PPE Cleaning Validation

Verification of Cleaning, Decontamination, and Sanitization of Fire Fighter Garments

SUPPLEMENT B: Preliminary Work for Assessing PPE Cleaning Procedures

Jason Allen
Intertek Testing Services
Cortland, New York

December 2018
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Final Report by:

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December 2018

(Part 3 of 9)
Firefighter exposure to personal protective equipment (PPE) that is dirty, soiled, and contaminated is an increasing concern for long-term firefighter health. Cancer and other diseases resulting from chronic exposures has become a leading issue and is presumed to be associated with fireground exposures relating to protection/hygiene practices and persistent harmful contamination found in firefighter PPE.

While general cleaning procedures have been established in NFPA 1851, Standard on Selection, Care, and Maintenance of Protective Ensembles for Structural Fire Fighting and Proximity Fire Fighting, there are no requirements that demonstrate whether current cleaning practices will adequately remove contaminants from firefighter PPE. Many manufacturer gear cleaning recommendations are vague and most cleaning product/process claims are unsubstantiated regarding contaminant removal effectiveness. Prior studies have identified persistent chemical and biological contaminants in structural firefighting PPE. Therefore, industry methodologies and practices are needed that can promote safe cleaning techniques so that firefighters are not continually exposed to unclean or inadequately cleaned gear. It also important to set cleanliness criteria for the continued use of firefighter protective clothing.

This project has established a relevant and credible procedure to validate “how clean is clean?” for fire service contaminated gear, and in doing so has addressed the primary goal of reducing firefighter exposure to harmful contaminants in PPE. This includes the establishment of a repeatable and reproducible standardized method that can be used to determine the decontamination effectiveness of cleaning methods, and establish the needed fire service guidance for maintaining contaminant-free PPE as well as show that cleaning processes do not damage clothing. The project deliverables directly support efforts to update NFPA 1851 and other information that ensures consistent, effective cleaning processes of fire service gear.

This report is part three of a nine-part series on this topic of “PPE Cleaning Validation”, with this part titled “Supplement B: Preliminary Work for Assessing PPE Cleaning Procedures”. The following are all the reports in this series:

1. Master Report
2. Supplement A: Annotated Bibliography
3. Supplement B: Preliminary Work for Assessing PPE Cleaning Procedures
4. Supplement C: Investigation of Simulated Fire Ground Exposures
5. Supplement D: Evaluation of Outer Shell Liquid Retention Properties
7. Supplement F: Report of Heavy Metals Contamination, Extraction, and Analysis Procedures
9. Supplement H: Evaluation of Microbial Cleanliness of Selected ISP Advanced Cleaning Procedures
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<table>
<thead>
<tr>
<th>Fire-Dex</th>
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</tr>
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<tbody>
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<td>Globe Manufacturing</td>
<td>PBI Performance Products</td>
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<tr>
<td>Lion First Responder Products</td>
<td>TenCate Protective Fabrics</td>
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The leading information and knowledge resource on fire, electrical and related hazards

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Executive Summary

A project was undertaken by Intertek for the Fire Protection Research Foundation as a preliminary effort to investigate the effectiveness of laundering for removing chemical contaminants from firefighter protective clothing. The project involved several parts that included an analysis of representative sets of contaminated clothing and the development of a prospective procedure for realistically and repeatably contaminating protective clothing. The latter part of the project was pursued as a means for creating contaminated clothing samples that could then be subjected to given laundering procedures and then evaluated for residual contamination as a means for assessing decontamination efficiency.

Intertek was provided a number of contaminated clothing items from the NFPA Task Group on Firefighter Clothing Cleaning that had been collected from various departments. Selected items of clothing were selected and samples were taken for analysis of contaminants. The identification of chemical contaminants involved a unique piece of analytical equipment that permitted simultaneous GC-MS and thermographic analysis. This testing showed a variety of contaminants that included some chemicals that were not typical fireground contaminants such as benzophenone, terephthalate, 1-phenoxypopropan-2-ol, and p-cresidine. One of the principal findings from this work was the difficult in consistently extracting samples for recovering contaminants and discriminating among contaminants that are present at very low levels.

