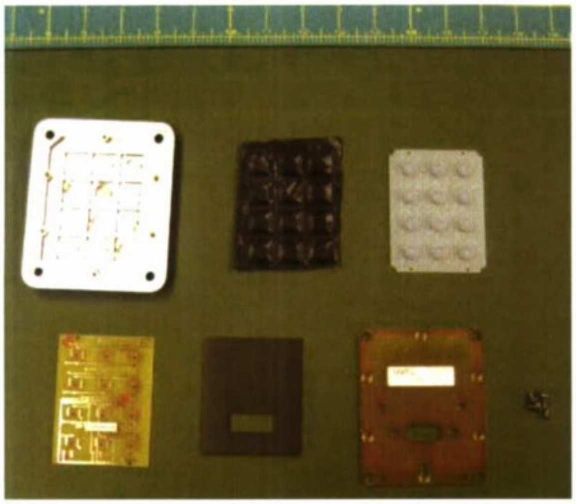


Figure 20. Completely disassembled keypad.

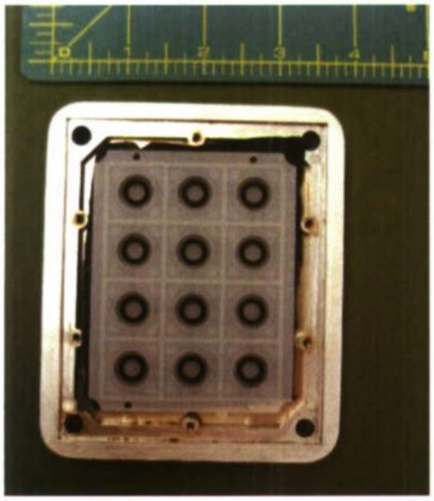


Figure 21. Keypad with protective PE layer and rubber keypad in place.

Figure 22. Back of assembled keypad.

Figure 23. Keypad at 45° angle in baking dish.

4.4.13 Contaminant Off-Gassing from Activated Carbon Fabrics

Compared to nonadsorbent fabrics, which could also be used to remove hazardous liquids contaminants from solid surfaces, the adsorptive properties of the activated carbon fabrics mitigate off-gassing from used wipes. When the used wipes are repackaged in a sealable hermetic envelope, the adsorptive properties provide a redundant means of agent isolation. Therefore, the used wipes can be safely handled until they are destroyed by incineration, for example, or decontaminated by standard means, such as immersion in bleach solution.

Off-gassing tests were performed with CEES and Bonide Fruit Tree Spray at the contractor facility, and with various CWA of interest. The purpose of these tests was to establish the effect of contaminant loading on swatches of activated carbon fabrics. Specifically the rate and extent of evaporation of a contaminant into an air stream flowing over a swatch were evaluated.

4.4.14 Off-Gassing of CEES

Off-gassing tests were performed with 1.75 in. (4.5 cm) diameter discs of the following Calgon Zorflex activated carbon fabrics: 50K knitted fabric, 100 micro woven fabric, and 100 meso woven fabric (Table 12). The volume average pore size and specific surface area of each of these fabrics are as follows:

Table 12. Calgon Zorflex activated carbon fabrics.

Fabric	50K	100 micro	100 meso
Vol-Avg. Pore Diameter, Å	6	6	29
Specific Surface Area, m^2/g	1100	1360	655
Total Pore volume, cc/g	0.72	0.75	0.81

A measured volume of CEES was added to 1 mL of HFE 7100. This solution was added to a fabric disc already placed in an off-gassing cell. The CEES concentration was adjusted to obtain CEES fabric loadings of 3.24 wt%, 10 wt%, or 20 wt%.

Off-gassing tests were also performed at CEES loadings of 3.24 wt% and 10 wt%, with the M 100 alumina powder from the M 295 decontamination kit. In these tests, the powder was sprinkled in a thin layer on the bottom of the off-gassing cell before adding 1 mL of the appropriate CEES/HFE 7100 solution. This powder had a specific surface area of 260 m^2/g, and a total pore volume of 0.701 cc/g. It was found to be primarily mesoporous.

The off-gassing cell containing the contaminated coupon was sealed, and after a dwell time of 30 min, placed in the off-gassing apparatus shown in Figure 24 and Figure 25. In this system, nitrogen gas was passed over the coupon at a constant flow rate of 500 mL/min for 1 h, at room temperature. The cell effluent gas passed (bubbled) through a liquid impinger containing 20 mL of GC grade 2-propanol (Aldrich 34863) to strip the volatilized CEES from the gas stream. The gas flow was interrupted periodically (5, 10, 15, 20, 30, 40, and 60 min) to allow the liquid in the impinger to be replaced with fresh solvent. The impingers were reweighed after removal to account for any loss of solvent by evaporation. A 2 mL sample of each scrubbing liquid was then analyzed for CEES by gas chromatography with a flame ionization detector (GC/FID).

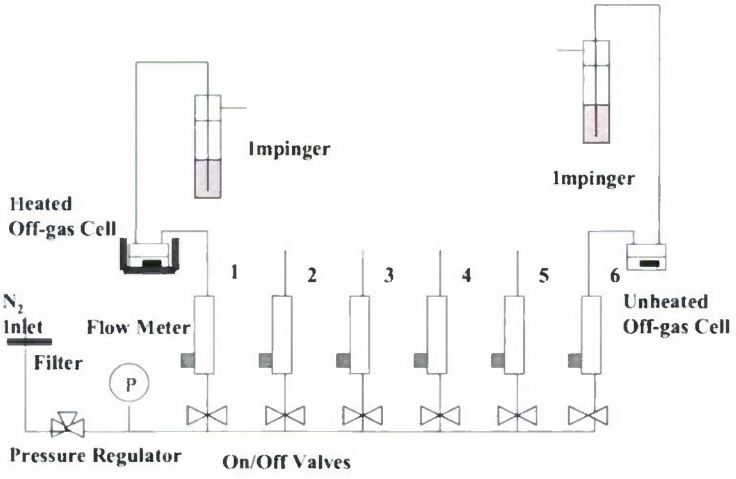


Figure 24. Flow Diagram of Coupon Off-Gassing Test System

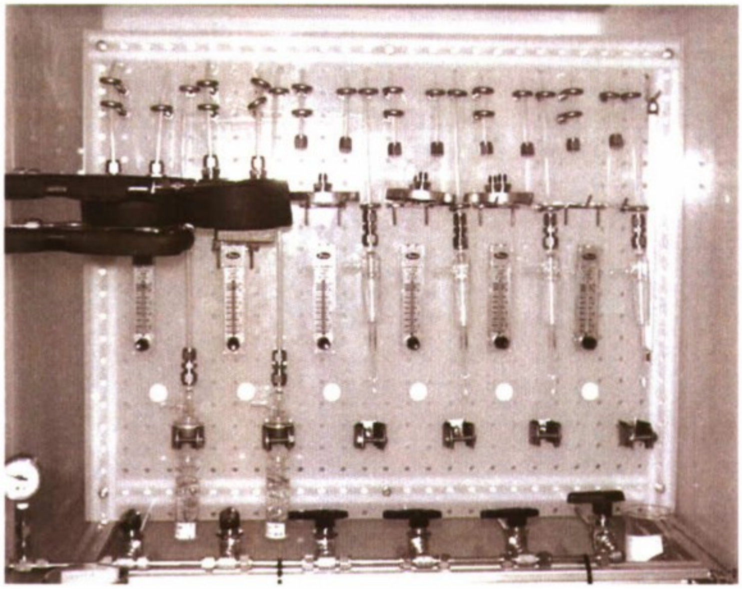


Figure 25. CEES Off-Gassing Test Stand

4.4.15 Off-Gassing of Bonide Fruit Tree Spray

Off-gassing from the wipes used in the Bonide Fruit Tree Spray wiping experiments was evaluated in the apparatus just described above. The major differences in procedure were the use of chloroform (instead of 2-propanol) as the gas-stripping liquid and of UV/Vis adsorption (instead of GC) as the method of analysis of the stripping solvent. This method of analysis was already described in the wiping efficacy test section. In these off-gassing tests, the receiving vessels were changed after 15, 30, and 60 min. After 90 min, the test was stopped.

4.4.16 Ballooning of Storage Bags Containing HFE-Wetted ACF Fabric Wipes at 71 °C

One of the design requirements for a CA wiper is that it needs to be stored at 71 °C for an extended period of time without any loss in performance. Based upon the results of the development and validation tests, the optimal wiper design was an HFE-wetted ACF laminate, enclosed in a resealable pouch. At this temperature, the vapor pressures of HFE 7200 and HFE 7300 are 87.7 and 40.6 kPa respectively, or less than one atmosphere (101 kPA). Tests have shown that the pouches containing HFE 7200- and HFE 7300-wetted wipers expand significantly at 71 °C, which is an indication that the pressure in the pouches is greater than one atmosphere. Significant pouch expansion is unacceptable because the pouches could burst.

Initially, tests were conducted by soaking a variety of ACF fabrics in HFE 7200, HFE 7300, or a mixture thereof; sealing the wet fabrics in a pouch; and then storing the pouches at 71°C. These tests also evaluated the manner in which the ACF and HFE was added to the pouch. For example, the ACF was pre-dried in an oven at 150 °C and then added to a boiling pot of HFE for extended periods of time. Hundreds of pouches were prepared. In all cases the pouches would eventually expand in a 71 °C oven. In general, the pouches containing ACF and HFE 7200 would expand within 24 h of being placed in the oven, while those containing ACF and HFE 7300 could last up to two weeks before expanding.

It was not surprising to find the pouch expand when untreated ACF was combined with HFE 7200 then sealed and heated to 71 °C. Water can be adsorbed into the pores of the ACF. Taking into account the vapor pressure of water (33.4 kPa @ 71 °C), as well as the pressure due to the thermal expansion of air in the pouch (13.7 kPa when increased from 27 to 71 °C), the total pressure within a fixed volume containing an HFE 7200 wetted wiper could be 134.4 kPa. This is greater than 1 atm (101 kPa) and thus likely to cause a pouch to expand. It was also found that a pouch containing a pre-dried ACF wiper, with no water, would expand as well when sealed in a pouch containing HFE 7300. A fixed-volume cell containing these materials should theoretically have a total pressure of 54.3 kPa at 71 °C, much less than 1 atm. This pressure should not cause a pouch to expand. It was also interesting to note that pouch expansion was not immediate and occurred slowly over time.

A limitation of these tests was that the only data recorded was the time in which the pouch expanded. In an effort to better understand the reason(s) for pouch expansion, a series of tests was performed with fixed-volume test cells to record the pressure within the cell versus time. This section will discuss these tests in detail, as well as their implications on the choice of solvents in terms of wiper and packaging design.

- Materials and Equipment:
    - Activated Carbon: Zorflex 50K ACF
    - HFE Solvents: Novec HFE 7200, Novec HFE 7300, Novec HFE 7500
    - Test Apparatus: Eight fixed-volume test cells were assembled from stainless steel NPT and Swagelok Fittings, connected through a three way ball valve to a 0–30 psig Pressure Gauge, as shown in Figure 26. The main body of the cell consisted of nominal 1 in. fittings. The internal volume of a cell was approximately 40 cm^3. Convection oven purchased from VWR Scientific, model # 13307R.

Figure 26. Photograph of test cell.

For each test, the Swagelok fitting in the end of the cell was opened to allow the as-received 50K and HFE to be charged into the cell, according to the test matrix shown below in Table 13.

It should be noted that in Tests 1–8, the test cell was nearly full with 50K and HFE, and the free volume in the cell was approximately 3 mL, the volume within the pressure gauge. After charging the cell was resealed, a thermocouple was taped to the side of cell, and the cell was then placed in a convection oven, which could be maintained at 71 ± 2 °C. The pressure and temperature of the cell were recorded on a daily basis. At the conclusion of Tests 1–8, the odd-numbered test cells were sent to Mr. David Hesselroth of 3M's Electronic Materials Division in St. Paul, MN for analysis of the contents by GC/MS.

Table 13. Test matrix.

Test #	ACF			HFE		
	Type	Amount (g)	Pre-Treatment	Type	Amount (ml)	Pre-Treatment
1	50K	7.72	None, As-Received	7200	30	None, As-Received
2	50K	7.78	None, As-Received	7200	30	None, As-Received
3	50K	7.36	None, As-Received	7300	30	None, As-Received
4	50K	7.44	None, As-Received	7300	30	None, As-Received
5	50K	7.41	None, As-Received	7500	30	None, As-Received
6	50K	7.54	None, As-Received	7500	30	None, As-Received
7	50K	7.53	None, As-Received	50/50 mix 7200/7500	30	None, As-Received
8	50K	7.78	None, As-Received	50/50 mix 7200/7500	30	None, As-Received
9	None			7200	13.0	None, As-Received
10	None			7200	22.8	None, As-Received
11	None			7200	32.7	None, As-Received
12	None			7200	35.0	De-Gassed, Boiled
13	None			10/90 (mol %) mix 7200/7500	35.0	None, As-Received
14	None			10/90 (mol %) mix 7200/7500	35.0	None, As-Received
15	None			10/90 (mol %) mix 7200/7500	35.0	De-Gassed, Boiled
16	None			10/90 (mol %) mix 7200/7500	35.0	De-Gassed, Boiled

5. RESULTS AND DISCUSSION

5.1 Characterization of Wipe Components for Shedding

The results for developmental rotary shedding tests are shown below in Table 14 A "-" sign indicates shedding was observed, while a "+" sign indicates no shedding was observed. Figure 27 shows the amount of shedding generated during the control test, in which the rotary-wiping operation was performed with a dry unprotected sheet of 50K ACF. The pictures clearly show a significant amount of carbon was deposited on the test piece, lending credence to the applicability and difficulty of this shedding test.

Table 14. Rotary shedding test results.

Rotary Shedding Test Matrix		% Open Area	Activated Carbon Fabrics			
			Dry			HFE-7200 Wetted
			50K	100 Micro	100 Meso	50K
Control	None	100%	-	N/A	N/A	N/A
PFG Polyester Fabrics	54717	8.1%	+	+	-	N/A
	64918	29.0%	+	-	-	N/A
PFG Nylon Fabrics	39278	8.2%	+	+	-	+
	66190	35.0%	-	-	-	-
Delstar Polyethylene Films	P520NAT-A	16.0%	+	N/A	N/A	N/A
	P520NAT-A	24.0%	+	N/A	N/A	N/A

The results in Table 14 show that all of the tested CFFs prevented shedding from the 50K ACF, both dry and HFE-wetted, except for PFG 66190, which has an open area of 35%, which was more than any other fabric tested. None of the tested fabrics prevented shedding from 100 meso, which was the most friable and prone to shed ACF. Polyester 54717 and nylon 39278, which both have percent open areas of around 8%, prevented shedding from 100 micro, while the more open PFG fabrics, 64918 and 66190, did not.

The test results clearly show that minimizing the ACF shedding potential requires CFFs with smaller percent open areas. The results rule out the possibility of using nylon PFG 66190 as the CFF. The test results also reinforce the use of 50K as the first ACF layer in a wiper to minimize shedding potential.

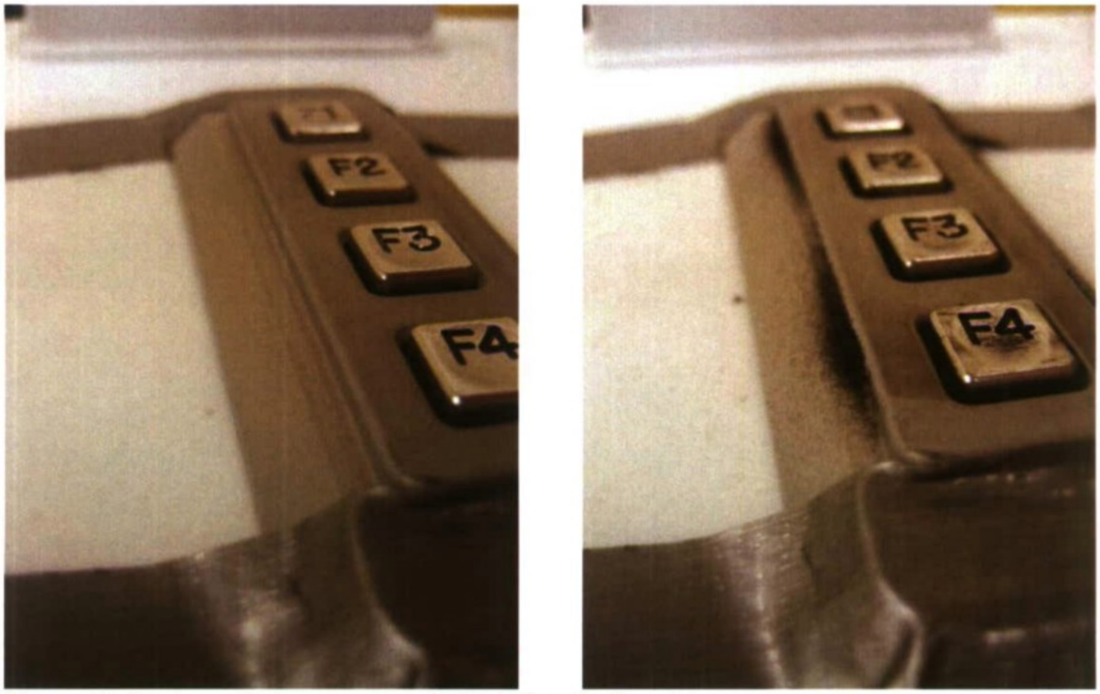


Figure 27. Wiping with dry unprotected 50K before & after pictures.

The objectives of these shedding tests were to validate that the recommended wiper facing fabric, PFG 39278 laminated to 50K ACF, did not shed, and to show that PFG 66387 was comparable to PFG 39278 in terms of preventing carbon shedding.

The procedure for these shedding tests was the same as that for the developmental shedding tests, except for the following changes:

- In all tests 50K was used as the ACF layer.
- All tests were performed both HFE 7200 wetted and dry.
- The CFF and ACF were tested both laminated and non-laminated.

- All tests were performed in triplicate.

The test matrix is shown in Table 15.

Table 15. Test matrix for the shedding test.

Test #	Candidate Facing Fabric (CFF)	Laminated (y/n)	Wetted (y/n)
1		yes	yes
2	39278		no
3		no	yes
4			no
5		yes	yes
6	66387		no
7		no	yes
8			no

Results of the rotary tests are outlined in Table 16. In the table, if a test failed it is shown in red with a "-", and if the test passed it is shown in green with a "+".

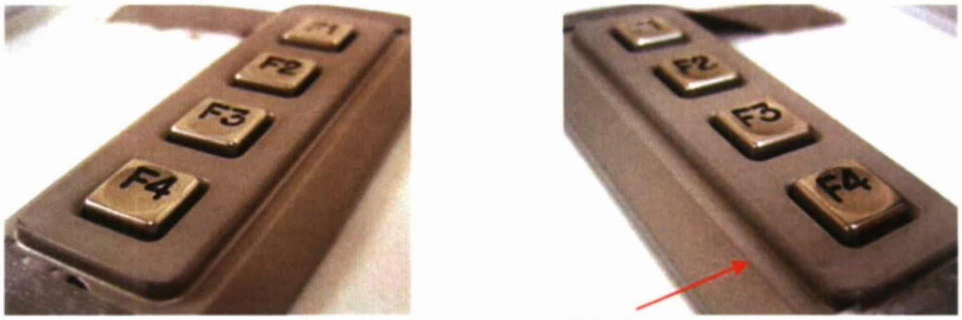
From the table, several important things can be seen:

- Twelve out of twelve tests using HFE 7200-wetted decontamination wipes were successful.
- Ten out of the twelve tests performed without any solvent still resulted in no shedding.
- The two failed tests were in two different test conditions. In other words, no test condition failed more than once out of the three trials performed.

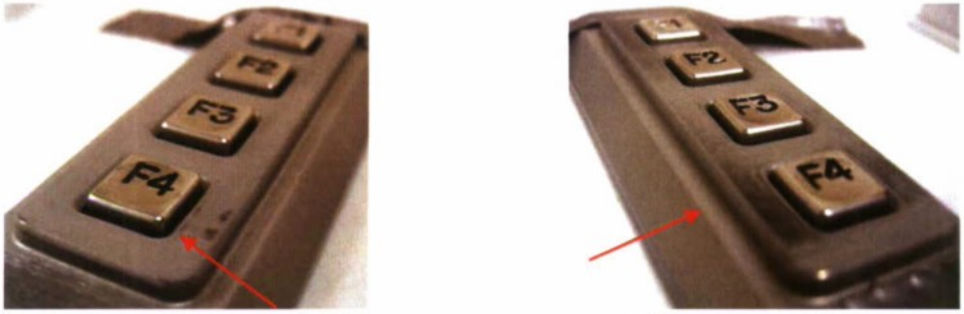
Table 16. Shedding test results.

Test #	Candidate Facing Fabric (CFF)	Laminated (y/n)	Wetted (y/n)	Trial #1	Trial #2	Trial #3
1		yes	yes	+	+	-
2	39278		no	+	+	+
3		no	yes	+	+	+
4			no	+	+	+
5		yes	yes	+	+	+
6	66387		no	-	+	+
7		no	yes	+	+	+
8			no	+	+	+

From these results it is clear that both the candidate facing fabrics are suitable for use in a decontamination wipe. They both prevent the shedding of the ACF on the keypad surface under normal conditions, and, most of the time, under harsher conditions as well. It can also be noted that from the photos the shedding seen in these tests was minimal in comparison to the shedding seen in previous tests of 50K without the use of a CFF (Figure 28).



Success example **39278, dry, laminated failure**



66387, dry, non-laminated failure **50K without CFF**

Figure 28. Keypad failure comparison.

5.2 Characterization of Wipe Components for Flexibility

The results of the various flexural rigidity tests performed are summarized in Table 17 and Table 18. Examination of these results indicates that:

1. The flexural stiffness of single layers of fabrics varies widely, depending on the construction of the fabric.
2. In general, the flexural stiffness of any of the single layer fabrics tested is significantly lower than the flexural stiffness of laminates.
3. The flexural stiffness of laminates increases rapidly as the numbers of layers in the laminate increase.
4. The flexural stiffness of two-ply laminates increases dramatically as the laminate thickness increases above approximately 1.2 mm, as shown in Figure 29. A much earlier onset in stiffness is observed for a three-ply laminate, the sole point above the trend line is at a thickness of 0.6 mm.

5. The stiffness of a laminate is somewhat dependent on the thickness of the glue layer, increasing somewhat with the weight of that layer.

6. The flexural stiffness of facing fabric/activated carbon fabric laminates, which are asymmetrical in structure, depends on the orientation of the laminate.

7. It was also noted that the differences in stiffness are most evident for the lightest weight adhesives. The effect of orientation is greater for laminates prepared with adhesive webs of 0.25 oz/yd^2 than for laminates prepared with adhesive webs of 0.60 oz/yd^2. With the heavier weight adhesives, the effects of orientation on the mechanical properties of the laminate are damped by the relatively heavy glue layer.

8. The most flexible laminate identified consists of PFG 39278 facing fabric, bonded to Zorflex 50K with 0.25 oz/yd^2 Spunfab web adhesive PA 1541C/0.25.

Some limited tests were also performed to demonstrate the relative effects of adding a second sheet of fabric to a fabric laminate by either area bonding or edge bonding. As is evident from Table 18, a PFG39278/50K laminate is significantly stiffer than both fabrics alone. Web bonding a second sheet of PFG39278 to a PFG39278/50K laminate, to make a PFG39278/50K/ PFG39278 laminate, results in a significantly stiffer structure. In accordance with the theoretical development outlined in Section 5.2, edge bonding a second sheet of PFG39278 to a PFG39278/50K laminate results in a structure that retains its flexibility.

Apart from their effects on the mechanical properties of a laminate, it was also observed that with the heavier glues, the glue layer between fabrics is much less open than the glue layer obtained with the lighter weight adhesives. The relatively low open area of the adhesive layer obtained with the heavier weight adhesives is of concern because it could be a barrier to liquid mass transfer between the facing layer and the activated carbon fabric.

Table 17. Flexural rigidity of fabrics of interest by ASTM Method D1388-96.

Sample	Facing Fabric	Adhesive	Carbon Fabric(s)	Backing	Thickness mm	Flexural Rigidity mg cm^3
Single Layer ACF						
50K			**50K**		**0.48**	**74**
FM 10 meso			FM 10 meso		0.39	22
FM70			FM70		0.46	74
FM100 micro			FM100 micro		0.76	738
FM 100 meso			FM 100 meso		0.64	536
CT 1001 Fabric			CT 1001 Fabric		0.33	3
Single Layer Front Fabric						
PFG s/39278 (S&HS)	PFG s/39278 (S&HS)				0.08	24
PFGs/66165(HS*S)	PFGs/66165(HS*S)				0.10	20
PFG54717 HSS	PFG54717 HSS				0.19	147
PFG64918	PFG64918				0.26	302
Delstar PQ218 NAT-E	PQ218Nat	EVA			0.14	15
Delstar P520 Nat-A	P520Nat	polyamide			0.14	13
Zorflex Laminates						
50K/100 micro		not specified	50K/100micro		1.40	443
50K/100micro/glue		not specified	50K/100micro	glue	1.36	21495
50K/100micro/Tychem		not specified	50K/100micro	Tychem	1.60	58401
PFG/50K/100micro/Tychem	PFG 54717 (HS*S)	PA1541C/1-.025-017 (front layer to laminate)	50K/100micro	Tychem	1.74	> 70000
50K Laminates						
Facing Fabric Down	PQ218Nat	EVA	50K		0.55	242
	P520Nat	Polyamide	50K		0.55	41
	PF64918	PO 4401/1-0175-017	50K		0.72	386
	PFG 54717 (HS*S)	PO 4401/1-0175-017	50K		0.68	1877
	PFG 54717 (HS*S)	PA 1008 0.60 osy	50K		0.68	1380
	PFG 54717 (HS*S)	PA1541C/1-.025-017	50K		0.67	685
	PFG s/66165(HS*S)	PA1541C/1-.025-017	50K		0.60	154
	PFG s/39278 (HS*S)	PA1541C/1-.025-017	50K		0.54	144
Facing Fabric Up	PQ218Nat	EVA	50K		0.55	319
	P520Nat	Polyamide	50K		0.55	273
	PF64918	PO 4401/1-0175-017	50K		0.72	318
	PFG 54717 (HS*S)	PO 4401/1-0175-017	50K		0.68	961
	PFG 54717 (HS*S)	PA 1008 0.60 osy	50K		0.68	317
	PFG 54717 (HS*S)	PA1541C/1-.025-017	50K		0.67	518
	PFG s/66165(HS*S)	PA1541C/1-.025-017	50K		0.60	154
	PFG s/39278 (HS*S)	PA1541C/1-.025-017	50K		0.54	144
50K/50K laminates						
Facing Fabric Down	PFG54717 (HS*S)	PO 4401/1-0175-017	50K +50K		1.20	2191
	PFG64918	PO 4401/1-0175-017	50K +50K		1.26	3368
Facing Fabric Up	PFG54717 (HS*S)	PO 4401/1-0175-017	50K +50K		1.20	1878
	PFG64918	PO 4401/1-0175-017	50K +50K		1.26	4131
50K/100 meso						
Facing Fabric Down	PFG54717 (HS*S)	PO 4401/1-0175-017	50K + 100 meso		1.42	34195
Facing Fabric Up	PFG54717 (HS*S)	PO 4401/1-0175-017	50K + 100 meso		1.42	17981
CT 1001/PFG 54717 HSS Laminates						
	PFG54717 HSS	SF PA549C/1-025-017	CT 1001 Fabric		0.48	999
	PFG54717 HSS	SF PE2900, 0.60 osy	CT 1001 Fabric		0.49	1228
	PFG54717 HSS	SF PO4401/1-.0175-017	CT 1001 Fabric		0.44	974
	PFG54717 HSS	SF PO 4401/1-025-017	CT 1001 Fabric		0.50	705
	PFG54717 HSS	SF PA 1008 0.60 osy	CT 1001 Fabric		0.48	1295

Table 18. Flexural rigidity of PFG 39278/50K laminates by ASTM Method D1388-96.

Sample	Facing Fabric	Adhesive	Carbon Fabric(s)	Backing	Thickness mm	Flexural Rigidity $mg\ cm^3$
Single Layer ACF						
50K			**50K**		**0.48**	**74**
Single Layer Front Fabric						
PFG s/39278 (S&HS)	PFG s/39278 (S&HS)	prior data			0.08	24
PFG s/39278 (S&HS)	PFG s/39278 (S&HS)				0.08	9
PFG s/39278 (S&HS)	PFG s/39278 (S&HS)				0.08	13
PFG s/39278 (S&HS)	PFG s/39278 (S&HS)				0.08	14
PFG s/39278 (S&HS)	PFG s/39278 (S&HS)				0.08	15
PFG s/39278 (S&HS)	PFG s/39278 (S&HS)	average of above 4 data points			0.08	13
50K Laminates						
Facing Fabric Down	PFG s/39278 (S&HS)	PO 4401/1-0175	50K		0.52	294
	PFG s/39278 (S&HS)	PO 4401/1-0125	50K		0.60	261
	PFG s/39278 (S&HS)	PA 1008 0.60 osy	50K		0.59	375
	PFG s/39278 (S&HS)	PE 2900 0.60 osy	50K		0.56	848
	PFG s/39278 (S&HS)	PA1541C/1-.025-017	50K		0.56	275
	PFG s/39278 (S&HS)	PA1541C/1-.025-017	50K		0.55	237
Facing Fabric Up	PFG s/39278 (S&HS)	PO 4401/1-0175	50K		0.52	2235
	PFG s/39278 (S&HS)	PO 4401/1-0125	50K		0.60	1925
	PFG s/39278 (S&HS)	PA 1008 0.60 osy	50K		0.59	1268
	PFG s/39278 (S&HS)	PE 2900 0.60 osy	50K		0.56	1084
	PFG s/39278 (S&HS)	PA1541C/1-.025-017	50K		0.56	1451
	PFG s/39278 (S&HS)	PA1541C/1-.025-017	50K		0.55	1203
average of Fabric Up/Down	PFG s/39278 (S&HS)	PO 4401/1-0175	50K		0.52	1264
	PFG s/39278 (S&HS)	PO 4401/1-0125	50K		0.60	1093
	PFG s/39278 (S&HS)	PA 1008 0.60 osy	50K		0.59	821
	PFG s/39278 (S&HS)	PE 2900 0.60 osy	50K		0.56	966
	PFG s/39278 (S&HS)	PA1541C/1-.025-017	50K		0.56	863
	PFG s/39278 (S&HS)	PA1541C/1-.025-017	50K		0.55	720
Facing Fabric Both Sides	PFG s/39278 (S&HS)	PA1541C/1-.025-017	50K		0.64	18783
	web-bonded both sides					
	PFG s/39278 (S&HS)	PA1541C/1-.025-017	50K			
	web-bonded one side -	end basted other side				
		Laminate Up			0.65	242
		Laminate down			0.65	1454
		Average			0.65	677

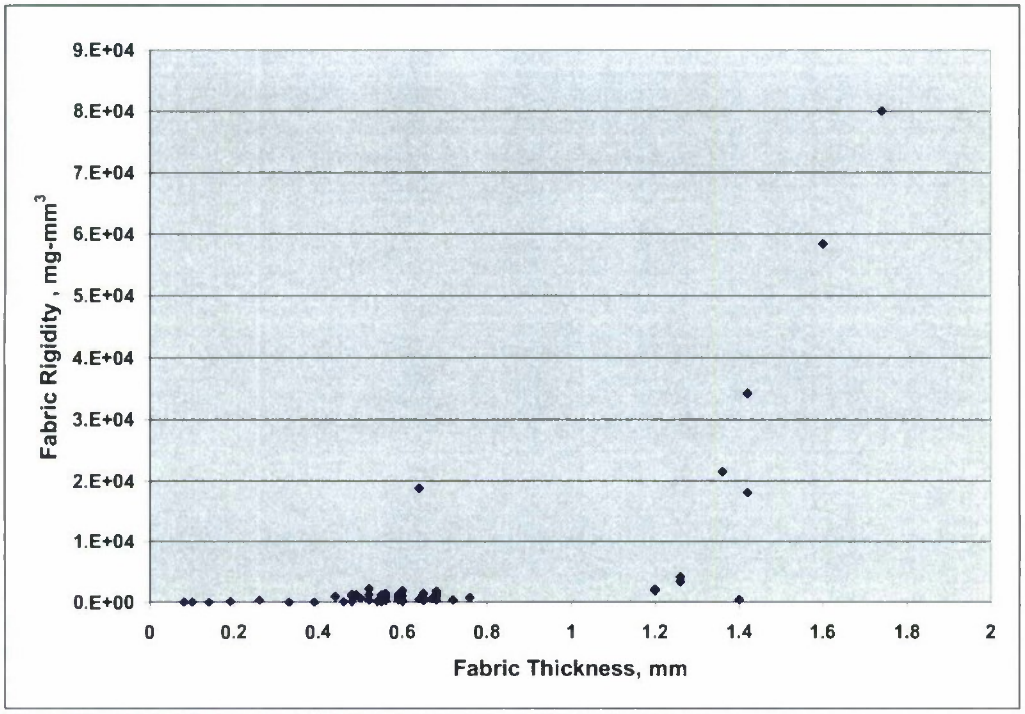


Figure 29. Fabric flexural stiffness as a function of fabric thickness.

5.3 Contaminant Adsorption Tests

CEES breakthrough curves for a low capacity material and a high capacity material are presented in Figure 30 and Figure 31 respectively.

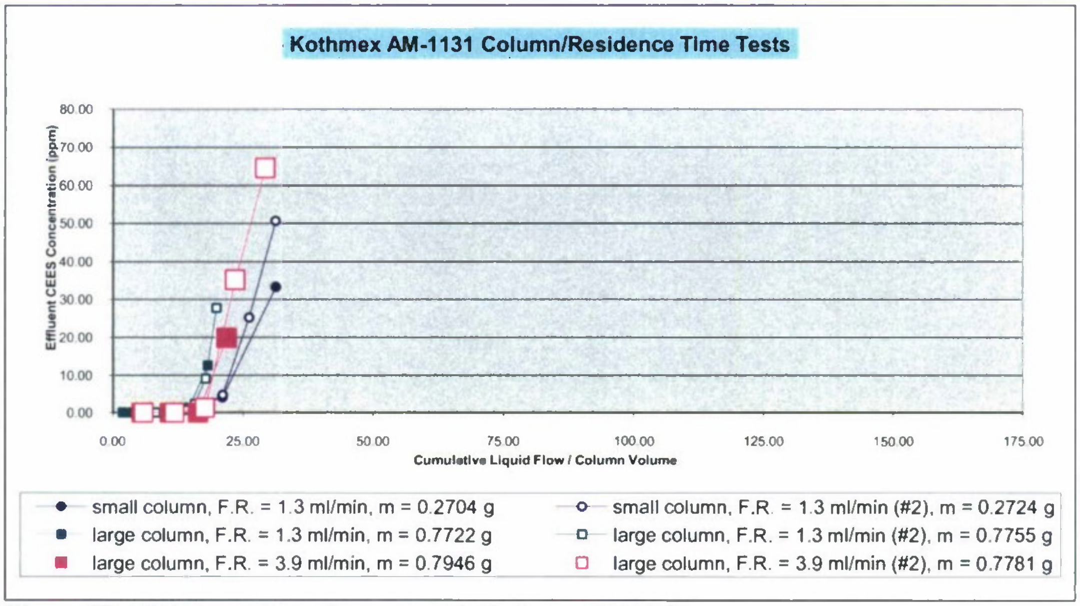


Figure 30. CEES breakthrough curves with Kothmex AM-1131.

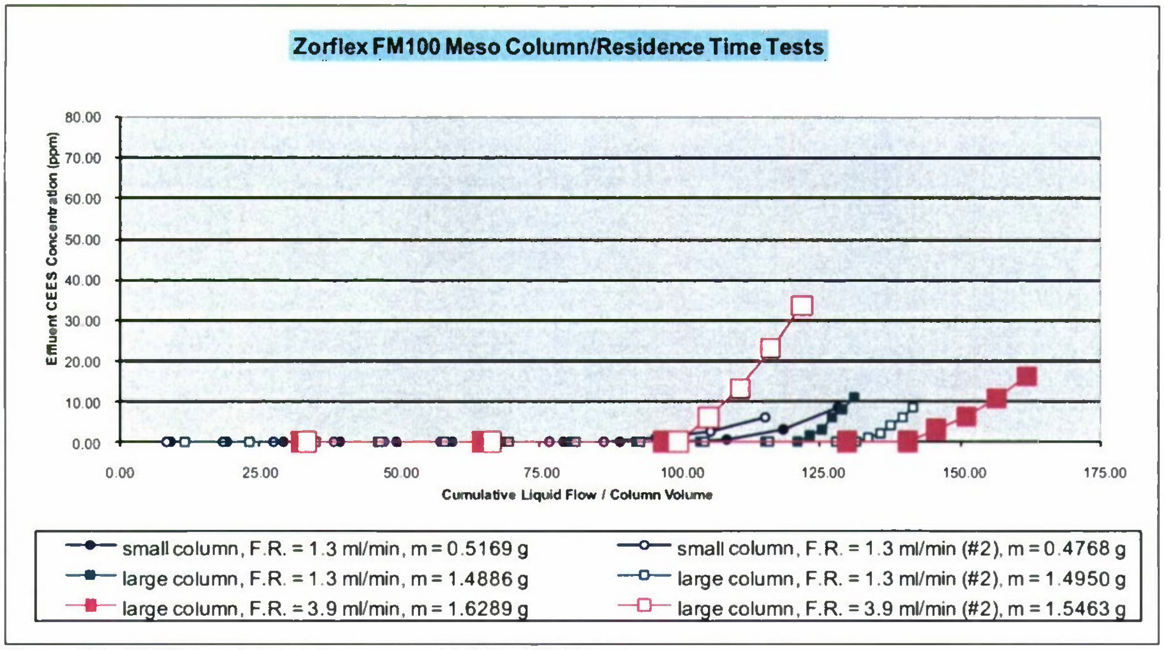


Figure 31. CEES breakthrough curves with FM 100 Meso.

Normalizing the results by graphing effluent CEES concentration versus cumulative liquid flow over the adsorbent volume shows that residence time does not have a large impact on CEES adsorption capacity, over the range of residence times examined, namely from 1 to 3 min. It should be noted that the residence times are much shorter than the residence times typically used for liquid phase adsorption with granular activated carbon. The tests that used large adsorbent volumes/masses with small liquid flow rates exhibited only slightly higher than average contaminant capacities.

Table 19 presents a comparison of the CEES and HD adsorption capacities of four different activated carbon fabrics that were examined. The effect of volume average pore diameter of the fabric on its adsorption capacity for CEES and HD is presented in Figure 32. Figure 33 presents the same data, corrected for differences in the specific surface areas of these fabrics.

Table 19. Comparison of CEES and HD adsorption capacities of activated carbon fabrics.

Supplier	Kothmex	Calgon	Calgon	Calgon
Material	1131 Felt	FM-100 Micro	FM-10 Meso	FM-100 Meso
Surface Area, m^2/g	1230	1360	995	655
Volume Percent Mesopores	13%	18%	60%	84%
Volume Mean Pore Diameter, AU	2	6	22	29
CEES Adsorption Capacity, wt-%				
Actual Data	0.87%	2.48%	2.66%	3.24%
Normalized to 1,000 m^2/g	0.71%	1.82%	2.67%	4.95%
HD Adsorption Capacity, wt-%				
Actual Data	2.12%	3.67%	5.04%	5.54%
Normalized to 1,000 m^2/g	1.72%	2.70%	5.07%	8.46%
Ratio HD/CEES	2.44	1.48	1.89	1.71

Examination of the data indicates that the adsorption with the specific surface area and as a function of the volume average pore diameter, over the range of 2 to 29 Å (0.2 to 2.9 nm). The adsorption capacity for HD increases from 1.72 to 8.46% (normalized to 1000 m^2/g), or about 4.9 fold over that range, while that of CEES increases from 0.71 to 4.95%, or about 6.7 fold. Given that CEES and HD have very similar molecular volumes (0.193 nm^3 for CEES, and 0.208 nm^3 for Agent HD), the higher adsorption capacity for HD is due in part to its higher molecular weight (159 Daltons vs. 124.6 Daltons for CEES) and its somewhat lower solubility in HFE 7100 (3.4 vol%) than CEES (12 vol%). This similarity in the experimental results reinforces the argument for using CEES as a simulant for HD in liquid-phase adsorption experiments, such as the ones described here.

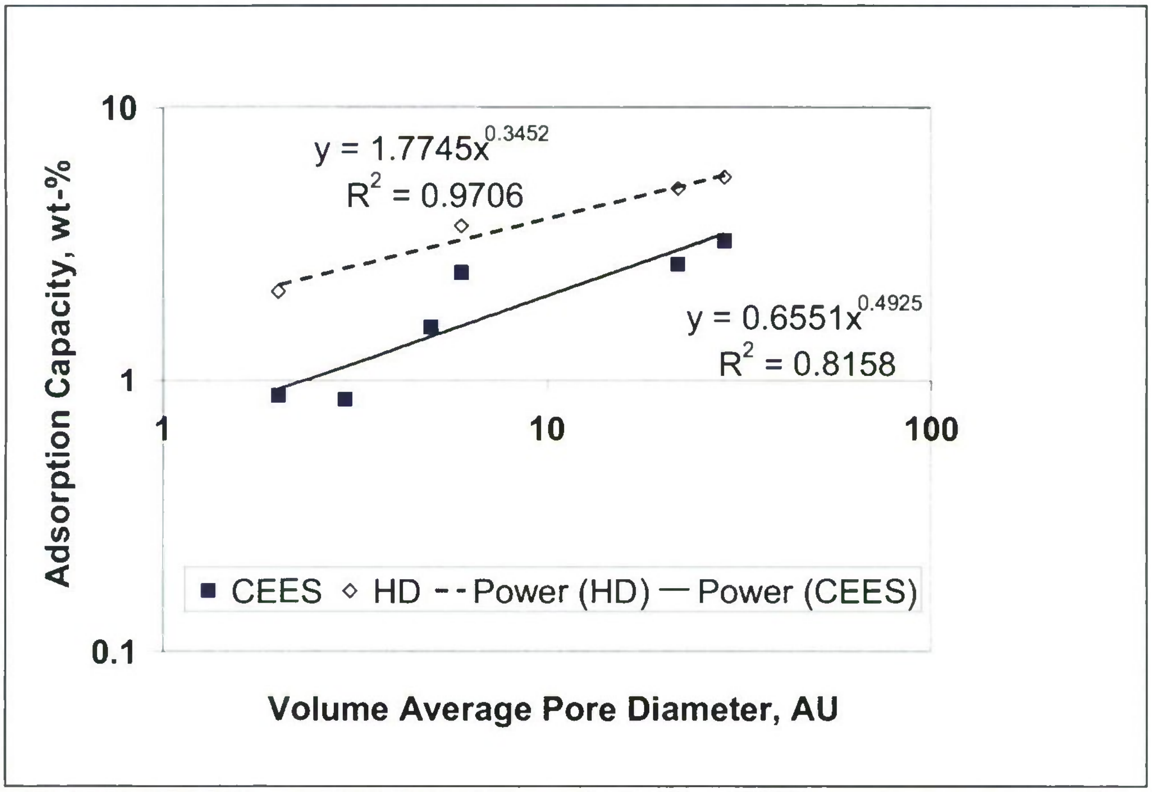


Figure 32. Activated carbon fabric adsorption capacity for HD and CEES vs. volume average pore diameter.