A principal portion of the research effort involved the development of a technique whereby contaminants could be deposited on a protective clothing sample for later analysis of the sample both before and after cleaning. Initial efforts by Intertek involved using a special tube furnace with a set burning fuel to push smoke across a sample to simulate how a fabric would be contaminated in the field. After conducting several experiments to optimize the heating conditions using a PVC-based fuel, samples were obtained that showed soot deposition. However, this approach proved difficult in getting uniform and detectable forms of relevant contamination.

Intertek then compared a more conventional way of doping samples with contaminants and added soot by grinding the soot onto the fabric. The placement of soot along with a finite amount of liquid chemicals was thought to be a hybrid approach that accounted for both ordinary soiling by soot deposition but also combined a more controllable way of putting chemicals onto the fabric. Several experiments were carried out using these techniques and the contaminated samples were laundered using the NFPA 1971 procedures for proving a general assessment of cleaning effectiveness. By using a range of representative fireground chemicals, Intertek was able to show different levels of chemical decontamination of the samples used in this fashion, though it was concluded that remaining soot on the samples was an indication of either excess soot in the contamination process or an incomplete cleaning process.

Overall, the work in this project showed that contamination of firefighter clothing can be complex and the determination of specific contaminants requires special attention to the selected extraction and analytical procedures. It was further demonstrated that while a smoke deposition contamination technique might have merit, this technique provided inconsistent results, which can be difficult to analyze. Nevertheless, some success was obtained when conventional chemical doping was combined with a mechanical means of applying soot-like substances to approximate fireground exposure conditions. The use of a soot surrogate was found to be important in affecting decontamination efficiency. Based on this work, it was possible to recommend preliminary test procedures for qualifying cleaning effectiveness.

Contaminated Gear Analysis

Gear Selection

A total of 81 samples of firefighter protective coats and pants were received via the NFPA Task Group on Firefighter Clothing Cleaning. This gear was provided from New York City, NY; Philadelphia, PA; Oxnard, CA; Irvine, CA; Chicago, IL; and Carlsbad, CA. Upon receipt, all samples were photographed as seen in the examples provided in Figures 1 and 2 below and specific observations were made regarding the areas of significant visible contamination.

Due to an uncertain exposure history, this gear was placed aside for quick, rough qualitative analysis and validation of sampling techniques. Additionally, sets of gear were provided directly to Intertek by Boston, MA, and Columbus, OH. This gear was selected specifically for this project immediately post fire, prepared within non-off gassing PTFE containers and shipped to Intertek with a brief incident report in an attempt to frame possible exposure contaminants versus those found via chemical analysis.
Samples were removed from garments and each individual layer was placed within glass vials with lids securely installed. Samples were then extracted utilizing a Soxhlet extraction method.

Initial Chemical Analysis

In order to analyze the turnout gear, a new instrument technology was used. This instrument technology was a combined analytical system that includes a thermogravimetric analyzer (TGA), a Fourier Transform-infrared (FT-IR) spectrometer instrument, and gas chromatograph coupled with a mass spectrometer (GC/MS) (Figure 3). This system allows three instruments to be run in tandem, collecting data from all of the instruments simultaneously.

The instrument was used running only the TGA and GC/MS modes in parallel. This configuration was selected to utilize the heat control of the TGA to produce a precisely controlled off gassing of the test samples. The off gassing products from the TGA were then passed though the GC/MS for characterization of the constituents of the off gassed material.

Initial setup was configured such that the temperature on the TGA was ramped to 800°C. During this method there were peaks that showed a strong resemblance to benzene and toluene. It is thought that these peaks were due to degradation of the base material.

Additional samples were run based upon a starting temperature of 100°C which was held for 30 seconds. Following the initial hold, the temperature was ramped at 200°C/min to a final temperature of 200°C and held at this temperature for an additional 4 minutes. The software for the tandem system turned the GC/MS on 0.1 minutes after the TGA programming began. The GC/MS used a sample loop that opened from 0.1–1.0 minutes, injecting onto the column at the end of this interval. The GC method was started at a temperature of 40°C for the first 5 minutes, followed by a ramp of 20°C/min for 10 minutes and finally held at 240°C for 5 minutes for a total of 20 minutes.