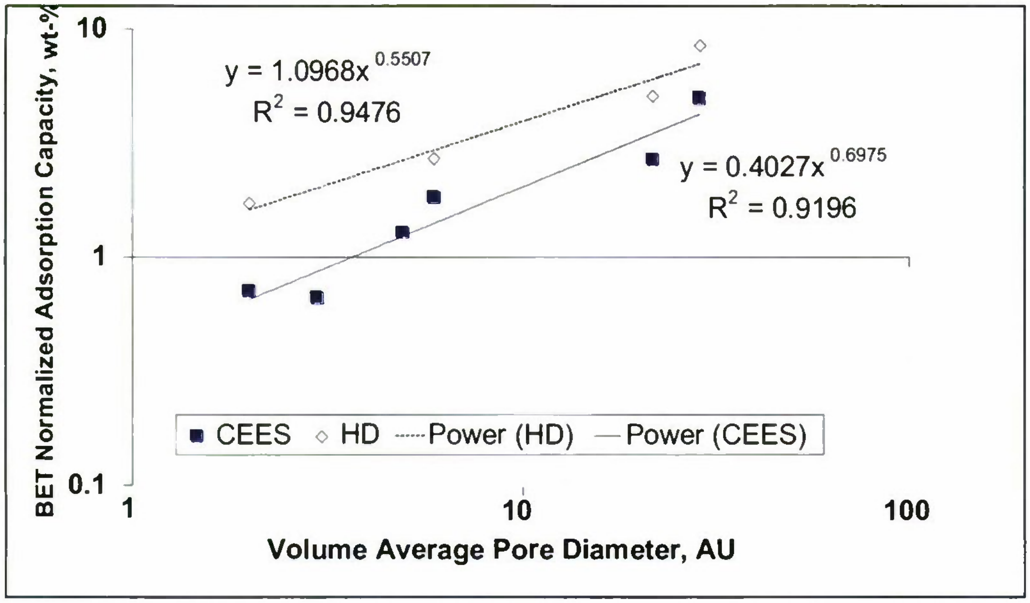


Figure 33. BET normalized adsorption capacity of activated carbon fabrics for CEES and agent HD vs volume average pore diameter.

5.4 Contaminant Transport/Wicking Tests with Chemical Agent Simulant

The results of the static contaminant transport developmental tests that were performed are shown below in Table 20 through Table 23 for:

- CEES transport through a dry fabric stack
- CEES transport through a HFE-wetted fabric stack
- DEP transport through a dry fabric stack, and
- DEP transport through a HFE-wetted fabric stack

The results present the percentage of the contaminant recovered from the aluminum tray surface, the contact layer (CFF), and the ACF layer versus the total contaminant recovered. Each result is the average of two runs.

The CEES transport results, both dry and HFE wetted, presented in Table 20 and Table 21 clearly show that as the contact time increases, the amount of contaminant transported through the contact layer and into the ACF layer increases as well. This point is well illustrated where after 3 min of contact time, 33.3–98.9% of the CEES had transferred into the ACF layer, after 10 min the range increases to 58.4–99.6%, and after 30 min the range increases again to 94.4–100%.

Table 20. CEES dry-static transport test results.

CEES - Dry Results		Tray			Contact Layer			ACF Layer		
Candidate Contact Layers		Contact Time (Min.)								
		3	10	30	3	10	30	3	10	30
PFG Polyesters	54717	0.0%	0.0%	0.0%	0.1%	0.2%	0.1%	99.9%	99.8%	99.9%
	60171	0.0%	0.0%	0.0%	4.0%	0.4%	0.1%	96.0%	99.6%	99.9%
	66290	0.0%	0.0%	0.0%	0.2%	0.2%	0.2%	99.8%	99.8%	99.8%
	1122	0.0%	0.0%	0.0%	2.7%	0.3%	0.2%	97.3%	99.7%	99.8%
	64918	0.0%	0.0%	0.0%	0.1%	0.2%	0.1%	99.9%	99.8%	99.9%
Cleaning Cloth Fabrics	3M Scotchbrite	0.0%	0.0%	0.0%	44.2%	5.9%	0.3%	55.8%	94.1%	99.7%
	Perfect Clean	0.0%	0.0%	0.0%	28.2%	0.8%	0.3%	71.8%	99.2%	99.7%
Wicking T-Shirt Fabrics	Dri-Fit (100% Polyester)	0.0%	0.0%	0.0%	34.9%	2.2%	0.1%	65.1%	97.8%	99.9%
	Heatgear (80% Polyester, 20% Elastine)	0.0%	0.0%	0.0%	35.5%	6.6%	0.3%	64.5%	93.4%	99.7%
	Heatgear (95% Polyester, 5% Elastine)	0.0%	0.0%	0.0%	18.3%	0.3%	0.1%	81.7%	99.7%	99.9%

Table 21. CEES HFE wetted-static transport test results.

CEES - HFE Wetted Results		Tray			Contact Layer			ACF Layer		
Candidate Contact Layers		Contact Time (Min.)								
		3	10	30	3	10	30	3	10	30
PFG Polyesters	54717	0.0%	0.0%	0.0%	2.2%	0.4%	0.2%	97.8%	99.6%	99.8%
	60171	0.0%	0.0%	0.0%	7.5%	0.5%	0.4%	92.5%	99.5%	99.6%
	66290	0.1%	0.0%	0.0%	2.8%	0.5%	0.0%	97.1%	99.5%	99.9%
	1122	0.0%	0.0%	0.0%	2.1%	0.4%	0.1%	97.9%	99.5%	99.9%
	64918	0.1%	0.3%	0.0%	0.9%	0.2%	0.0%	98.9%	99.5%	100.0%
Cleaning Cloth Fabrics	3M Scotchbrite	0.0%	0.1%	0.0%	66.7%	41.5%	5.6%	33.3%	58.4%	94.4%
	Perfect Clean	0.0%	0.0%	0.0%	39.6%	9.0%	1.3%	60.4%	91.0%	98.6%
Wicking T-Shirt Fabrics	Dri-Fit (100% Polyester)	0.0%	0.0%	0.0%	31.6%	6.9%	0.7%	68.4%	93.1%	99.2%
	Heatgear (80% Polyester, 20% Elastine)	0.0%	0.1%	0.1%	23.1%	6.9%	4.3%	76.9%	93.0%	95.5%
	Heatgear (95% Polyester, 5% Elastine)	0.0%	0.0%	0.0%	18.2%	4.5%	2.0%	81.8%	95.5%	98.0%

The results in Table 22 and Table 23 also clearly demonstrate that the CEES transport ability of the CFFs varies widely among the fabrics tested. In both dry and wetted wicking scenarios, the results show that the PFG polyester fabrics significantly outperform the cleaning cloth and wicking T-shirt fabrics in terms of CEES transport ability. In a dry fabric stack, with 3 min of contact time, the amount of CEES transferred into the ACF layer ranges from 96.0 to 99.9% for PFG polyester fabrics, from 55.8 to 71.8% for cleaning cloth fabrics, and from 64.5 to 81.7% for T-shirt fabrics. In a HFE-wetted fabric stack, with 3 min of contact time, the amount of CEES transferred into the ACF layer ranges from 92.5 to 98.9% for PFG polyester fabrics, from 33.3 to 60.4% for cleaning cloth fabrics, and from 68.4 to 81.8% for T-shirt fabrics. The relatively poor wicking performance of the cleaning cloths and the wicking T-shirt materials was due to the physical attributes of the fabric, in particular differences in fabric thickness as well as fiber diameter and shape. Overall, all of the PFG polyester fabrics tested effectively wick >99.5% of the CEES into the ACF layer within 10 min in both dry and HFE-wetted fabric stacks.

It is also interesting to note that the CEES wicking rates through dry and HFE-wetted fabric stacks were very similar for all CFFs at all of the contact times tested.

The contaminant wicking results through a dry fabric stack presented for DEP in Table 22 were very different than those presented for CEES in Table 20. Whereas previously it was shown that >99.6% of CEES was transferred into the ACF in 10 min in a dry fabric stack with PFG polyester as the CFF, the DEP wicking results revealed that, under the same conditions, <19.5% of the DEP reaches the ACF layer. The majority of the DEP was recovered from the PFG polyester CFF and, in general, less than 0.7% was recovered from the surface. These results clearly showed that the more viscous DEP simulant, with properties similar to those of VX, was a much tougher wicking challenge because it wicks much more slowly, and there was less wicking than CEES. All subsequent wicking tests were performed with DEP.

Table 22. DEP dry-static transport test results.

DEP - Dry Results		Tray			Contact Layer			ACF Layer		
Candidate Contact Layers		Contact Time (Min.)								
		3	10	30	3	10	30	3	10	30
PFG Polyesters	54717		0.5%			84.0%			19.5%	
	60171		0.4%			91.2%			16.2%	
	66290		0.6%			95.2%			0.3%	
	1122		0.7%			81.2%			18.0%	
Delstar Polyethylene	P520NAT-A		42.3%			14.5%			32.2%	
	PQ218NAT-E		35.6%			29.7%			29.8%	
Other Fabrics	3M Scotchbrite		0.5%			100.2%			1.0%	
	Perfect Clean		0.3%			96.6%			0.6%	
	Polypropylene		0.9%			98.3%			1.1%	

DEP wicking tests in a dry fabric stack were also performed with Delstar films, cleaning cloth fabrics, and a polypropylene sock material. The Delstar films were able to transport about 30% of the DEP into the ACF layer, which was the most of any CFF tested. However, the films also left behind a significant amount of DEP on the surface, 35.6–42.3%. The results for the cleaning cloth fabrics and the sock material revealed that thicker fabrics were almost completely ineffective, with about 100% of the DEP being recovered from the CFF and <1.1% being recovered from the ACF layer.

The DEP-wicking test results for HFE-wetted fabric stacks are shown below in Table 23. The results revealed that DEP wicked much more effectively in a wetted fabric stack than in a dry fabric stack. At 10 min of contact time, 37.9–72.3% of the DEP wicked through thick cleaning cloth fabrics and sock material into the ACF layer in a HFE wetted stack versus <1.1% in a dry stack. Also, with 10 min of contact time, 90.5–96.3% of the DEP wicked through the PFG polyester fabrics into the ACF layer in a HFE-wetted stack versus <19.5% in a dry stack.

DEP-wicking tests for HFE-wetted fabric stacks were also performed using Delstar films and PFG nylons fabrics as the CFF. The Delstar films wicked about 96% of the DEP into the ACF layer after 10 min, comparable to the amount wicked by the PFG polyester fabrics. However, 1.3–2.1% of the DEP was recovered from the aluminum tray surface. The PFG nylon fabrics were the most effective CFFs tested, wicking 92.1–95.2% into the ACF layer in 3 min and 97.0–98.6% in 10 min. Nylon PFG 39278 was the best CFF tested in terms of DEP contaminant transport, effectively wicking 98.6% of the DEP into the ACF and leaving 0% behind on the aluminum tray after 10 min.

As previously mentioned, the results in Table 23 and Table 24 present the percentage of the contaminant recovered from the aluminum tray surface, the contact layer (CFF), and the ACF layer versus the total contaminant recovered. The results were presented in this fashion because the primary concern was the fate of simulant during a wicking test. However, good mass balances do strengthen the validity of the method and the data. The mass balances for all static wicking tests are shown below in Table 23. Overall the mass balances were very strong and consistent. During static wicking tests, on average of 96–102% of the DEP and 79–90% of the CEES was accounted for.

Table 23. DEP HFE wetted-static transport test results.

DEP Results		Tray			Contact Layer			ACF Layer		
Candidate Contact Layers		Contact Time (Min.)								
		3	10	30	3	10	30	3	10	30
PFG Polyesters	54717	0.9%	0.1%	0.0%	9.8%	3.7%	1.4%	89.3%	96.3%	98.6%
	60171	0.0%	0.5%	0.2%	7.0%	3.4%	1.4%	93.0%	96.1%	98.4%
	66290		0.5%			3.5%			96.0%	
	1122		0.6%			8.9%			90.5%	
PFG Nylons	39278	1.3%	0.0%		3.5%	1.3%		95.2%	98.6%	
	66190	2.1%	0.9%		4.5%	1.9%		93.4%	97.2%	
	66165	1.6%	0.2%		6.4%	2.7%		92.1%	97.0%	
Delstar Polyethylene	P520NAT-A		2.0%			2.2%			95.8%	
	PQ218NAT-E		1.6%			2.1%			96.3%	
Other Fabrics	3M Scotchbrite		0.3%			38.5%			61.2%	
	Perfect Clean		0.2%			27.5%			72.3%	
	Polypropylene		0.4%			61.7%			37.9%	

Table 24. Mass balances static contaminant transport tests.

Mass Balances		CEES						DEP					
		Dry			HFE-Wetted			Dry			HFE-Wetted		
		3	10	30	3	10	30	3	10	30	3	10	30
PFG Polyesters	54717	97%	99%	97%	92%	95%	98%		104%		101%	93%	
	60171	96%	93%	92%	93%	96%	95%		108%			95%	
	66290	86%	79%	82%	76%	76%	84%		96%			103%	
	1122	75%	79%	79%	75%	78%	71%		100%			103%	
	64918	78%	84%	77%	78%	81%	76%						
PFG Nylons	39278										104%	99%	
	66190										94%	98%	
	66165										108%	101%	
DelStar Polyethylene	P520NAT-A								89%			97%	
	PQ218NAT-E								95%			94%	
Other Fabrics	3M Scotchbrite	97%	92%	89%	82%	77%	76%		102%			93%	
	Perfect Clean	96%	90%	88%	76%	78%	77%		98%			88%	
	Polypropylene								100%			93%	
Wicking T-Shirt Fabrics	Dri-Fit (100% Polyester)	93%	92%	90%	79%	78%	69%						
	Heatgear (80% Polyester, 20% Elastine)	96%	91%	90%	77%	78%	74%						
	Heatgear (95% Polyester, 5% Elastine)	93%	91%	90%	78%	69%	73%						
Average		90%	89%	87%	81%	81%	79%		99%		102%	96%	
Standard Deviation		8%	6%	6%	7%	8%	10%		5%		6%	5%	

5.5 Chemical Agent (Wicking Tests)

The results from the last in a series of screening tests, conducted on three of the best CFF, are shown in Table 25.

Table 25. VX wicking (uptake) through CFF.

Test No.	1	2	3	4	5	6	7	8	9	10
Agent	VX	VX	VX	VX	VX	VX	VX	VX	VX	VX
Load, g/m2	10	10	10	10	10	10	10	10	10	10
Substrate	Al	Al	Al	Al	Al	Al	Al	Al	Al	Al
HFE	None	None	7200	7200	7200	7200	7200	7200	7200	7200
Facing Layer	None	None	None	None	39278	39278	66190	66190	66165	66165
Carbon Layer	None	None	50K	50K	50K	50K	50K	50K	50K	50K
Volume of VX added, µL	20	20	20	20	20	20	20	20	20	20
VX, d-24	1.008	1.008	1.008	1.008	1.008	1.008	1.008	1.008	1.008	1.008
Weight of VX added, mg	20.16	20.16	20.16	20.16	20.16	20.16	20.16	20.16	20.16	20.16
Weight of VX Recovered, mg										
Panel (aluminum surface)	24.75	21.21	0.27	0.25	0.35	0.50	0.21	0.29	0.65	0.65
Facing Fabric					0.64	0.51	0.08	0.11	3.18	1.50
Carbon Layer			19.57	23.28	19.86	20.86	21.30	20.29	19.26	18.85
Total amount recovered VX[mg]			19.84	23.53	20.85	21.86	21.59	20.69	23.10	21.01
Positive Control										
Weight of VX Recovered, mg	19.65	20.71								

The three CFF are #39278 (final CFF selected), #66190, and #66165, two nylons and a polyester, respectively. The test was conducted with a slight modification from that described in Section 4. The contact time was 10 min. The material coupons facing fabric (CFF) and the ACF fabric (carbon layer) were all extracted in chloroform. The results indicated that the nylons were better at allowing VX transport through into the carbon layer than the polyester.

The TGA results are shown in Figure 34 and Figure 35. In both tests, little to no weight loss was detected throughout the temperature range selected. The temperature range was a few degrees above the boiling point for the CA, 198 and 217 °C, for GD and HD, respectively.

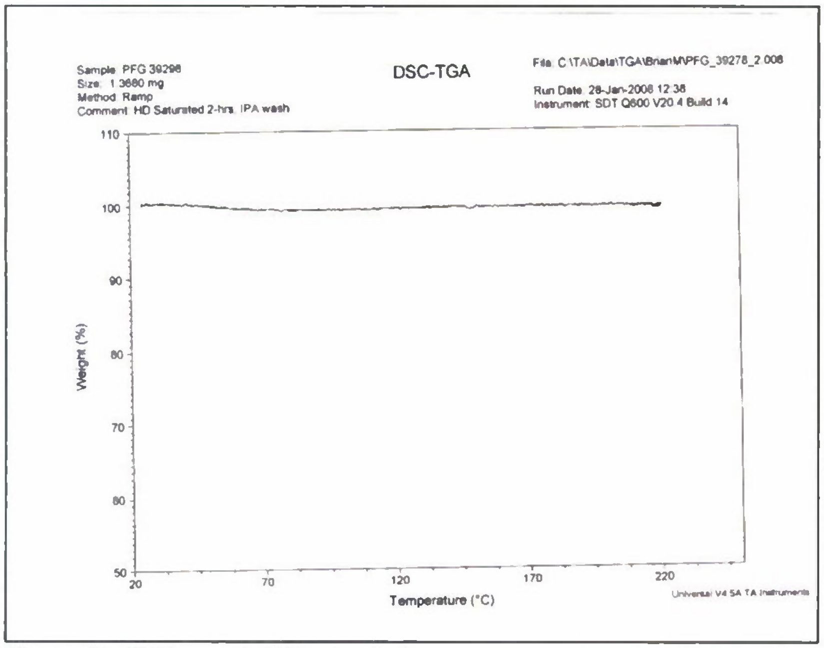


Figure 34. TGA results HD saturated.

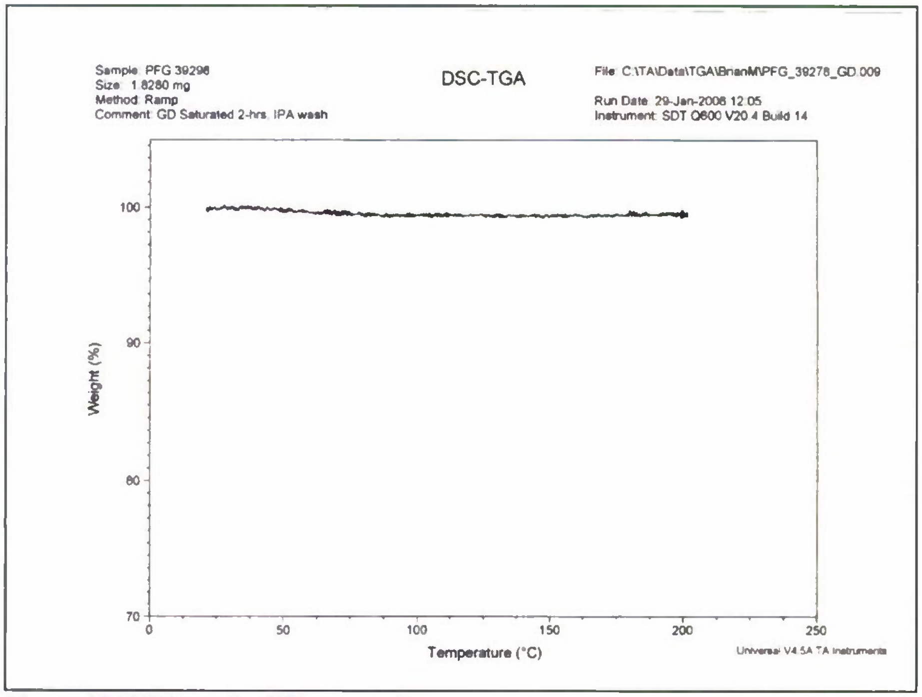


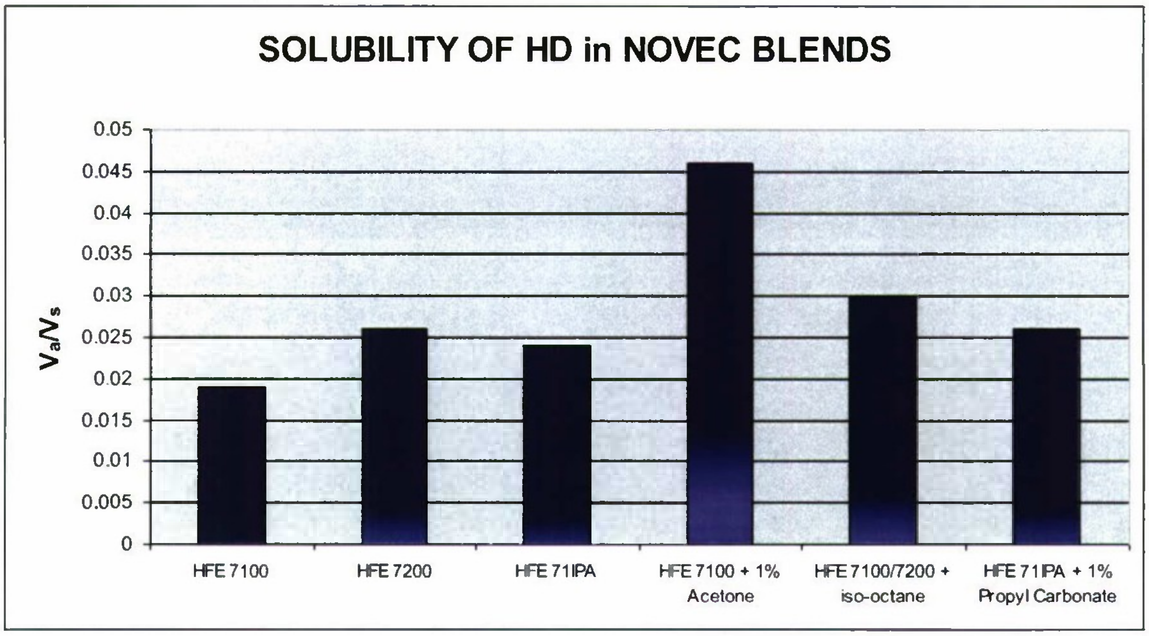
Figure 35. TGA results GD saturated.

The graphs show there is no mass loss during the temperature variation as a result of the analyte. Therefore, it does not appear that HD or GD are chemisorbed into the PFG 39278 facing fabric. The CA VX was not included due to instrument problems.

5.6 Solubility of HD in Modified Transfer Solvents

The result of the solubility tests are summarized in Table 26.

Table 26. HD Solubility in Select Novec® HFE Blends.



From the data table, the addition of a slight amount of the keytone (1% v/v acetone) provides the best, increase in solubility (v/v) for HD, although it is small. The issue of solubility of CA, specifically HD, was addressed extensively by Battelle as part of the JSSED solvent wash[8] system development. Their conclusion was that the increase in materials affects, resulting from the additives to HFE 7100, was not worth the increase in solubility. The poor solubility with HD could be overcome through mechanical agitation and slightly elevated temperature. Taking this into consideration, the pursuit of enhanced solubility was not worth the effort that would be required to promote a solvent containing a flammable and toxic material, notwithstanding the potential increased cost of materials tests.

5.7 M8 Paper Test

The results for the M8 Paper test are listed in Table 27–Table 34. A "+" symbol indicates the presence of a color change, as indicated on the M8 package key. A "-" symbol indicates no observable presence of color change. The "+/-" signifies the possible[***] presence of agent, due to the appropriate color change, as indicated on the M8 packet. N/A indicates a combination was not tested.

[***] In some cases the positive or negative presence of a color change was difficult to ascertain due to very small spots on the Paper. The +/- designation should be considered inconclusive for either condition (CA presence or not), but a possible positive CA presence.

Positive and negative controls were included in the test matrix. The positive control (+ Control) included the addition of 1 g/m^2 (Table 27–Table 30) or 10 g/m^2 (Table 31–Table 34) of the appropriate CA to the appropriate test material coupon. No decontamination procedure was performed, and the M8 Paper was applied directly following the 60 min incubation period. Similar to the positive controls, the negative controls (- Control) were treated using the same conditions, except the material test coupons did not receive CA.

The Zorflex 1-3 and M295 1-3, reflect three test replicates. The CARC–T was prepared to the same MIL-spec (MIL-C-53039A); the difference is the color is tan (#284) versus green (#383). PC indicates polycarbonate.

Table 27. HD at 1 g/m^2 starting contamination density.

HD	**Surface**					
Decon	**Aluminum**	**CARC-G**	**CARC-T**	**PC**	**Kapton**	**Viton**
Zorflex 1	-	-	-	-	-	-
Zorflex 2	-	-	-	-	-	-
Zorflex 3	-	-	-	-	-	-
M295 1	-	-	-	-	-	-
M295 2	-	-	-	-	-	-
M295 3	-	-	-	-	-	-
+ Control	+	+	n/a	+	n/a	n/a
- Control	-	-	n/a	-	n/a	n/a

Table 28. GD at 1 g/m^2 starting contamination density.

GD	**Surface**					
Decon	**Aluminum**	**CARC-G**	**CARC-T**	**PC**	**Kapton**	**Viton**
Zorflex 1	-	-	-	-	-	-
Zorflex 2	-	-	-	-	-	-
Zorflex 3	-	-	-	-	-	-
M295 1	-	-	-	-	-	-
M295 2	-	-	-	-	-	-
M295 3	-	-	-	-	-	-
+ Control	+	+	n/a	+	n/a	n/a
- Control	-	-	n/a	-	n/a	n/a

Table 29. VX at 1 g/m^2 starting contamination density.

VX	**Surface**					
Decon	**Aluminum**	**CARC-G**	**CARC-T**	**PC**	**Kapton**	**Viton**
Zorflex 1	-	-	-	-	-	-
Zorflex 2	-	-	-	-	-	-
Zorflex 3	-	-	-	-	-	-
M295 1	-	-	-	-	-	-
M295 2	-	-	-	-	-	-
M295 3	-	-	-	-	-	-
+ Control	+	+	n/a	+	n/a	n/a
- Control	-	-	n/a	-	n/a	n/a

Table 30. TGD at 1 g/m^2 starting contamination density.

TGD	**Surface**					
Decon	**Aluminum**	**CARC-G**	**CARC-T**	**PC**	**Kapton**	**Viton**
Zorflex 1	-	-	-	-	-	-
Zorflex 2	-	-	-	-	-	-
Zorflex 3	-	-	-	-	-	-
M295 1	-	-	-	-	-	-
M295 2	-	-	-	-	-	-
M295 3	-	-	-	-	-	-
+ Control	+	n/a	n/a	n/a	n/a	n/a
- Control	-	n/a	n/a	n/a	n/a	n/a

Table 31. HD at 10 g/m^2 starting contamination density.

HD	**Surface**					
Decon	**Aluminum**	**CARC-G**	**CARC-T**	**PC**	**Kapton**	**Viton**
Zorflex 1	-	-	-	-	-	-
Zorflex 2	-	-	-	-	-	-
Zorflex 3	-	-	-	-	-	-
M295 1	-	+	+/-	-	-	-
M295 2	-	-	+/-	-	-	-
M295 3	-	+	-	-	-	-
+ Control	+	+	n/a	+	n/a	n/a
- Control	-	-	n/a	-	n/a	n/a

Table 32. GD at 10 g/m^2 starting contamination density.

GD	**Surface**					
Decon	**Aluminum**	**CARC-G**	**CARC-T**	**PC**	**Kapton**	**Viton**
Zorflex 1	-	-	-	-	-	-
Zorflex 2	-	-	-	-	-	-
Zorflex 3	-	-	-	-	-	-
M295 1	-	-	-	-	-	-
M295 2	-	-	-	-	-	-
M295 3	-	-	-	-	-	-
+ Control	+	+	n/a	+	n/a	n/a
- Control	-	-	n/a	-	n/a	n/a

Table 33. VX at 10 g/m^2 starting contamination density.

VX	**Surface**					
Decon	**Aluminum**	**CARC-G**	**CARC-T**	**PC**	**Kapton**	**Viton**
Zorflex 1	-	-	-	-	-	-
Zorflex 2	-	-	-	-	-	-
Zorflex 3	-	-	-	-	-	-
M295 1	+	+/-	+/-	-	-	-
M295 2	+	-	+/-	-	-	-
M295 3	-	+/-	+/-	-	-	-
+ Control	+	+	n/a	+	n/a	n/a
- Control	-	-	n/a	-	n/a	n/a

Table 34. TGD at 10 g/m^2starting contamination density.

TGD	Surface					
Decon	Aluminum	CARC-G	CARC-T	PC	Kapton	Viton
Zorflex 1	-	-	-	-	-	-
Zorflex 2	-	-	-	-	-	-
Zorflex 3	-	-	-	-	-	-
M295 1	-	-	-	-	-	-
M295 2	-	-	-	-	-	-
M295 3	-	-	-	-	-	-
+ Control	+	n/a	n/a	n/a	n/a	n/a
- Control	-	n/a	n/a	n/a	n/a	n/a

5.8 Mass of Chemical Agent Removed by Wiper

5.8.1 Room Temperature Tests

The results of the manual wiping tests are summarized in Figure 36 through Figure 39. All of the tests conducted with the ACF wipes were moistened with HFE 7200, IAW the wipe use decontamination procedures described in Section 3.

Decontamination Proficiency (DP) is calculated from the following equation:

$$\text{DP} = \frac{\text{(Amount of Agent Initially Deposited - Amount of Residual Agent)}}{\text{(Amount of Agent Initially Deposited)}} \text{ X } 100\% \qquad \text{Equation 6}$$

The contact time of the ACF fabric coupon onto the contaminated material was approximately 10 s for each test material coupon, using the 370 g aluminum block. This time is much less than for those tests performed during the initial screening of candidate materials and reported in Volume I, 32 to 48 s. The lower contact time was derived from the observation of field trials by the 20th Support Command.

The CA data are listed in the following graphs and corresponding tables. The Technology Transition Agreement (TTA, #08-JMDS-06-001T)[6] coordinating the wipe technology development defines the "minimum acceptable" performance thresholds as a reduction of the initial 10 gm/m^2 challenge of Nerve-G, Nerve-V and Blister-H to 90% or below, upon completion of the process. The data in this section was an attempt to satisfy this performance threshold. The data represent the mass of the CA removed by the ACF fabric wipe process, following a 60 min contact at 10 g/m^2 for a variety of surfaces expected to be associated with sensitive military equipment and/or military vehicles and/or aircraft. The interaction between the CA and the surface was not controlled in the tests described within this report. The adsorptive or absorptive processes that regulate diffusion come into play when considering any other material besides aluminum. The steady-state linear diffusion through a one-dimensional substrate, defined by Flick's first law, indicates that the mass flux is proportional to the chemical diffusion coefficient. And, because this coefficient was not published, no attempt can be made to calculate the effect diffusion has on surface cleaning. For example, the wipe process effectively removed >98% of the mass of HD from aluminum, but <70% from polycarbonate.

The data shown in the graphs compare the test material surface to the mass of the CA removed. The materials used in this section included: aluminum, CARC painted aluminum, Air Force Topcoat (AF Top), polycarbonate (PC), Kapton (polyimide), viton, and nylon cloth.

Figure 36 includes the HD data for the mass removal tests and the data is listed in Table 35and Table 36.

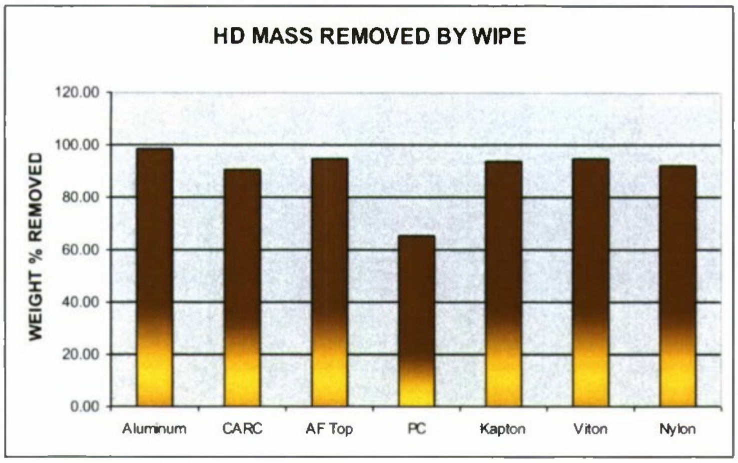


Figure 36. HD mass removed by wipe.

Table 35. HD data for positive controls.

Test #	Sample #	Agent	Cont Density	Surface	Replicate	Fabric	Drop Spread	Extract (mL)	HD [ng/ul]	HD [mg]	AVG
1	PC-A-10-1	HD	10	Aluminum	1			10	2093.96	20.93959	
2	PC-A-10-2	HD	10		2			10	2016.36	20.16360	
3	PC-A-10-3	HD	10		3			10	2239.89	22.39890	21.16736
4	PC-C-10-1	HD	10	CARC	1			10	1268.96	12.68959	
5	PC-C-10-2	HD	10		2			10	1203.17	12.03165	
6	PC-C-10-3	HD	10		3			10	1309.03	13.09028	12.60384
7	PC-AF-10-1	HD	10	AF TopCoat	1			10	1591.82	15.91824	
8	PC-AF-10-2	HD	10		2			10	1605.22	16.05220	
9	PC-AF-10-3	HD	10		3			10	1723.57	17.23572	16.40205
10	PC-PC-10-1	HD	10	PC	1			10	800.26	8.00265	
11	PC-PC-10-2	HD	10		2			10	813.34	8.13339	
12	PC-PC-10-3	HD	10		3			10	772.59	7.72585	7.95396
13	PC-K-10-1	HD	10	Kapton	1			10	1774.18	17.74177	
14	PC-K-10-2	HD	10		2			10	1728.37	17.28367	
15	PC-K-10-3	HD	10		3			10	1857.70	18.57698	17.86747
16	PC-V-10-1	HD	10	Viton	1			10	2234.73	22.34733	
17	PC-V-10-2	HD	10		2			10	2215.11	22.15111	
18	PC-V-10-3	HD	10		3			10	2417.95	24.17952	22.89265
19	PC-N-10-1	HD	10	Nylon	1			10	1883.85	18.83846	
20	PC-N-10-2	HD	10		2			10	1922.67	19.22666	
21	PC-N-10-3	HD	10		3			10	1902.26	19.02259	19.02924

Table 36. HD post panel extract data.

Post Panel Extracts												
Test #	Sample #	Agent	Cont Density	Surface	Replicate	Fabric Area	Drop Spread	Extract (mL)	HD [ng/ul]	HD [mg]	AVG	Wt% Removed
1	PE-A-10-1	HD	10	Aluminum	1	20.2	N	10	33.71	0.33705		
2	PE-A-10-2	HD	10		2	20.2	N	10	31.87	0.31872		
3	PE-A-10-3	HD	10		3	20.2	Y	10	40.78	0.40776	0.35451	98.33
4	PE-C-10-1	HD	10	CARC	1	20.2	Y	10	119.58	1.19584		
5	PE-C-10-2	HD	10		2	20.2	Y	10	126.43	1.26433		
6	PE-C-10-3	HD	10		3	20.2	Y	10	109.27	1.09269	1.18429	90.60
7	PE-AF-10-1	HD	10	AF TopCoat	1	20.2	N	10	55.91	0.55905		
8	PE-AF-10-2	HD	10		2	20.2	N	10	173.09	1.73089		
9	PE-AF-10-3	HD	10		3	20.2	N	10	21.86	0.21860	0.83618	94.90
10	PE-PC-10-1	HD	10	PC	1	20.2	N	10	270.03	2.70027		
11	PE-PC-10-2	HD	10		2	20.2	N	10	293.90	2.93899		
12	PE-PC-10-3	HD	10		3	20.2	N	10	263.26	2.63258	2.75728	65.33
13	PE-K-10-1	HD	10	Kapton	1	20.2	N	10	118.42	1.18415		
14	PE-K-10-2	HD	10		2	20.2	N	10	116.76	1.16763		
15	PE-K-10-3	HD	10		3	20.2	N	10	97.69	0.97686	1.10955	93.79
16	PE-V-10-1	HD	10	Viton	1	20.2	N	10	82.36	0.82358		
17	PE-V-10-2	HD	10		2	20.2	N	10	85.78	0.85780		
18	PE-V-10-3	HD	10		3	20.2	N	10	195.13	1.95128	1.21088	94.71
19	PE-N-10-1	HD	10	Nylon	1	20.2	N	10	160.70	1.60700		
20	PE-N-10-2	HD	10		2	20.2	N	10	193.23	1.93234		
21	PE-N-10-3	HD	10		3	20.2	N	10	106.43	1.06429	1.53454	91.94

Figure 37 includes the VX data for the mass removal tests and the data is listed in Table 37 and Table 38.

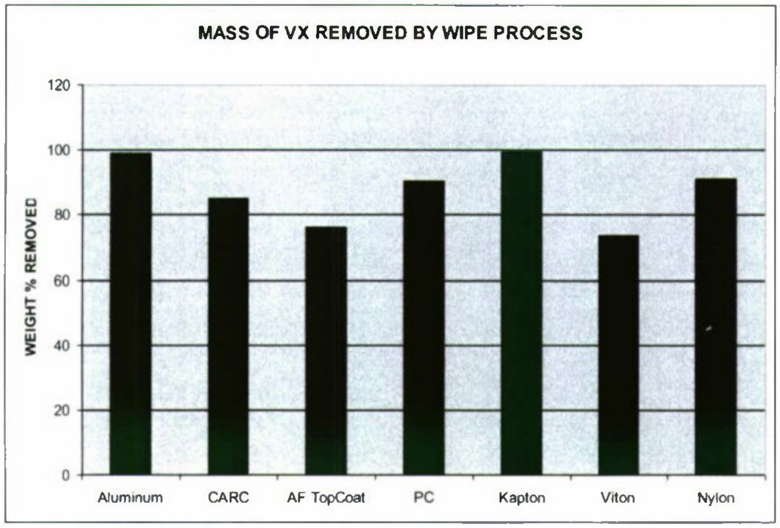


Figure 37. Mass of VX removed by wipe process.

Table 37. VX positive controls.

Positive Controls

Test #	Sample #	Agent	Cont Density	Surface	Replicate	Fabric Area	Drop Spread	Extract (mL)	HD [ng/ul]	HD [mg]	AVG
1	PC-A-10-1	VX	10	Aluminum	1			20	3624.3946	18.1220	
2	PC-A-10-2	VX	10		2			20	3114.4046	15.5720	
3	PC-A-10-3	VX	10		3			20	2956.5481	14.7827	16.158912
4	PA-C-10-1	VX	10	CARC	1			20	3631.6060	18.1580	
5	PC-C-10-2	VX	10		2			20	3223.2038	16.1160	
6	PC-C-10-3	VX	10		3			20	3565.7443	17.8287	17.36759
7	PC-AF-10-1	VX	10	AF Top Coat	1			20	2776.0652	13.8803	
8	PC-AF-10-2	VX	10		2			20	2355.4896	11.7774	
9	PC-AF-10-3	VX	10		3			20	2441.9269	12.2096	12.622469
10	PC-PC-10-1	VX	10	PC	1			20	3818.9034	19.0945	
11	PC-PC-10-2	VX	10		2			20	3338.0566	16.6903	
12	PC-PC-10-3	VX	10		3			20	3291.9765	16.4599	17.414894
13	PC-K-10-1	VX	10	Kapton	1			20	3641.4969	18.2075	
14	PC-K-10-2	VX	10		2			20	3606.9285	18.0346	
15	PC-K-10-3	VX	10		3			20	3762.7671	18.8138	18.351988
16	PC-V-10-1	VX	10	Viton	1			20	3328.3311	16.6417	
17	PC-V-10-2	VX	10		2			20	2972.8895	14.8644	
18	PC-V-10-3	VX	10		3			20	3458.4337	17.2922	16.266091
19	PC-N-10-1	VX	10	Nylon	1			20	3563.0979	17.8155	
20	PC-N-10-2	VX	10		2			20	2804.1829	14.0209	
21	PC-N-10-3	VX	10		3			20	3628.2650	18.1413	16.659243
22	PC-ABS-10-1	VX	10	ABS	1			20	4264.1088	21.3205	
23	PC-ABS-10-2	VX	10		2			20	4087.4574	20.4373	
24	PC-ABS-10-3	VX	10		3			20	3987.4517	19.9373	20.56503

Table 38. VX post panel extract data.

Sample #	Agent	Cont Density	Surface	Replicate	Fabric Area	Drop Spread	Extract (mL)	HD [ng/ul]	HD [mg]	AVG	Wt% Removed
PE-A-10-1	VX	10	Aluminum	1	19.6	N	10	54.1002	0.1353		
PE-A-10-2	VX	10		2	19.6	N	10	147.6166	0.3690		
PE-A-10-3	VX	10		3	19.6	N	10	36.6341	0.0916	0.1986258	98.86
PE-C-10-1	VX	10	CARC	1	19.6	N	10	1037.8250	2.5946		
PE-C-10-2	VX	10		2	19.6	N	10	940.0083	2.3500		
PE-C-10-3	VX	10		3	19.6	Y	10	1142.1915	2.8555	2.6000207	85.03
PE-AF-10-1	VX	10	AF Top Coat	1	19.6	N	10	1244.1763	3.1104		
PE-AF-10-2	VX	10		2	19.6	N	10	1315.1654	3.2879		
PE-AF-10-3	VX	10		3	19.6	N	10	1049.6345	2.6241	3.0074802	76.17
PE-PC-10-1	VX	10	PC	1	19.6	N	10	673.1872	1.6830		
PE-PC-10-2	VX	10		2	19.6	N	10	631.1760	1.5779		
PE-PC-10-3	VX	10		3	19.6	N	10	531.6060	1.3290	1.5299744	90.53
PE-K-10-1	VX	10	Kapton	1	19.6	N	10	24.9901	0.0625		
PE-K-10-2	VX	10		2	19.6	N	10	18.8703	0.0472		
PE-K-10-3	VX	10		3	19.6	N	10	102.9590	0.2574	0.1223495	99.33
PE-V-10-1	VX	10	Viton	1	19.6	Y	10	1880.5971	4.7015		
PE-V-10-2	VX	10		2	19.6	N	10	1601.6358	4.0041		
PE-V-10-3	VX	10		3	19.6	N	10	1616.9848	4.0425	4.2493481	73.88
PE-N-10-1	VX	10	Nylon	1	19.6	N	10	846.2934	2.1157		
PE-N-10-2	VX	10		2	19.6	N	10	646.9550	1.6174		
PE-N-10-3	VX	10		3	19.6	N	10	701.0404	1.7526	1.828574	91.11

Figure 38 includes the GD data for the mass removal tests and the data is listed in Table 39 and Table 40.

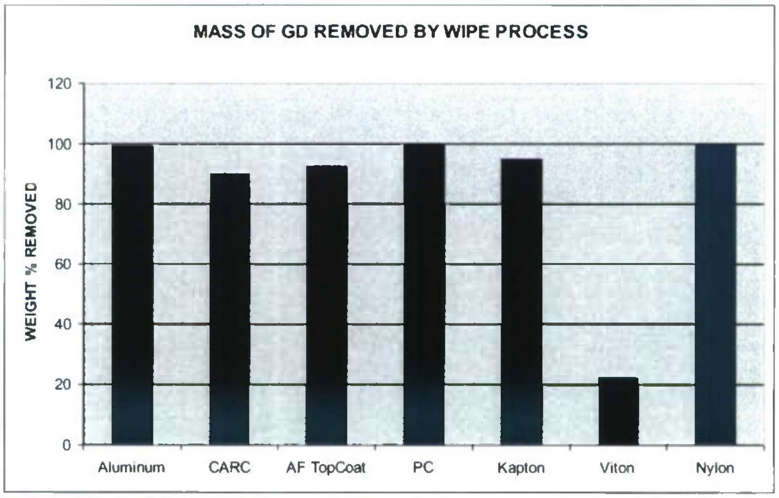


Figure 38. Mass of GD removed by wipe process.

Table 39. GD positive control data.