The results showed three major peaks in the analysis of the outer shell of the turnout gear. The first of the peaks showed a mass spectrum that had characteristics of benzophenone derivatives. The second peak showed similarities to a terephthalate
**Figure 2:** Bunker pants log-in and labeling.

**Figure 3:** Combined TGA, FT-IR, and GC/MS analysis system.
compound with a strong peak at an atomic mass of 163, which is very common for a terephthalate compound, such as dimethyl or another dialkyl compound. The last of the peaks showed strong similarities to long chain aliphatic acids or alcohols.

Further in-depth analysis was performed on additional sets of bunker gear in order to better identify the contaminants on the exposed turnout gear. Once again, a 4-hour Soxhlet extraction was used. From each sample to be analyzed a 2″ circle was cut and then each layer of the turnout gear was placed in a Soxhlet extractor with 75 mL of methanol. After the 4 hour extraction, a fraction of the extraction solution was collected for analysis on GC-MS to attempt to identify extracted contaminants.

These analyses showed the presence of several different contaminants. One contaminant that was detected in several of the extractions was a peak occurring at a retention time of approximately 11.88 minutes. The mass spectrum pattern of this peak shows similarities to the compound 1-phenoxypropan-2-ol as can be seen in Figure 4 below.

Figure 4: Mass spectrum showing 1-phenoxypropan-2-ol.

According the PubChem database, 1-phenoxypropan-2-ol is used in ink, toner, and colorant products. Another peak that occurred frequently amongst the extracted samples was a peak occurring at a retention time of 12.74 minutes and that had a mass spectrum pattern with characteristics that match p-cresidine, shown in Figure 5.

Figure 5: Mass spectrum showing p-cresidine.
According to the PubChem, the sole purpose of the p-cresidine is to be used as an intermediate in the production of dyes. A final peak that showed a strong match to a compound within the NIST MS library was that of N-methyldioctylamine (Figure 6).

**Figure 6**: Mass spectrum showing N-methyldioctylamine.

The primary use of N-methyldioctylamine is as a surfactant per the PubChem Database. There were several additional peaks that were not consistently identified via matching to the NIST database and further analysis of the peaks was needed to identify these compounds. This investigation of used bunker gear repeatedly demonstrated inconsistent at best recovery of contaminants and showed significant variations from sample to sample.

### Controlled Contamination of Clothing Samples

#### Soot Deposition Approach

In an effort to repeatedly create byproducts of common fire contaminants, a multi-stage tube furnace was configured as seen below. The tube furnace allowed pelletized items such as PVC to be heated in a controlled manner, creating smoke with desired particulates. These particulates were then flushed through the 4 inch heating tube into a sample contamination chamber as seen in Figures 7 and 9 below. Within this chamber a stainless steel sample holder was mounted such that the airstream impacted the surface of an outer shell sample (Figure 8). Air sampling probes were also inserted both before and after the sample holder to collect contaminants. The absorbent within the tubes was also analyzed to assist in determining volume and types of contaminants the fabrics were exposed to.
Figure 7: Tube furnace assembly.

Figure 8: Sample mounting fixture.

Figure 9: Sample contamination chamber.
Multiple test runs were performed in this configuration utilizing simple chemicals such as pelletized lab-grade PVC. While most of the test runs created visibly similar deposition of combustion by-products, chemical analysis showed significantly varied results at best, and the consistent deposition of contaminants proved to be challenging. Additionally, during some of the experiments, it was suspected that some of the by-products were being pushed off of the fabric due to the chamber temperatures following deposition. Below in Figures 10 and 11 are examples of some of the more common situations in which the post analysis of a fabric exposed to a burn of PVC only resulted in the presence of 14-methyl pentadecanoic ester and 14-methyl hexadecanoic ester. Both of these esters are possibly present within the pellets as they are fatty acids used in the lubrication of equipment during plastic production, but the desired plastic by-products were not measurably present on the fabric.

Figure 10: Mounted tube furnace contaminated materials.

Figure 11: Contaminated material.
Conventional Sample Doping Approach (Unwashed Samples)

Based upon the non-repeatable results from the tube furnace experiment, an alternative procedure was explored in which pristine outer shell material was immersed for one hour in 100,000 µg/ml concentrations of three representative chemical types highlighted from an original suspected contaminant list (Figure 12). Fabric swatches were then allowed to fully dry within a chemical hood and then extracted utilizing a Soxhlet and methanol as the solvent.