Positive Controls												
Test #	Sample #	Agent	Cont Density	Surface	Replicate	Fabric Area	Drop Spread	Extract (mL)	GD [ng/ul]	GD [mg]	AVG	% Removed
1	PC-A-10-1	GD	10	Aluminum	1		N	20	473.85	9.47705		
2	PC-A-10-2	GD	10		2		N	20	487.85	9.75700	9.61702	
3	PC-C-10-1	GD	10	CARC	1		N	20	345.59	6.91172		
4	PC-C-10-2	GD	10		2		N	20	336.18	6.72359	6.81766	
5	PC-AF-10-1	GD	10	AF TopCoat	1		N	20	389.64	7.79282		
6	PC-AF-10-2	GD	10		2		N	20	393.84	7.87681	7.83481	
7	PC-PC-10-1	GD	10	PC	1		N	20	166.27	3.32533		
8	PC-PC-10-2	GD	10		2		N	20	167.32	3.34640	3.33587	
9	PC-K-10-1	GD	10	Kapton	1		N	20	278.29	5.56585		
10	PC-K-10-2	GD	10		2		N	20	272.18	5.44363	5.50474	
11	PC-V-10-1	GD	10	Viton	1		N	20	346.73	6.93460		
12	PC-V-10-2	GD	10		2		N	20	351.11	7.02220	6.97840	
13	PC-N-10-1	GD	10	Nylon	1		N	20	311.28	6.22569		
14	PC-N-10-2	GD	10		2		N	20	322.68	6.45357	6.33963	

Table 40. GD post panel extract data.

Post Panel Extracts												
Test #	Sample #	Agent	Cont Density	Surface	Replicate	Fabric Area	Drop Spread	Extract (mL)	GD [ng/ul]	GD [mg]	AVG	% Removed
1	PE-A-10-1	GD	10.00	Aluminum	1	20.2		10	7.27	0.07266		
2	PE-A-10-2	GD	10.00		2	20.2		10	6.66	0.06664	0.06965	99.28
3	PE-C-10-1	GD	10.00	CARC	1	20.2		10	67.07	0.67071		
4	PE-C-10-2	GD	10.00		2	20.2		10	67.46	0.67456	0.67263	90.13
5	PE-AF-10-1	GD	10.00	AF TopCoat	1	20.2		10	44.40	0.44397		
6	PE-AF-10-2	GD	10.00		2	20.2		10	68.24	0.68238	0.56318	92.81
7	PE-PC-10-1	GD	10.00	PC	1	20.2		10	0.00	0.00000		
8	PE-PC-10-2	GD	10.00		2	20.2		10	0.00	0.00000	0.00000	100.00
9	PE-K-10-1	GD	10.00	Kapton	1	20.2		10	30.05	0.30053		
10	PE-K-10-2	GD	10.00		2	20.2		10	26.53	0.26531	0.28292	94.86
11	PE-V-10-1	GD	10.00	Viton	1	20.2		10	541.39	5.41387		
12	PE-V-10-2	GD	10.00		2	20.2		10	549.20	5.49199	5.45293	21.86
13	PE-N-10-1	GD	10.00	Nylon	1	20.2		10	0.24	0.00237		
14	PE-N-10-2	GD	10.00		2	20.2		10	-1.03	-0.01027	-0.00395	100.06

Figure 39 includes the TGD data for the mass removal tests and the data is listed in Table 41 and Table 42.

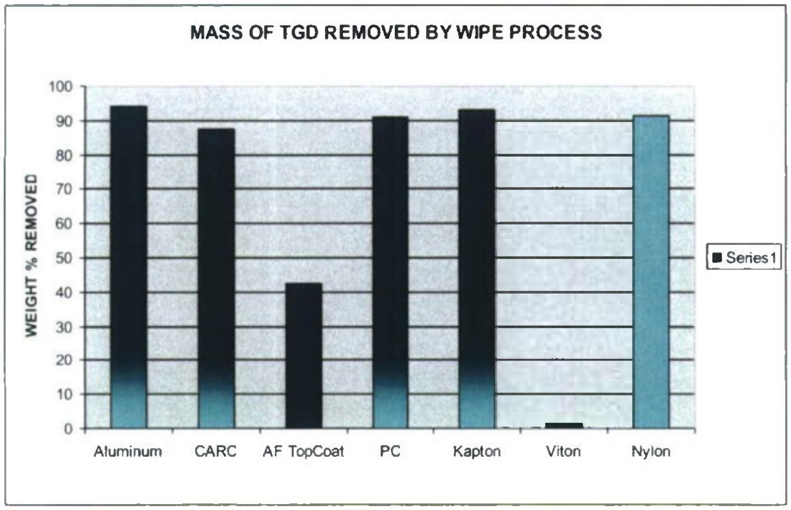


Figure 39. Mass of TGD removed by wipe process.

Table 41. TGD positive control data.

Test #	Sample #	Agent	Cont Density	Surface	Replicate	Fabric Area	Drop Spread	Extract (mL)	GD [ng/ul]	GD [mg]	AVG
1	PC-A-10-1	T-GD	10	Aluminum	1		Y	20	604.00	12.07992	
2	PC-A-10-2	T-GD	10		2		Y	20	603.11	12.06228	12.07110
3	PC-C-10-1	T-GD	10	CARC	1		Y	20	375.59	7.51175	
4	PC-C-10-2	T-GD	10		2		Y	20	373.98	7.47966	7.49571
5	PC-AF-10-1	T-GD	10	AF TopCoat	1		Y	20	235.70	4.71401	
6	PC-AF-10-2	T-GD	10		2		Y	20	235.34	4.70678	4.71039
7	PC-PC-10-1	T-GD	10	PC	1		Y	20	296.62	5.93246	
8	PC-PC-10-2	T-GD	10		2		Y	20	295.70	5.91395	5.92320
9	PC-K-10-1	T-GD	10	Kapton	1		Y	20	381.36	7.62712	
10	PC-K-10-2	T-GD	10		2		Y	20	381.04	7.62076	7.62394
11	PC-V-10-1	T-GD	10	Viton	1		Y	20	293.47	5.86942	
12	PC-V-10-2	T-GD	10		2		Y	20	290.03	5.80061	5.83502
13	PC-N-10-1	T-GD	10	Nylon	1		Y	20	395.51	7.91019	
14	PC-N-10-2	T-GD	10		2		Y	20	399.64	7.99289	7.95154

Table 42. TGD post panel extract data.

Post Panel Extracts												
Test #	**Sample #**	**Agent**	**Cont Density**	**Surface**	**Replicate**	**Fabric Area**	**Drop Spread**	**Extract (mL)**	**TGD [ng/ul]**	**TGD [mg]**	**AVG**	**Wt% Removed**
1	PE-A-10-1	T-GD	10	Aluminum	1			10	71.13	0.71135		
2	PE-A-10-2	T-GD	10		2			10	74.01	0.74012	0.72573	93.99
3	PE-C-10-1	T-GD	10	CARC	1			10	93.88	0.93876		
4	PE-C-10-2	T-GD	10		2			10	94.09	0.94093	0.93984	87.46
5	PE-AF-10-1	T-GD	10	AF TopCoat	1			10	263.91	2.63906		
6	PE-AF-10-2	T-GD	10		2			10	278.45	2.78450	2.71178	42.43
7	PE-PC-10-1	T-GD	10	PC	1			10	53.16	0.53165		
8	PE-PC-10-2	T-GD	10		2			10	52.09	0.52095	0.52630	91.11
9	PE-K-10-1	T-GD	10	Kapton	1			10	53.45	0.53454		
10	PE-K-10-2	T-GD	10		2			10	54.87	0.54871	0.54162	92.90
11	PE-V-10-1	T-GD	10	Viton	1			10	631.25	6.31248		
12	PE-V-10-2	T-GD	10		2			10	805.92	8.05919	7.18583	1.23
13	PE-N-10-1	T-GD	10	Nylon	1			10	67.81	0.67810		
14	PE-N-10-2	T-GD	10		2			10	67.93	0.67925	0.67868	91.46

These data were the result of a single ACF fabric (multi-layered) wipe used on each contaminated test material. The ACF fabric wipe coupon used consisted of several layers and was contacted as follows:

- Layer 1 (contaminant contact layer): PFG 39278
- Layer 2: Zorflex 50K
- Layer 3: Zorflex 100 meso
- Layer 4: Tyvek® 1443R

The fabrics were all punch cut using the same punch dye, with a surface area of 16.9 cm^2 for each layer. The weights of each layer are shown in Table 43 and were the result of five replicates of each layer:

Table 43. Weights of associated ACF fabric layers.

Layer	Fabric	Weight [gm]
1	PFG 39278	0.070
2	Zorflex 50K	0.340
3	Zorflex 100	0.480

5.8.2 Elevated and Reduced Surface Temperature Tests

The test results are reported for mass removal of HD by the ACF wipes under three different surface temperatures: room temperature (24 ± 2 °C), reduced temperature (14 °C), and an elevated test material coupon temperature (49 °C). The results are shown in Table 44 through Table 45. The tests were conducted with one CA (HD) and two test material surfaces (coupons), aluminum and CARC. The following Figure 40, shows the data in a bar chart.

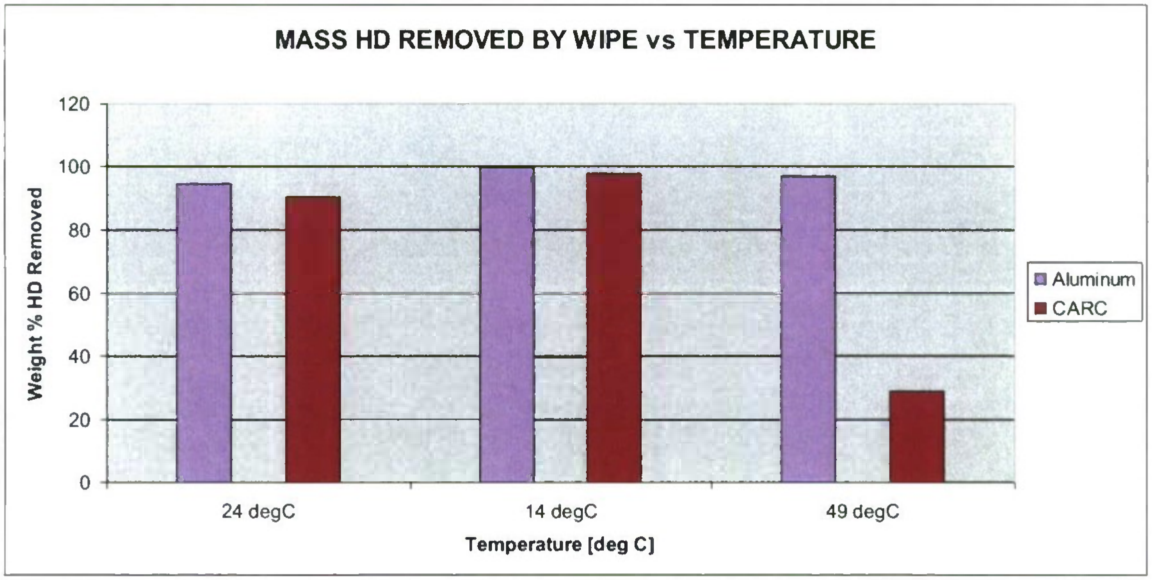


Figure 40. Mass HD removal and effect on elevated and reduced temperature.

From the preceding graph (Figure 40), the results indicate that the ACF fabric wipe removed >90% by weight of the HD from both aluminum and CARC at all three temperature conditions, except the 49 °C CARC. Under this condition it was observed that the HD had completely wicked into the paint, as evidenced by a dramatic drop spread. Again, the interaction between surface and contaminant was evident, and no further determination (measurements) was made of this interaction, because it was outside the scope of this effort.

Table 44. Mass HD removal at 24 °C.

Test #	Sample #	Agent	Cont Density	Surface	Replicate	Fabric Area	Drop Spread	Extract (mL)	HD [ng/µL]	HD [mg]	AVG	% Removed
1	ICV	HD							158.793			
2	A-10-24-1	HD	10	Aluminum	1	20.2	N	20	39.134	0.78268		
3	A-10-24-2	HD	10	Aluminum	2	20.2	N	20	58.797	1.17594		
4	A-10-24-3	HD	10	Aluminum	3	20.2	N	20	47.71	0.95420		
5	A-10-24-4	HD	10	Aluminum	4	20.2	N	20	48.496	0.96992	0.97069	94.52
6	A-10-PC-1	HD	10	Aluminum	1		N	20	873.634	17.47268		
7	A-10-PC-2	HD	10	Aluminum	2		N	20	896.942	17.93884	17.70576	
8	Z-A-10-24-1	HD	10	Aluminum	1		N	20	850.436	17.00872		
9	Z-A-10-24-2	HD	10	Aluminum	2		N	20	839.989	16.79978		
10	Z-A-10-24-3	HD	10	Aluminum	3		N	20	824.292	16.48584		
11	Z-A-10-24-4	HD	10	Aluminum	4		N	20	724.156	14.48312	16.19437	
12	CCV	HD							160.083			
13	C-10-24-1	HD	10	CARC	1	20.2	N	20	86.745	1.73490		
14	C-10-24-2	HD	10	CARC	2	20.2	N	20	94.564	1.89128		
15	C-10-24-3	HD	10	CARC	3	20.2	N	20	78.303	1.56606		
16	C-10-24-4	HD	10	CARC	4	20.2	N	20	94.652	1.89304	1.77132	90.14
17	C-10-24-PC1	HD	10	CARC	1		N	20	900.735	18.01470		
18	C-10-24-PC2	HD	10	CARC	2		N	20	895.94	17.91880	17.96675	
19	Z-C-10-24-1	HD	10	CARC	1		N	20	815.217	16.30434		
20	Z-C-10-24-2	HD	10	CARC	2		N	20	799.321	15.98642		
21	Z-C-10-24-3	HD	10	CARC	3		N	20	766.252	15.32504		
22	Z-C-10-24-4	HD	10	CARC	4		N	20	763.013	15.26026	15.71902	
23	ABS-1	HD	10		1		N	20	874.102	17.48204		
24	ABS-2	HD	10		2		N	20	895.388	17.90776		
25	ABS-3	HD	10		3		N	20	897.308	17.94616	17.77865	
26	CCV	HD							158.443			

Table 45. Mass HD removal at 14 °C.

Test #	Sample #	Agent	Cont Density	Surface	Replicate	Fabric Area	Drop Spread	Extract (mL)	HD [ng/µL]	HD [mg]	AVG	% Removed
	ICV	HD							**169.43**			
1	A-10-15-1	HD	10	Aluminum	1	20.2	N	10	9.093	0.09093		
2	A-10-15-2	HD	10	Aluminum	2	20.2	N	10	5.49	0.05490		
3	A-10-15-3	HD	10	Aluminum	3	20.2	N	10	9.795	0.09795		
4	A-10-15-4	HD	10	Aluminum	4	20.2	Y	10	6.736	0.06736		
5	A-10-15-5	HD	10	Aluminum	5	20.2	N	10	7.724	0.07724	0.07768	99.55
6	A-10-15-PC1	HD	10	Aluminum	1		N	20	821.858	16.43716		
7	A-10-15-PC2	HD	10	Aluminum	2		N	20	888.75	17.77500	17.10608	
8	Z-A-10-15-1	HD	10	Aluminum	1			20	847.124	16.94248		
9	Z-A-10-15-2	HD	10	Aluminum	2			20	849.252	16.98504		
10	Z-A-10-15-3	HD	10	Aluminum	3			20	856.93	17.13860		
11	Z-A-10-15-4	HD	10	Aluminum	4			20	858.608	17.17216		
12	Z-A-10-15-5	HD	10	Aluminum	5			20	839.05	16.78100	17.00386	
13	CCV	HD							168.546			
14	C-10-15-1	HD	10	CARC	1	20.2	N	10	36.351	0.36351		
15	C-10-15-2	HD	10	CARC	2	20.2	N	10	35.653	0.35653		
16	C-10-15-3	HD	10	CARC	3	20.2	N	10	32.304	0.32304		
17	C-10-15-4	HD	10	CARC	4	20.2	N	10	48.174	0.48174		
18	C-10-15-5	HD	10	CARC	5	20.2	N	10	35.018	0.35018	0.37500	97.92
19	C-10-15-PC1	HD	10	CARC	1		N	20	898.052	17.96104		
20	C-10-15-PC2	HD	10	CARC	2		N	20	908.578	18.17156	18.06630	
21	Z-C-10-15-1	HD	10	CARC	1			20	871.964	17.43928		
22	Z-C-10-15-2	HD	10	CARC	2			20	876.676	17.53352		
23	Z-C-10-15-3	HD	10	CARC	3			20	871.816	17.43632		
24	Z-C-10-15-4	HD	10	CARC	4			20	860.942	17.21884		
25	Z-C-10-15-5	HD	10	CARC	5			20	856.854	17.13708	17.35301	
26	ABS-1	HD	10		1			20	932.642	18.65284		
27	ABS-2	HD	10		2			20	940.067	18.80134		
28	ABS-3	HD	10		3			20	925.099	18.50198	18.65205	
29	CCV	HD							167.865			

Table 46. Mass HD removal at 49 °C.

Test #	Sample #	Agent	Cont Density	Surface	Replicate	Fabric Area	Drop Spread	Extract (mL)	HD [ng/µL]	HD [mg]	AVG HD [mg]	% Removed
	ICV	HD							167.159			
1	A-10-49-1	HD	10	Aluminum	1	20.2	Y	10	31.433	0.3143		
2	A-10-49-2	HD	10	Aluminum	2	20.2	Y	10	62.35	0.6235		
3	A-10-49-3	HD	10	Aluminum	3	20.2	Y	10	63.518	0.6352		
4	A-10-49-4	HD	10	Aluminum	4	20.2	Y	10	52.003	0.5200		
5	A-10-49-5	HD	10	Aluminum	5	20.2	Y	10	29.144	0.2914	0.4769	96.71
6	A-10-49-PC1	HD	10	Aluminum	1		Y	20	803.587	16.0717		
7	A-10-49-PC2	HD	10	Aluminum	2		Y	20	644.252	12.8850	14.4784	
8	Z-A-10-49-1	HD	10	Aluminum	1			20	411.573	8.2315		
9	Z-A-10-49-2	HD	10	Aluminum	2			20	213.739	4.2748		
10	Z-A-10-49-3	HD	10	Aluminum	3			20	150.377	3.0075		
11	Z-A-10-49-4	HD	10	Aluminum	4			20	288.673	5.7735		
12	Z-A-10-49-5	HD	10	Aluminum	5			20	576.436	11.5287	6.5632	
	CCV								151.411			
13	C-10-49-1	HD	10	CARC	1	20.2	Y	10	1146.83	11.4683		
14	C-10-49-2	HD	10	CARC	2	20.2	Y	10	1191.747	11.9175		
15	C-10-49-3	HD	10	CARC	3	20.2	Y	10	1325.148	13.2515		
16	C-10-49-4	HD	10	CARC	4	20.2	Y	10	1141.286	11.4129		
17	C-10-49-5	HD	10	CARC	5	20.2	Y	10	1244.764	12.4476	12.0996	29.00
18	C-10-49-PC1	HD	10	CARC	1		Y	20	830.661	16.6132		
19	C-10-49-PC2	HD	10	CARC	2		Y	20	873.524	17.4705	17.0419	
20	Z-C-10-49-1	HD	10	CARC	1			20	4.227	0.0845		
21	Z-C-10-49-2	HD	10	CARC	2			20	5.628	0.1126		
22	Z-C-10-49-3	HD	10	CARC	3			20	6.7	0.1340		
23	Z-C-10-49-4	HD	10	CARC	4			20	6.049	0.1210		
24	Z-C-10-49-5	HD	10	CARC	5			20	6.117	0.1223	0.1149	
25	ABS-1	HD	10		1			20	887.68	17.7536		
26	ABS-2	HD	10		2			20	884.152	17.6830		
27	ABS-3	HD	10		3			20	871.789	17.4358	17.6241	
	CCV								156.649			

In the following three tables (Table 47 through Table 49), the HD mass balances are provided from the tests as an indication of the quality of the mass removal efficiencies, shown in Figure 40. The %PC is the percent of recovery following the decontamination process (wipe) as compared to the positive control (PC). The mass balance for the elevated surface temperature clearly shows an effect from increase in temperature. Two issues could be ascertained from this data—(1) evaporative effects caused a loss of the HD, and (2) the HD paint interaction was pronounced.

Table 47. Mass balance, room temperature.

Surface	HD [mg]	%PC
Aluminum	17.16505	96.95
CARC	17.49034	97.35

Table 48. Mass balance, reduced temperature.

Surface	HD [mg]	%PC
Aluminum	17.08153	99.86
CARC	17.72801	98.13

Table 49. Mass balance, elevated temperature.

Surface	HD [mg]	%PC
Aluminum	7.0401	48.62
CARC	12.2144	71.67

The following results are from a side-by-side comparison of the difference between 1 and 10 g/m^2 initial contamination density, and the wipe's ability to remove this mass. These tests were conducted at room temperature using HD on aluminum and CARC test coupons. The results are shown in the following graph and displayed in Table 50 through Table 51. The following graph (Figure 41) displays the y-axis plot is in log scale compared to the initial starting challenge.

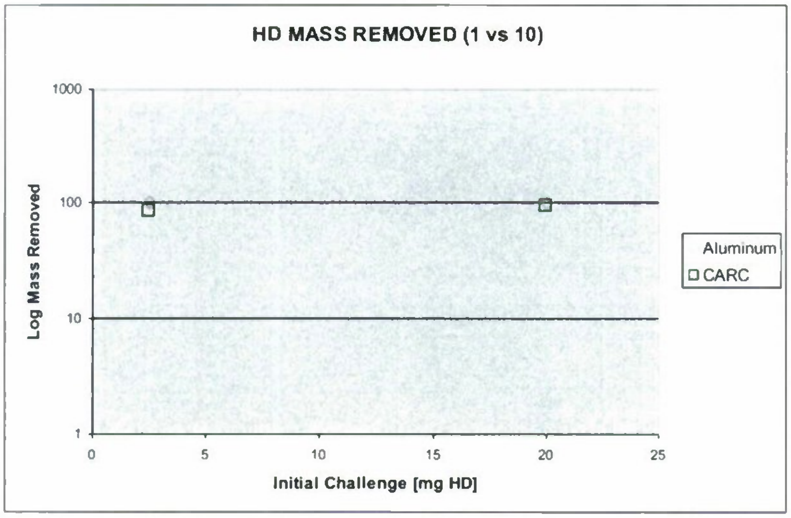


Figure 41. HD mass removed (1 vs 10).

Table 50. Mass HD removed (1 vs 10) aluminum coupon.

Sample #	Agent	Cont Density	Surface	Replicate	Fabric Area	Drop Spread	Extract (mL)	HD [ng/µL]	HD [mg]	AVG	% Removed By Wt
A-1-1	HD	1	Aluminum	1	20.2	Y	10	0.3245	0.0032		
A-1-2	HD	1	Aluminum	2	20.2	Y	10	0.0000	0.0000		
A-1-3	HD	1	Aluminum	3	20.2	Y	10	0.3958	0.0040		
A-1-4	HD	1	Aluminum	4	20.2	N	10	0.5573	0.0056		
A-1-5	HD	1	Aluminum	5	20.2	N	10	0.5184	0.0052	0.0036	99.83
A-PC-1-1	HD	1	Aluminum	1			10	228.9277	2.2893		
A-PC-1-2	HD	1	Aluminum	2			10	185.4343	1.8543		
A-PC-1-3	HD	1	Aluminum	3			10	222.9487	2.2295	2.1244	
A-NC-1	▨	0	Aluminum	1			10	ND	0.0000		
ABS-1-1	HD	1	ABS	1			10	263.7926	2.6379		
ABS-1-2	HD	1	ABS	2			10	217.3559	2.1736		
ABS-1-3	HD	1	ABS	3			10	229.2964	2.2930	2.3681	
A-10-1	HD	10	Al	1	20.2	N	10	5.5350	0.0554		
A-10-2		10		2	20.2	N	10	5.4026	0.0540		
A-10-3		10		3	20.2	Y	10	6.2577	0.0626		
A-10-4		10		4	20.2	N	10	2.1052	0.0211		
A-10-5		10		5	20.2	N	10	1.0494	0.0105	0.0407	99.78
A-PC-10-1	HD	10	Pos Panel Cont	1			20	889.1737	17.7835		
A-PC-10-2		10		2			20	916.3621	18.3272		
A-PC-10-3		10		3			20	944.1597	18.8832	18.3313	
A-NC-10		0	Neg Panel Cont				10	5.9119	0.0591		
ABS-10-1		10		1			20	967.3197	19.3464		
ABS-10-2		10		2			20	823.3810	16.4676		
ABS-10-3		10		3			20	996.6602	19.9332	18.5824	
Z-A-1-1	HD	1	Zorflex extracts	1			10	100.1377	1.0014		
Z-A-1-2	HD	1	Zorflex extracts	2			10	78.3807	0.7838		
Z-A-1-3	HD	1	Zorflex extracts	3			10	115.9137	1.1591		
Z-A-1-4	HD	1	Zorflex extracts	4			10	234.8155	2.3482		
Z-A-1-5	HD	1	Zorflex extracts	5			10	188.3341	1.8833	1.4352	
Z-A-10-1	HD		Zorflex extract	1			20	665.6464	13.3129		
Z-A-10-2			Zorflex extract	2			20	679.6499	13.5930		
Z-A-10-3			Zorflex extract	3			20	763.5712	15.2714		
Z-A-10-4			Zorflex extract	4			20	891.0105	17.8202		
Z-A-10-5			Zorflex extract	5			20	600.5567	12.0111	14.4017	

Table 51. Mass HD removed (1 vs 10) CARC coupon.

Sample #	Agent	Cont Density	Surface	Replicate	Fabric Area	Drop Spread	Extract (mL)	HD [ng/µL]	HD [mg]	AVG	% Removed
C-1-1	HD	1	CARC	1	20.2	N	10	27.484	0.2748		
C-1-2	HD	1	CARC	2	20.2	N	10	38.187	0.3819		
C-1-3	HD	1	CARC	3	20.2	N	10	34.424	0.3442		
C-1-4	HD	1	CARC	4	20.2	N	10	34.641	0.3464		
C-1-5	HD	1	CARC	5	20.2	N	10	33.101	0.3310	0.3357	84.01
C-PC-1-1	HD	1	Pos Panel Cont	1		N	10	204.634	2.0463		
C-PC-1-2	HD	1	Pos Panel Cont	2		N	10	215.11	2.1511	2.0987	
ABS-1-1	HD	1	Absolute Cont	1			10	211.7	2.1170		
ABS-1-2	HD	1	Absolute Cont	2			10	209.804	2.0980	2.1075	
C-10-1	HD	10	CARC	1	20.2	N	10	132.222	1.3222		
C-10-2	HD	10	CARC	2	20.2	N	10	144.342	1.4434		
C-10-3	HD	10	CARC	3	20.2	N	10	142.913	1.4291		
C-10-4	HD	10	CARC	4	20.2	N	10	186.798	1.8680		
C-10-5	HD	10	CARC	5	20.2	N	10	112.71	1.1271	1.4380	91.31
C-PC-10-1	HD	10	Pos Panel Cont	1		N	20	836.433	16.7287		
C-PC-10-2	HD	10	Pos Panel Cont	2		Y	20	819.112	16.3822	16.5555	
ABS-1-1	HD	10	Absolute Cont	1			20	838.383	16.7677		
ABS-1-2	HD	10	Absolute Cont	2			20	859.328	17.1866	16.9771	
Zorflex Extracts											
Z-1-1	HD	1	Zorflex	1			20	86.749	1.7350		
Z-1-2	HD	1	Zorflex	2			20	89.481	1.7896		
Z-1-3	HD	1	Zorflex	3			20	90.158	1.8032		
Z-1-4	HD	1	Zorflex	4			20	99.717	1.9943		
Z-1-5	HD	1	Zorflex	5			20	78.538	1.5708	1.7786	
Z-10-1	HD	10	Zorflex	1			20	655.51	13.1102		
Z-10-2	HD	10	Zorflex	2			20	589.051	11.7810		
Z-10-3	HD	10	Zorflex	3			20	532.82	10.6564		
Z-10-4	HD	10	Zorflex	4			20	624.366	12.4873		
Z-10-5	HD	10	Zorflex	5			20	577.024	11.5405	11.9151	

The mass balances are included in the tables below (Table 52 and Table 53), which calculates the mass of the HD added from the positive controls (PC) against the mass of the HD recovered from the coupon surface added to the Zorflex extracts.

Table 52. HD mass recovered from aluminum.

HD Mass Balance			
Surface	Cont Density	HD [mg]	% PC
Aluminum	1	2.1280	100.17
Aluminum	10	14.4424	77.72

Table 53. HD mass recovered from CARC

HD Mass Balance			
Surface	Cont Density	HD [mg]	% PC
CARC	1	2.1142	100.7398
CARC	10	13.3531	80.6565

5.9 Vapor Analysis of Spent Wipe

The concentration of CA from a spent wipe, contaminated by CA from a decontamination process, is shown in the following tables and corresponding graphs. A measured amount (spike) of CA (HD or GD) was pipetted onto a 2 in. diameter borosilicate glass disc (McMaster-Carr, part # 01250200) as a film across the surface. The layered fabric was positioned directly onto the glass disc onto which a 1 kg weight was applied for 10 min. The fabric layers consisted of PFG 39278, Zorflex 50K, and Zorflex 100 meso. The PFG layer made the initial contact with the contaminated surface. Following the 10 min contact period, the fabric layers for each sample (1–6) were inserted into separate vapor cups and monitored using the Perkin-Elmer system for HD or the Marks system for GD. The results for HD are shown in Table 54 and shown in the corresponding graph in Figure 42.

Table 54. Test results for HD off-gassing from spent wipe .

Sample	Fabric	Spike [µL]	Spike [gm/m^2]	Volume [mL]	Area Counts	HD [ng]	HD [mg]	HD [mg/m^3]
15-1	PFG/50K/100meso	1.6	1.0031	4500	1604.66	720.8068	0.000721	0.16018
15-2	PFG/50K/100meso	3.2	2.0061	4500	3462.19	1555.2017	0.001555	0.34560
15-3	PFG/50K/100meso	4.8	3.0092	4500	7709.16	3462.9234	0.003463	0.76954
15-4	PFG/50K/100meso	8	5.0153	4500	12173.1	5468.1071	0.005468	1.21513
15-5	PFG/50K/100meso	16	10.0306	4500	36854.3	16554.8019	0.016555	3.67884
15-6	50K-100meso	4.8	3.0092	4500	5712.4	2565.9869	0.002566	0.57022
				60-min				
60-1	PFG/50K/100meso	1.6	1.0031	13500	205	92.0852	0.000092	0.00682
60-2	PFG/50K/100meso	3.2	2.0061	13500	1552.75	697.4890	0.000697	0.05167
60-3	PFG/50K/100meso	4.8	3.0092	13500	1310.92	588.8599	0.000589	0.04362
60-4	PFG/50K/100meso	8	5.0153	13500	1547.49	695.1262	0.000695	0.05149
60-5	PFG/50K/100meso	16	10.0306	13500	4416.34	1983.8020	0.001984	0.14695
60-6	50K-100meso	4.8	3.0092	13500	1437.87	645.8854	0.000646	0.04784
				120-min				
120-1	PFG/50K/100meso	1.6	1.0031	18000	75.25	33.8020	0.000034	0.00188
120-2	PFG/50K/100meso	3.2	2.0061	18000	281.68	126.5295	0.000127	0.00703
120-3	PFG/50K/100meso	4.8	3.0092	18000	319.84	143.6708	0.000144	0.00798
120-4	PFG/50K/100meso	8	5.0153	18000	271.82	122.1004	0.000122	0.00678
120-5	PFG/50K/100meso	16	10.0306	18000	1330.4	597.6103	0.000598	0.03320
120-6	50K-100meso	4.8	3.0092	18000	1017.3	456.9670	0.000457	0.02539
				180-min				
180-3	PFG/50K/100meso	4.8	3.0092	18000	72.3	32.4769	0.000032	0.00180
180-4	PFG/50K/100meso	8	5.0153	18000	98.8	44.3806	0.000044	0.00247
180-5	PFG/50K/100meso	16	10.0306	18000	205.1	92.1301	0.000092	0.00512

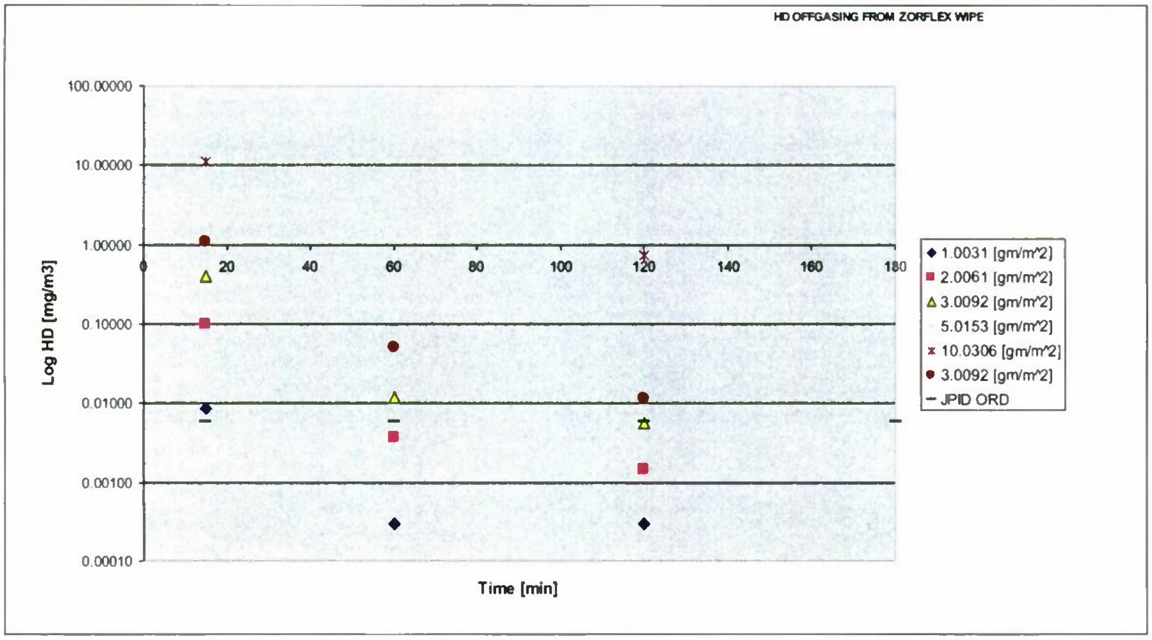


Figure 42. Semi-log plot of HD off-gassing from spent wipe.

The fabric layers were punch cut into 20.25 cm^2 layers. The layers were stacked one on top of the other with the edge stapled together, to assist in transport from the glass disc to the vapor cup. The results were reported as the CA concentration per volume, as a function of time, and the initial CA contamination load per area (m^2) of wipe fabric.

Similar to HD, the results for GD are shown in Table 55 and corresponding Figure 43.

Table 55. Test results for GD off-gassing from spent wipe.

Sample	Fabric	Spike [µL]	Spike [gm/m^2]	Volume [mL]	Tube #	GD [ng]	GD [mg]	GD [mg/m3]
				15-min				
1_15	PFG/50K/100meso	2	1.01	4500	96317	94.03	0.000094	0.020896
2_15	PFG/50K/100meso	4	2.02	4500	96316	247.51	0.000248	0.055002
3_15	PFG/50K/100meso	6	3.03	4500	96315	500	0.000500	0.111111
4_15	PFG/50K/100meso	10	5.05	4500	96329	500	0.000500	0.111111
5_15	PFG/50K/100meso	20	10.09	4500	96326	500	0.000500	0.111111
6_15	50K-100meso	6	3.03	3300	96328	324.69	0.000325	0.098391
				60-min				
1_60	PFG/50K/100meso	2	1.01	13500	96325	11.63	0.000012	0.000861
2_60	PFG/50K/100meso	4	2.02	13500	96327	31.79	0.000032	0.002355
3_60	PFG/50K/100meso	6	3.03	13500	96324	89.44	0.000089	0.006625
4_60	PFG/50K/100meso	10	5.05	13500	96321	115.48	0.000115	0.008554
5_60	PFG/50K/100meso	20	10.09	13500	96314	367.24	0.000367	0.027203
6_60	50K-100meso	6	3.03	9900	96346	75.31	0.000075	0.007607
				145-min				
1_145	PFG/50K/100meso	2	1.01	25500	96349	7.63	0.000008	0.000299
2_145	PFG/50K/100meso	4	2.02	25500	96332	16.39	0.000016	0.000643
3_145	PFG/50K/100meso	6	3.03	25500	96339	41.65	0.000042	0.001633
4_145	PFG/50K/100meso	10	5.05	25500	96342	62.09	0.000062	0.002435
5_145	PFG/50K/100meso	20	10.09	25500	96347	252	0.000252	0.009882
6_145	50K-100meso	6	3.03	18700	96345	45.06	0.000045	0.002410
				180-min				
1_180	PFG/50K/100meso	2	1.01	10500	96337	3.49	0.000003	0.000332
2_180	PFG/50K/100meso	4	2.02	10500	96340	5.77	0.000006	0.000550
3_180	PFG/50K/100meso	6	3.03	10500	96322	12.61	0.000013	0.001201
4_180	PFG/50K/100meso	10	5.05	10500	96338	21.06	0.000021	0.002006
5_180	PFG/50K/100meso	20	10.09	10500	96336	96.69	0.000097	0.009209
6_180	50K-100meso	6	3.03	7700	96341	19.17	0.000019	0.002490
				240-min				
3_240	PFG/50K/100meso	6	3.03	18000	96350	8.56	0.000009	0.000476
4_240	PFG/50K/100meso	10	5.05	18000	96354	10.45	0.000010	0.000581
5_240	PFG/50K/100meso	20	10.09	18000	96351	12.94	0.000013	0.000719

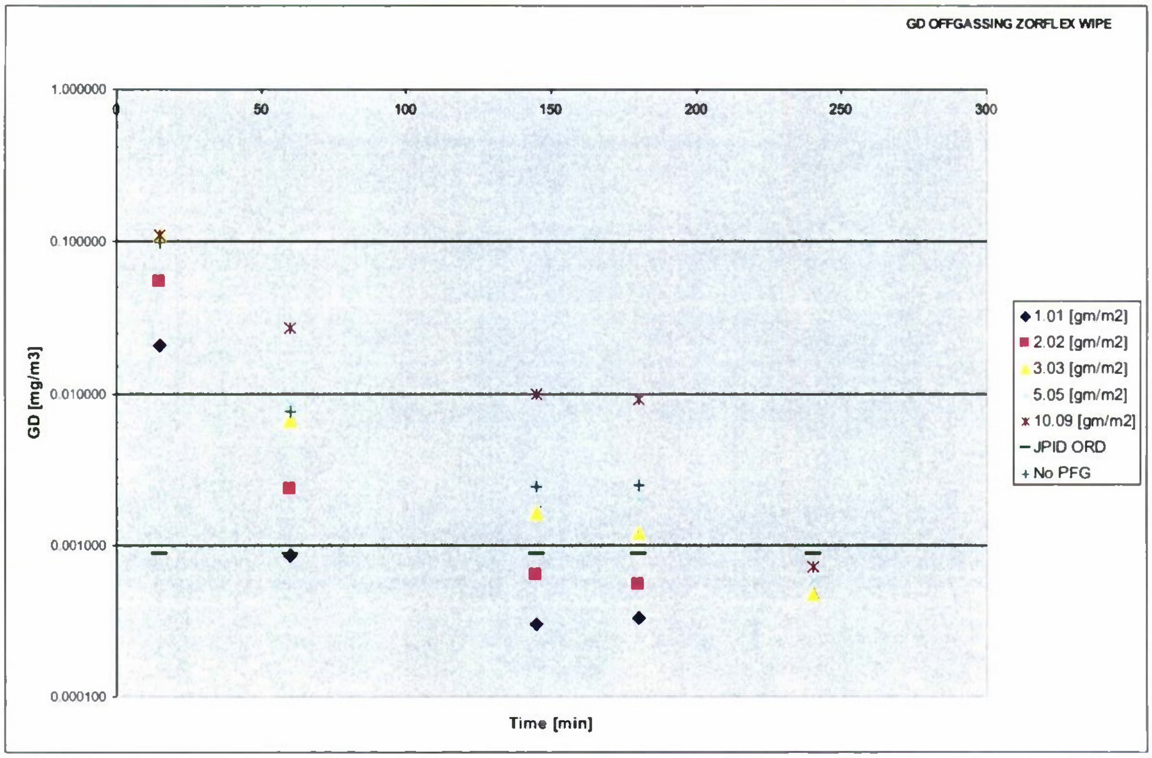
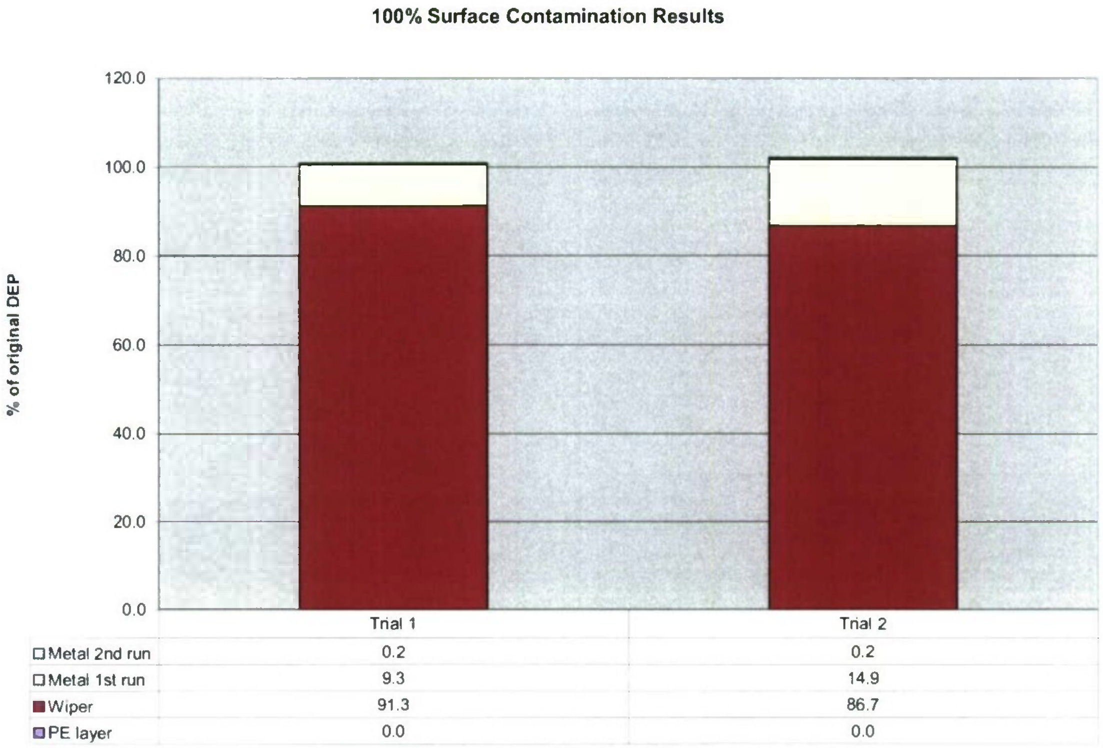


Figure 43. Semi-log plot of GD off-gassing from spent wipe.

The approximate surface area of the ACF fabric mitt breadboard shown in Figure 43 is 0.0486 m^2. The key to the right of the graph shows the initial contamination density in g/m^2. Using the data represented from Figure 43, the approximate surface area measurement of the breadboard mitt, and the surface area of the test coupon, at a starting contamination density of 1 g/m^2, a 24 m^2 would be decontaminated before the level of GD off-gassing exceeded the JPID ORD Threshold Vapor Level of 0.00087 mg/m^3.

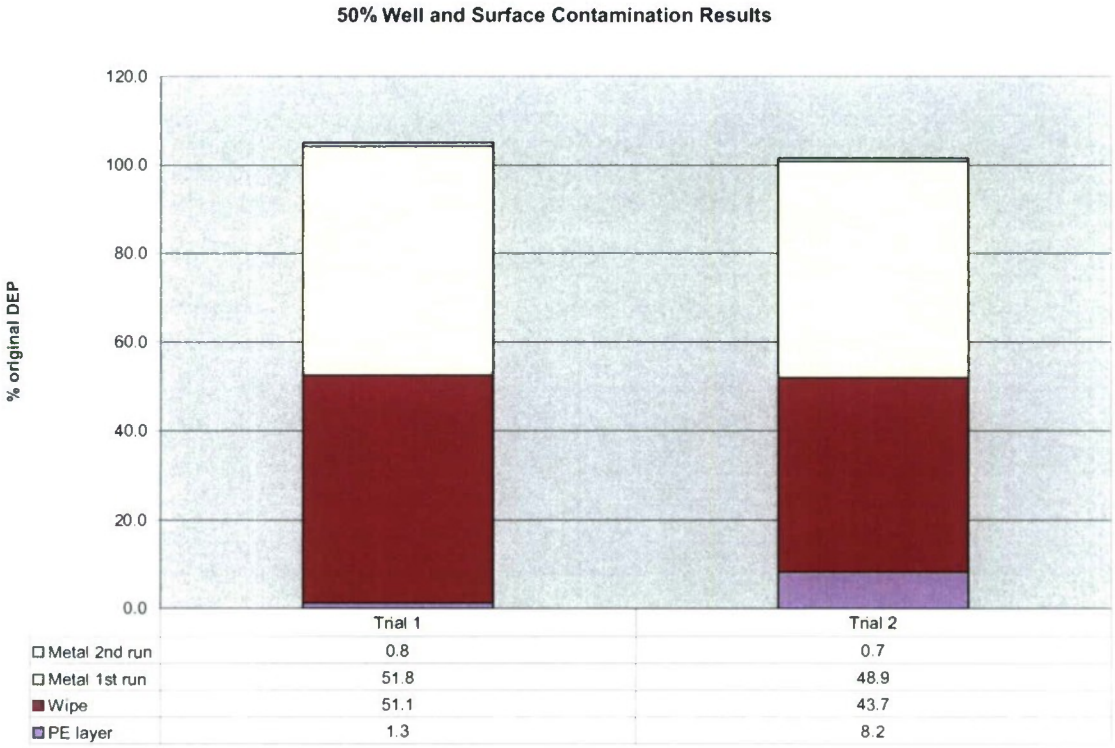
5.10 Wiping Efficacy and Complex Geometries

Results of the preliminary wiping tests are presented in Figure 44, Figure 45, and Figure 46. As can be seen from these results, wiping efficacy can range from very low to very high, depending on the initial placement of the contaminant. The wipe can clean the metal components of the keypad with ~90% efficiency when only the surface of the keypad is contaminated. In comparison, less than 5% is removed when only the wells are contaminated. These data are supported by the results of the third experiment in which 50% of the contaminant was put in the wells and 50% of the contaminant was put on the surface. In this case, the wipe was able to pick up ~50% of the contaminant from the surface. From this data it is clear that a wiper, by itself, cannot effectively clean the wells of a complex object. A separate strategy, such as spraying with pressurized HFE and wiping, must be used in order to clean these areas, as discussed in the next section.



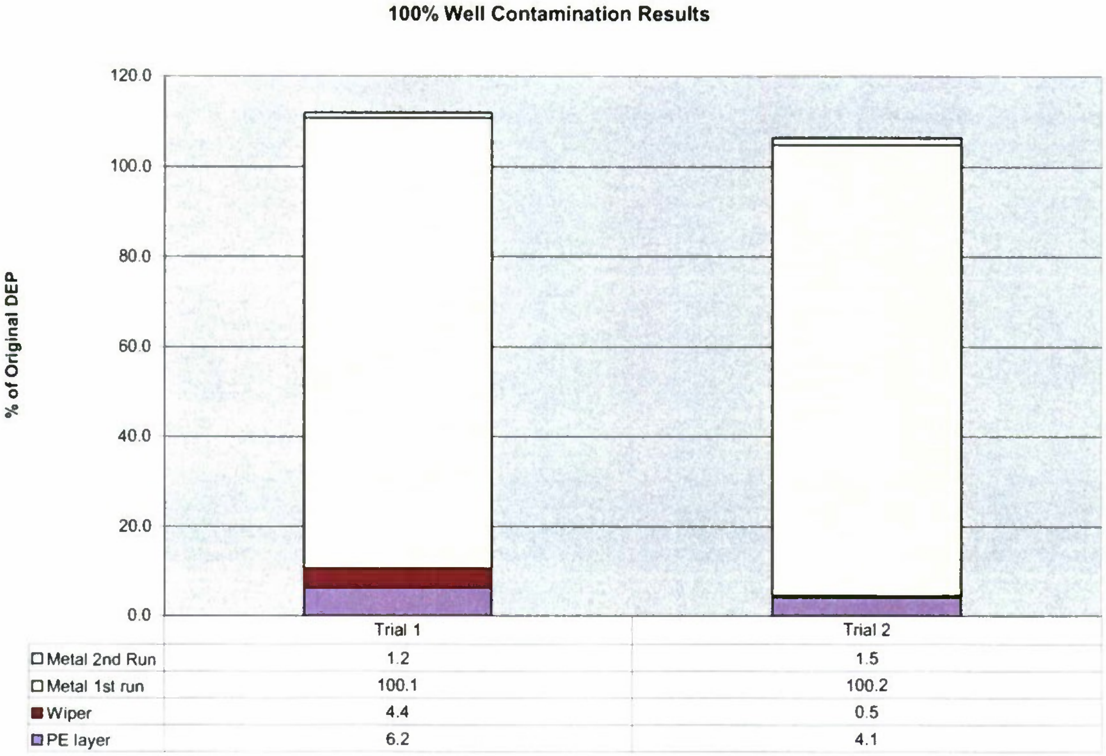
	Trial 1	Trial 2
☐ Metal 2nd run	0.2	0.2
☐ Metal 1st run	9.3	14.9
■ Wiper	91.3	86.7
■ PE layer	0.0	0.0

Figure 44. Results with all contamination applied to the surface of the keypad.



	Trial 1	Trial 2
☐ Metal 2nd run	0.8	0.7
☐ Metal 1st run	51.8	48.9
■ Wipe	51.1	43.7
■ PE layer	1.3	8.2

Figure 45. Results with 50% surface/50% well contamination of the keypad.



	Trial 1	Trial 2
☐ Metal 2nd Run	1.2	1.5
☐ Metal 1st run	100.1	100.2
■ Wiper	4.4	0.5
▨ PE layer	6.2	4.1

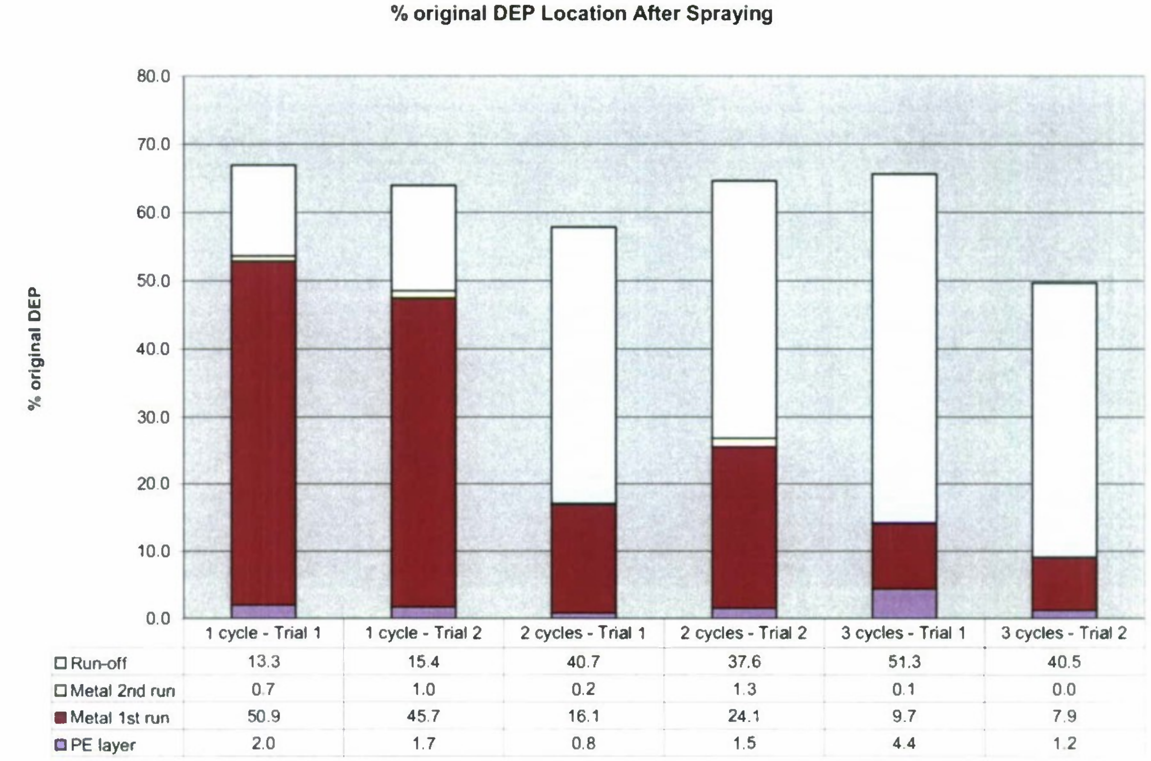
Figure 46. Results with100% well contamination of the keypad.

5.11 Spray and Wipe Tests

Results of the spray experiments are presented in Figure 47 and Figure 48, and in Table 56 to Table 57. From Figure 47 and Table 57, it is clear that decontamination efficacy increases with increased time/cycles of spraying. With only one cycle of spraying, nearly 50% of the contaminant remains on the keypad. With an increase to two cycles, this number is reduced to about 25%, and with three cycles, less than 10% of the original contaminant in the wells remains on the keypad.

Increasing the number cycles should result in reduced contaminant levels, but at a decreasing rate as shown in Figure 48. Also, focusing the spray on the edges of the keys should result in more rapid evacuation of the contaminant from the wells. Spraying appears to be a very valuable adjunct to wiping for the decontamination of a complex object, such as a keypad.

As shown in Table 57, it should be noted that between 50 and 67% of the original DEP contaminant could be collected in the end of each of the spray tests. From a preliminary validation test of an un-wiped keypad it was found that all the DEP on the keypad could be collected using the methods described above. This suggests that the DEP loss was more likely during the collection of the run-off. By simply pouring the run-off out of the baking dish into a jar, a significant amount of DEP could have stuck to the dish and not been collected.



	1 cycle - Trial 1	1 cycle - Trial 2	2 cycles - Trial 1	2 cycles - Trial 2	3 cycles - Trial 1	3 cycles - Trial 2
□ Run-off	13.3	15.4	40.7	37.6	51.3	40.5
□ Metal 2nd run	0.7	1.0	0.2	1.3	0.1	0.0
■ Metal 1st run	50.9	45.7	16.1	24.1	9.7	7.9
▨ PE layer	2.0	1.7	0.8	1.5	4.4	1.2
Total of Above	66.9	63.9	57.8	64.6	65.5	49.6

Figure 47. Location of recovered DEP after spray tests.

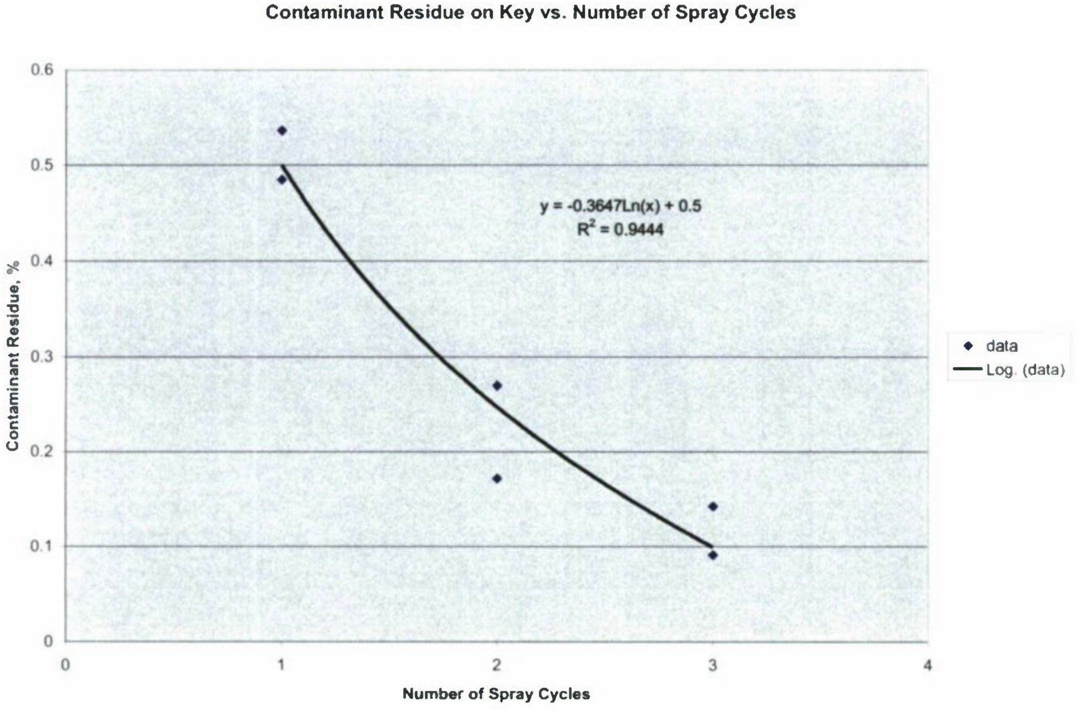


Figure 48. Fraction original DEP left on keypad vs. spray cycles.

Table 56. Analytical data from spraying tests.

GC calibration data:			130ppm DEP in chloroform = 266000 area units				
			300ppm DEP in HFE 7100 = 981000 area units				
Spraying one time over the keypad							
Trial 1							
#	name	area	time		ppm DEP	□L DEP	% DEP
25	JS 7PE	40795	6,024		19.94	1.00	1.99
26	JS 7M1	226266	6.021		110.58	25.43	50.87
27	JS 7M2	3084	6.054		1.51	0.35	0.69
28	JS 7D	4363388	6.086		1334.37	6.67	13.34
						Total:	66.90
Trial 2							
#	name	area	time		ppm DEP	□L DEP	% DEP
29	JS 8PE	35301	6.029		17.25	0.86	1.73
30	JS 8M1	203144	6.021		99.28	22.83	45.67
31	JS 8M2	4628	6.063		2.26	0.52	1.04
32	JS 8D	2102399	6.071		642.94	7.72	15.43
						Total:	63.87
Spraying two times over the keypad							
Trial 1							
#	name	area	time		ppm DEP	□L DEP	% DEP
17	JS 5PE	16546	6.045		8.09	0.40	0.81
18	JS 5M1	71829	6.025		35.10	8.07	16.15
19	JS 5M2	711	6.093		0.35	0.08	0.16
20	JS 5D	875132	6.047		267.62	20.34	40.68
						Total:	57.80
Trial 2							
#	name	area	time		ppm DEP	□L DEP	% DEP
21	JS 6PE	31598	6.032		15.44	0.77	1.54
22	JS 6M1	107178	6.021		52.38	12.05	24.09
23	JS 6M2	5708	6.073		2.79	0.64	1.28
24	JS 6D	854664	6.045		261.37	18.82	37.64
						Total:	64.56
Spraying three times over the keypad							
Trial 1							
#	name	area	time		ppm DEP	□L DEP	% DEP
33	JS 9PE	89705	6.021		43.84	2.19	4.38
34	JS 9M1	43224	6.02		21.12	4.86	9.72
35	JS 9M2	400	6.081		0.20	0.04	0.09
36	JS 9D	723752	6.041		221.33	25.67	51.35
						Total:	65.54
Trial 2							
#	name	area	time		ppm DEP	□L DEP	% DEP
37	JS 10PE	25093	6.032		12.26	0.61	1.23
38	JS 10M1	34925	6.028		17.07	3.93	7.85
39	JS 10M2	0	0		0.00	0.00	0.00
40	JS 10D	988917	6.047		302.42	20.26	40.52
						Total:	49.60

Table 57. Fate and material balance of DEP and HFE 7100 consumption.

Total % of original DEP left on keypad:		
# of cycles	Trial 1	Trial 2
1	53.55	48.43
2	17.12	26.92
3	14.19	9.08
Mass balances for trials:		
# of cycles	Trial 1	Trial 2
1	66.90	63.87
2	57.80	64.56
3	65.54	49.60
Amount of HFE 7100 used in each trial:		
# of cycles	Trial 1	Trial 2
1	13.01g	13.00g
2	34.17g	31.62g
3	47.19g	44.02g
Volume of HFE 7100 gathered after spraying:		
# of cycles	Trial 1	Trial 2
1	0.75g	1.8g
2	11.4g	10.8g
3	17.4g	10.05g

5.12 Effect of Multiple Wipe Cycles

In each test the data are presented as the percent of the recovered DEP found on each of the stainless steel sheets and in each layer. Three trials were performed for each experiment. The data presented in Table 58 show the average and standard deviation of these trials.

Table 58. Three wipe test results.

	Average					Standard Deviation				
Wipe Cycle #	1st Sheet	2nd Sheet	3rd Sheet	Laminate	ACF #2	1st Sheet	2nd Sheet	3rd Sheet	Laminate	ACF #2
1				58.07	31.49				5.04	6.62
2				8.92	0.99				1.44	0.08
3	0.01	0.00	0.00	0.45	0.06	0.00	0.01	0.00	0.16	0.01

After three wiping cycles, the results show that all three sheets were decontaminated to <0.01% of the original contaminant load. The results do not show the amount of DEP on the plates after two wipes; however, the amount of DEP removed during each wipe cycle can be examined more closely by examining the residual DEP on the wiper. After the first wipe cycle, 58 and 31.5% of the DEP load was found in the laminate and 2nd ACF layer, respectively. For the second wiper used, 9 and 1% of the initial DEP load was found in the laminate and 2nd ACF layer, respectively. For the third wiper, these numbers dropped to 0.45 and 0.06%.

Additionally, the previous wiping tests demonstrated that nearly identical results were obtained with PFG 39278 and PFG 66387 faced wipes. Thus, it can be presumed that the results that would be obtained with PFG 663987 faced wipes results would be similar to those shown in Table 58.

5.13 Removal of Other Contaminants with Motor Oil Wiping Tests

Results for the motor oils are shown in Table 59 and Table 60. In each test the data are presented as the percent of the recovered contaminant found in each layer. Three trials were performed for each experiment and the data given were an average of these trials along with the standard deviation.

From Table 59 it was clear that the wipers removed over 98% of the motor oils, less than <2% of the original oil remained on the wiped surface in all cases. An additional test was done for motor oil SAE 40, in which three wipe cycles were completed, instead of just two. The result of this test indicated that the amount of oil left on the surface was reduced by wiping a third time; however, the reduction was very small. This could probably be explained by the fact that all three motor oils were insoluble in HFE 7200, the transfer solvent used in all cases. Due to the insolubility, a certain amount of the oil became extremely difficult to pick up using the decontamination wipe.

Table 59. Motor oil wiping test results.

		Average						
Test #	**Motor Oil**	**1st Sheet**	**2nd Sheet**	**3rd Sheet**	**LAM #1**	**ACF #1**	**LAM #2**	**ACF #2**
1	SAE 40	0.88	0.73	0.65	79.03	15.58	2.58	0.55
2	SAE 10W-30	1.04	1.07	1.61	76.69	15.38	3.59	0.62
3	SAE 80W-85W-90	0.74	0.90	1.22	73.47	20.79	2.00	0.87
Test #	**Motor Oil**	**Standard Deviation**						
1	SAE 40	0.14	0.18	0.07	3.60	3.89	0.64	0.18
2	SAE 10W-30	0.18	0.04	0.22	1.16	1.04	0.46	0.17
3	SAE 80W-85W-90	0.46	0.45	0.37	3.00	2.55	0.12	0.05

The mass balances are shown in Table 60. All the mass balances close to +/- 10% of the average.

Table 60. Mass balances.

DEP Recovered (% of Initial Load)					
Test #	**Trial 1**	**Trial 2**	**Trial 3**	**Average**	**Standard Deviation**
1	110.41	96.91	93.24	100.19	7.38
2	101.8	99.64	99.33	100.26	1.1
3	99.95	106.37	110.51	105.61	4.35

Overall, the motor oil wiping tests showed that more than 98% of the contaminant was removed when wiping a contaminant that is insoluble in HFE 7200 and more viscous than DEP,

5.14 Removal of Other Contaminants

5.14.1 Heavy Liquids

The results for the additional wiping tests are shown in Table 61 and Table 62.

In each test the data is presented as the percent of the recovered contaminant found in each layer. For example, "LAM #1" is the % of the initial load found in the laminate part of the first wiper used, and "ACF #3" is the percentage of the initial load found in the 50K layer for the third wiper used. Three trials were performed for each experiment, and the data given are an average of these trials along with the standard deviation.

Table 61. Additional wiping test results.

		Average								
Test #	**Contaminant**	**1st Sheet**	**2nd Sheet**	**3rd Sheet**	**LAM #1**	**ACF #1**	**LAM #2**	**ACF #2**	**LAM #3**	**ACF #3**
1	Citroflex	0.14	0.25	2.81	61.13	20.04	14.12	1.51	3.15	0.16
2	PDMS 10	0.01	0.46	1.87	57.41	26.38	11.63	2.25	2.84	0.39
3	Krytox	0.00	0.51	1.87	53.43	27.13	14.26	2.8	3.95	0.03
Test #	**Contaminant**	**Standard Deviation**								
1	Citroflex	0.10	0.18	0.65	1.74	1.5	0.69	0.05	0.17	0.03
2	PDMS 10	0.01	0.11	0.07	1.45	2.83	1.59	0.37	0.67	0.02
3	Krytox	0.00	0.65	0.36	1.38	2.1	0.93	0.47	0.18	0.02

As shown in Table 61, the results for the Citroflex, PDMS 10cs, and Krytox oil were similar to the motor oil results because in general, 98% or more of the contaminant was removed from each plate. It should be noted that the removal of these contaminants from the first two sheets was well above 99% and above 97% for the third sheet.

Unlike motor oil, all three of these contaminants were fairly soluble in HFE 7200. This fact was reflected in the more effective transfer of the contaminants into the ACF layers of the wipes. This also suggests the possibility that an additional wiping would produce better results than the results produced with the motor oils utilizing an additional wipe cycle.

Table 62. Mass balances.

DEP Recovered (% of Initial Load)					
Test #	**Trial 1**	**Trial 2**	**Trial 3**	**Average**	**Standard Deviation**
1	100.20	94.38	100.41	98.33	2.80
2	97.72	96.04	98.52	97.42	1.03
3	91.19	93.84	95.80	93.61	1.89

All mass balances were within +/- 10% of the average.

5.14.2 Barrierta L55/2 Grease-Wiping Tests

Data for the grease-wiping test is shown in Table 63. In each test, the data is presented as the percent of the recovered contaminant found on each metal sheet. The data for the three replicate tests that were performed are shown along with the average standard deviation.

Table 63. Grease-wiping results.

	1st Sheet	2nd Sheet	3rd Sheet
Replicate #1	0.24	0.62	0.91
Replicate #2	0.00	0.67	1.10
Replicate #3	0.24	0.43	0.67
Average	0.16	0.57	0.89
Standard Deviation	0.11	0.10	0.18

From Table 63 it was again clear that the decontamination wipes effectively removed a significant amount of the original contaminant from the metal sheets. In all trials, after three wipes, >99% of the original contaminant load was removed from the first two metal sheets, and more than 98% was removed from the third sheets. It should be noted, although the contaminant was removed from the metal sheets, subsequent extraction of the contaminant from the decontamination wipe proved to be difficult. Even when using HFE 7200, the available solvent that most readily dissolved the grease, it was not possible to extract enough grease from the wipe to perform a mass balance.

5.14.3 Fruit Tree Spray

Table 64 shows the amount of contaminant removed from each plate per wash.

Table 64. Wiping efficacy of pesticide removal.

Plate Recovery						
Sample	Wiped	Wash	Total Volume of Contaminant in wash (µL)	Volume of Contaminant in wash (µL)	PPM	Volume of Sample (mL)
Plate 1	Immediately	1	0.63	0.59	53	11.07
		2		0.04	4	10.76
		3		0.00	1	9.33
Plate 2	After 1 H	1	0.64	0.52	50	10.30
		2		0.07	7	15.56
		3		0.05	5	9.60
Plate 3	Not Wiped	1	185.78	183.60	158000	11.62
		2		0.95	86	11.08
		3		1.23	101	12.15

Table 64 indicates that most of the contaminant originally on Plates 1 and 2 was removed from the plates during wiping. Using the total volume recovered from Plate 3 as a basis for the amount of contaminant initially present on Plates 1 and 2, the wiping efficacy was 99.66% for both of these plates. The measured volume of contaminant on Plate 3 was within 5% of the expected value of 174 μL.

5.15 Ballooning of Storage Bags Containing HFE Wetted ACF Fabric Wipes at 71 °C

The results for the fixed-volume cell tests are shown below in Figure 49 through Figure 52. Figure 49 and Figure 50 show the results for cells that contained only HFE, and Figure 51 and Figure 52 show the results for cells that contained ACF and HFE.

Figure 49 shows that all four test cells, the three cells containing degassed HFE 7200 and the cell containing AR HFE 7200,all maintained a relatively steady pressure close to that of the vapor pressure of HFE 7200, plus the pressure due to the thermal expansion of air, for nine and seven days, respectively. One test cell full of degassed 7200, represented by the teal colored data series in Figure 49, slowly lost pressure over time due to a leak.

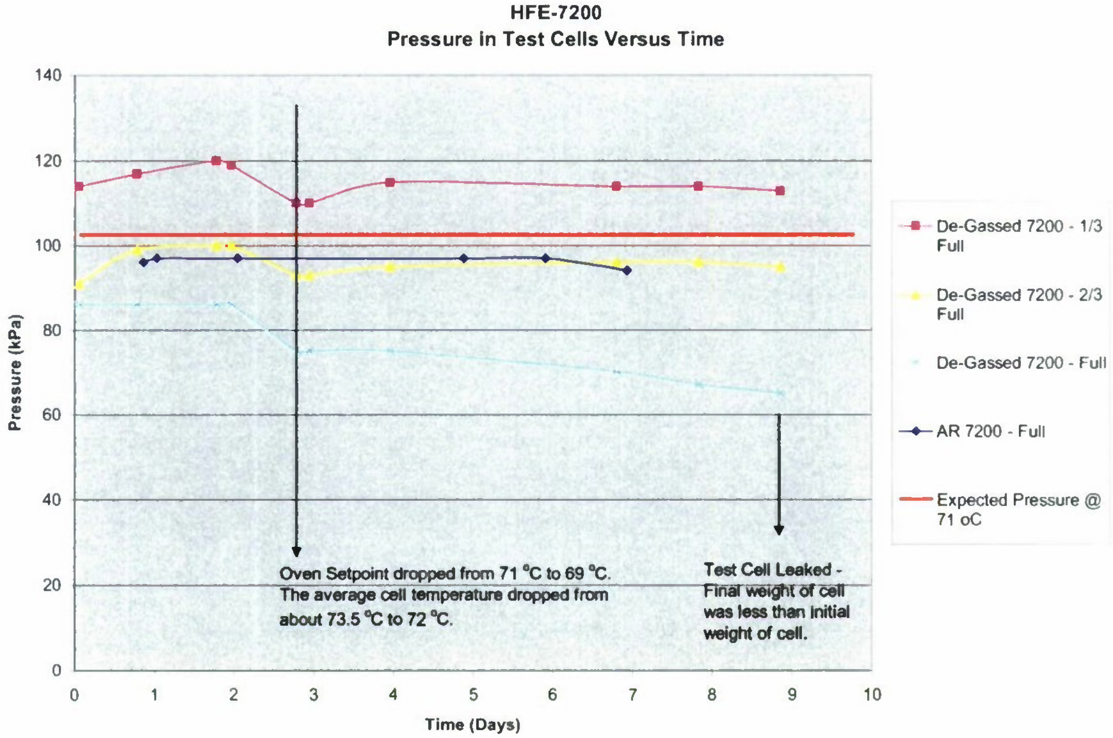


Figure 49. HFE 7200 fixed volume cell tests.

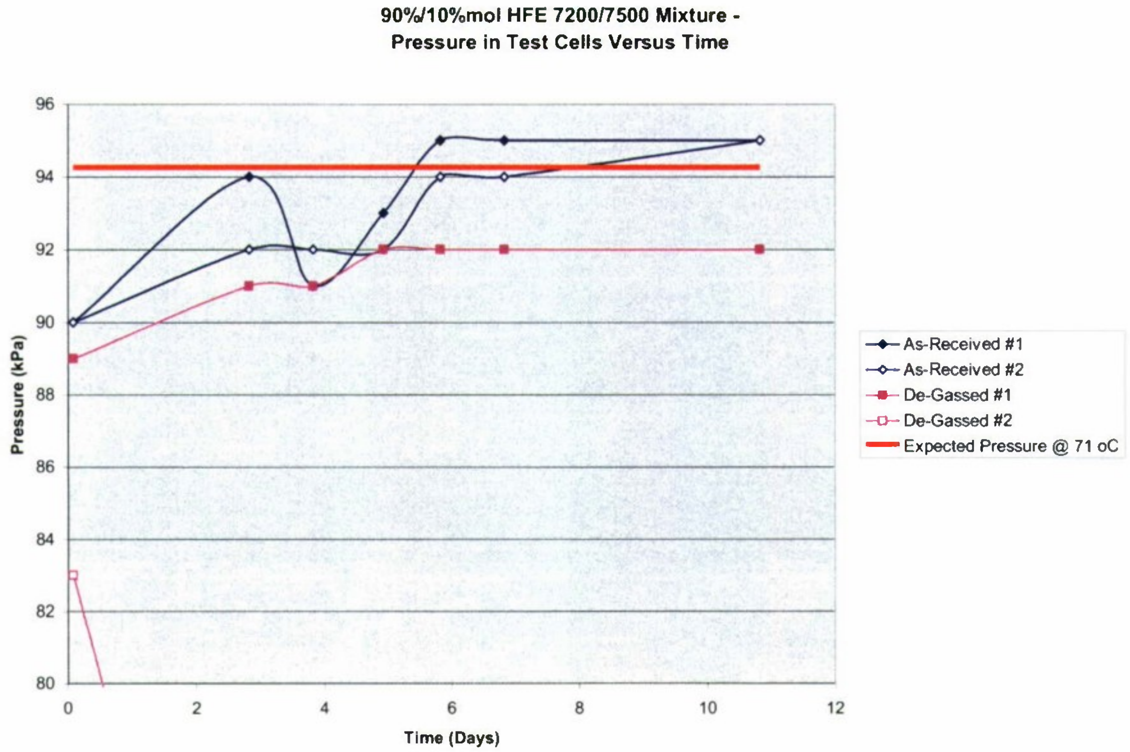


Figure 50. HFE 7200/7500 mixture fixed volume cell tests.

A similar fixed-volume cell test was performed in duplicate for both as-received and degassed 90/10 mol% mixture of HFE 7200/7500. The results of these tests are shown in Figure 50. With the exception of second degassed replicate, all test cells maintained a steady pressure close to that of the vapor pressure of HFE 7200, plus the pressure due to the thermal expansion of air, for the entire test period.

The results of these tests demonstrate that when packaged alone, HFEs or a mixture thereof, either de-gassed by boiling or simply AR, should not exert a pressure greater than that of their vapor pressure plus that due to the thermal expansion of air. The conclusion was that the HFEs are stable under these conditions.

In comparison to the fixed-volume cell tests in which only HFE was in the cells, and where the pressure was constant for entire test period, the pressure in the cells containing ACF and HFE steadily rose well above the vapor pressure of HFE plus the pressure due to the thermal expansion of air, as shown below in Figure 51 and Figure 52.

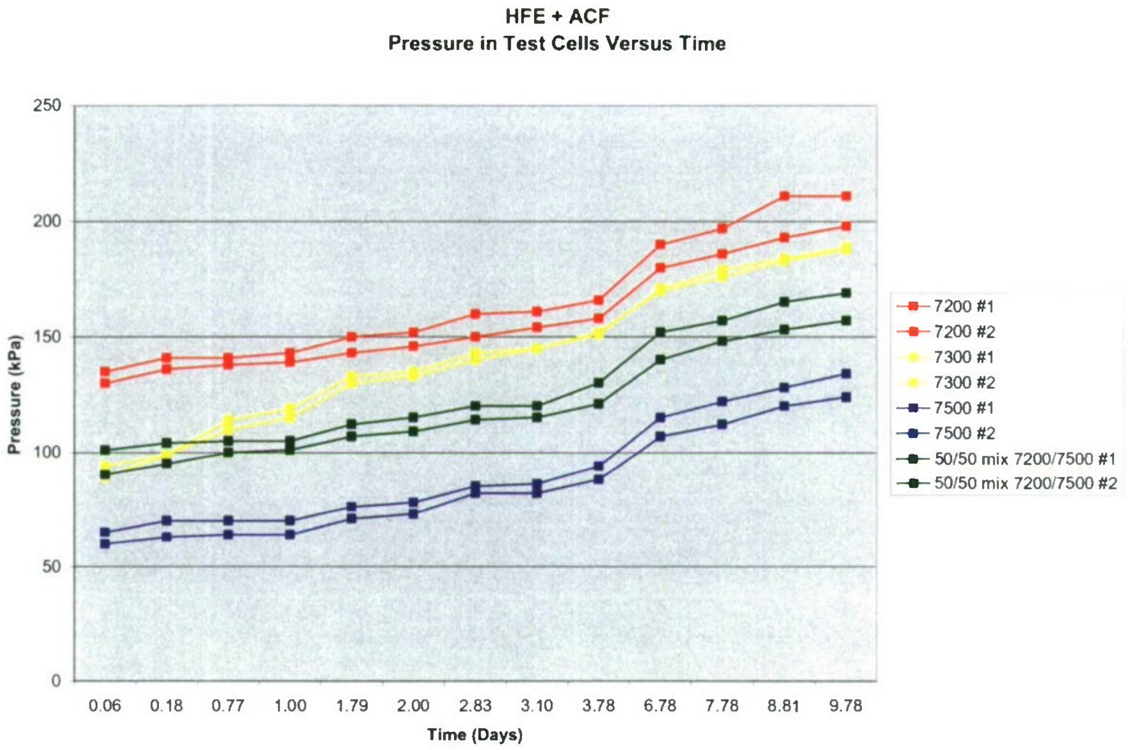


Figure 51. HFE + ACF fixed volume cell tests.

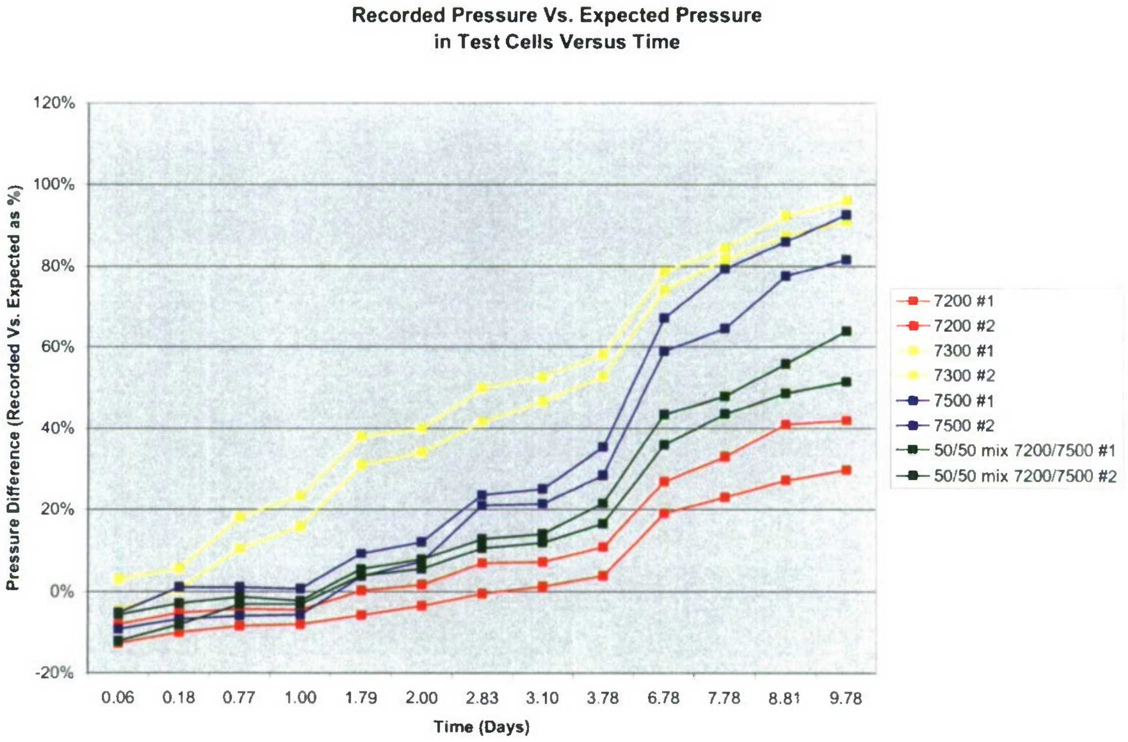


Figure 52. HFE + ACF deviation from expected pressure.

The results in Figure 52 clearly show that the pressure in all eight cells:

- Rose steadily over the ten day time period in which they were in the oven.
- Was continuing to rise when they were removed from the oven.
- Was greater than 101 kPa (1 atm) in all cases.

Cells containing HFE 7200 had the highest pressure, followed by HFE 7300, the 50/50 mix of HFE 7200 and HFE 7500, and lastly HFE 7500. This result was not entirely surprising given that the vapor pressure of HFE 7200 was greater than the vapor pressure of HFE 7300, which was greater than the vapor pressure of HFE 7500. What was surprising was that the total pressure level accumulated in the cells was significantly higher than the expected pressure, based on the temperatures of the cells as shown in Figure 52. For example, the combined vapor pressure of HFE 7500 and water, plus the thermal expansion of air at 71 °C, is 63.4 kPa. (In this example, 16.3 kPa is contributed by the HFE 7500, 33.4 kPa is contributed by the water, and 13.7 kPa is due to the expansion of air). However, the cells containing HFE 7500 and ACF reached pressures in excess of 134 kPa and were still rising after ten days in the oven. This pressure was much greater than the 101 kPa required to cause a pouch to expand, and was 90% greater than the expected pressure.

The odd-numbered test cells were sent to 3M so that the contents of the cells could be analyzed by GC/MS. The results of this analysis are summarized in Table 65. The results indicated that, for all HFEs, a small, but significant amount of the HFE was converted to lower molecular weight gaseous species. The last column of the table is an estimate of the volume that these gaseous species would occupy. In general, between 13 and 65 mL of gas was generated within the test cells. This was a gas volume capable of generating a significant pressure increase within a 40 mL fixed volume cell, which only had approximately 3 mL of free space. It was interesting to note that the branched ethers, HFE 7300 and HFE 7500, were more easily broken down and converted to a gaseous species than the linear HFE 7200.

Table 65. GC/MS results.

Cell #	Contents	Initial HFE Content (Mol %)	Final HFE Content (Mol %)	Final Gaseous Species Content (Mol %)	Volume of Gaseous Species (mL)
1	50K + 7200	99.71	98.11	0.27	12.7
3	50K + 7300	99.91	96.95	1.85	65.0
5	50K + 7500	99.69	98.15	0.70	20.3
7	50K + 72/75 Mixture	99.70	96.94	0.53	19.0

This increase in pressure was attributed to the presence of alkaline metal oxide nanoparticles on the surface of the activated carbon fibers (as was evident from the photomicrographs presented as Figure 53), which promote the degradation of the HFEs at elevated temperature. As previously discussed, these same particles are believed to be responsible for the *in-situ* degradation of adsorbed agent with time.

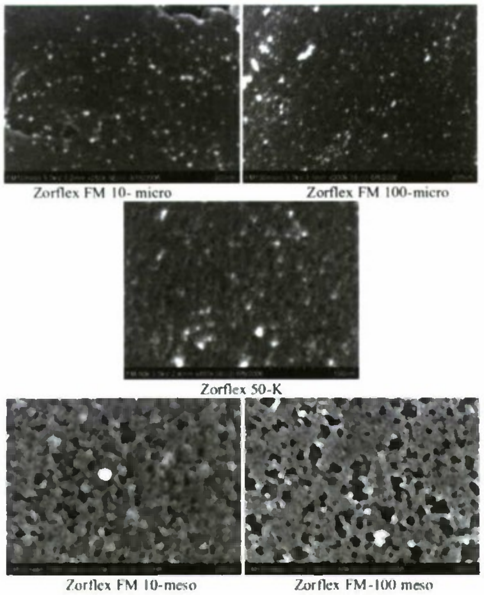

Zorflex FM 10- micro
Zorflex FM 100-micro
Zorflex 50-K
Zorflex FM 10-meso
Zorflex FM-100 meso

Figure 53. Photomicrographs of Zorflex ACF Fabrics (180K to 450K magnification).

The results of these tests (Table 66) clearly demonstrate that the ACF mitt and the HFE solvent must be segregated when packaged for the system to meet military storage requirements.

Table 66. Data from preliminary mass balance tests.

GC calibration data:				130ppm DEP in chloroform = 266000 area units			
Trial 1:							
#	name	area	time		ppm DEP	mL DEP	% DEP
1	JS PE	15935	6.067		7.79	0.00	0.43
2	JS M1	805333	6.046		393.58	0.09	99.59
3	JS M2	9552	6.029		4.67	0.00	1.18
						Total:	101.20
Trial 2:							
#	name	area	time		ppm DEP	mL DEP	% DEP
4	JS 2PE	0	0		0.00	0.00	0.00
5	JS 2M1	837252	6.048		409.18	0.09	103.53
6	JS 2M2	9150	6.083		4.47	0.00	1.13
						Total:	104.67
Trial 3:							
#	name	area	time		ppm DEP	mL DEP	% DEP
7	JS 3PE	147180	6.024		71.93	0.00	3.96
8	JS 3M1	752082	6.045		367.56	0.08	93.00
9	JS 3M2	9577	6.028		4.68	0.00	1.18
						Total:	98.14
						Average Recovered Material:	101.33

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ACRONYMS

ACF	Area Cost Factor/Activated Carbon Fiber
AoA	Analysis of Alternative
BAM	Business Area Manager
CA	Chemical Agent
CAM	Commodity Area Manager
CARC	Chemical Agent Resistant Coating
CFF	Candidate Facing Fabric
CFU	Colony Forming Unit
COTS	Commercial Off the Shelf
DEP	Fluorescent diethyl phthalate
ECBC	U.S. Army Edgewood Chemical Biological Center
ESI	Entropic Systems Inc.
FID	Flame Ionization Detector
FPD	Flame Photometric Detector
GC	Gas Chromatography
GD	Soman, non-persistent agent
HD	Distilled mustard agent
HFE	hydrofluoroether
JMDS	Joint Material Decontamination System
JPEO-CBD JPM	Joint Program Executive Office for Chemical Biological Defense Joint Program Manager
JPID	Joint Platform Interior Decontamination
JSSED	Joint Service Sensitive Equipment Decontamination
JSTO	Joint Science and Technology Office
MINICAMS	Miniature Continuous Air-Monitoring System
RH	Relative Humidity
SNAP	Significant New Alternatives Policy
TEU	Tech Escort Unit
TGD	Thickened GD
TIC	Toxic Industrial Chemical
TOP	Test Operating Procedure
TTA	Technology Transition Agreement
TWA	Time-Weighted Average
UHMW	Ultra-High Molecular Weight
VX	Methylphophonothioic acid, persistent nerve agent

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