Extraction liquids were then analyzed and resulted in the following fabric contamination levels. These contamination levels were not as high as anticipated, but did show significant potential for a repeatable deposition of chemicals upon a fabric. It was suspected that the pristine materials topical finishes reduced the amount of chemical that could be absorbed by the fabric. It is noteworthy that the final concentrations of each chemical in fabric samples varied even though the fabric samples were exposed to the same concentration of chemical in the doping liquid. This further demonstrated that fabric absorption would be affected by the specific chemical(s) being used.

<table>
<thead>
<tr>
<th>Target Contaminant</th>
<th>Classification</th>
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<tbody>
<tr>
<td>Phenanthrene, Pyrene</td>
<td>PAH</td>
</tr>
<tr>
<td>4-Methyl Phenol, Pentachlorophenol</td>
<td>Phenol</td>
</tr>
<tr>
<td>DEHP</td>
<td>Phthalate</td>
</tr>
<tr>
<td>PBDE-47</td>
<td>Fire retardant</td>
</tr>
<tr>
<td>Octacosane</td>
<td>Hydrocarbon</td>
</tr>
<tr>
<td>Arsenic, Lead</td>
<td>Heavy metal</td>
</tr>
<tr>
<td>Xylene (mixed)</td>
<td>VOC</td>
</tr>
</tbody>
</table>

Figure 12: Original suspected contaminant list.

Figure 13: Fabric contamination levels using conventional doping technique.

<table>
<thead>
<tr>
<th>Chemical</th>
<th>Concentration, µg/mL</th>
<th>Total µg found</th>
<th>µg/cm²</th>
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<tr>
<td>Naphthalene</td>
<td>12.32</td>
<td>924</td>
<td>2.92</td>
</tr>
<tr>
<td>Pentachlorophenol</td>
<td>8.96</td>
<td>672</td>
<td>2.03</td>
</tr>
<tr>
<td>Diocyl terephthalate</td>
<td>18.05</td>
<td>1354</td>
<td>4.28</td>
</tr>
</tbody>
</table>

Chemical Doping Approach (Washed Samples)

With the prior experiments, it was suspected that topical finishes on the outer shell material inhibited absorption of the surface contaminants. To determine if greater amount of chemicals could be absorbed through the fabric, samples of the selected Fabric A (7.5 osy 55% para-aramid, 37% PBI, 8% liquid crystal polymer, multi-filament ripstop fabric) were laundered for 10 cycles in accordance with NFPA 1971-13 in accordance with Section 8.1.2.

This experiment was also expanded further to look at a more complete grouping of contaminants that included:

- Bis(2-ethylhexyl)phthalate
- Pentachlorophenol
- Phenanthrene
- Naphthalene
- Octacosane
Controlled Contamination of Clothing Samples

• Xylene
• 2,2′,4,4′,5,5′-hexachlorobiphenyl
• 2,2′,4,4′-tetrabromodiphenyl ether
• Arsenic
• Lead

This portion of the project was performed in collaboration with Intertek’s Allentown, PA lab which specializes in complex chemical analytics. Different analytical approaches were applied to different chemical groups (organic versus inorganic) as described in the following sections.

Organic Components

To prepare the contaminated fabric, approximately 0.5 g of the fabric was placed in a large mouth jar and a known quantity of a standard solution containing bis(2-ethylhexyl) phthalate, pentachlorophenol, phenanthrene, naphthalene, octacosane and m-xylene was added to the fabric by dripping small amounts on the fabric. The 2,2′,4,4′,5,5′-hexachloro-biphenyl and 2,2′,4,4′-tetrabromodiphenyl ether were also added in the same way using standards purchased as solutions and added as received. After the fabric had dried, the analytes were extracted using methylene chloride, and the solution was analyzed to determine how much chemical was absorbed in the fabric and how much remained in solution.

The analysis was performed by gas chromatography mass spectrometry using a 30-meter Rtx-35 capillary column (0.25 mm ID with 0.25 µm film thickness) interfaced to a gas chromatograph combined with a mass spectral detector (GC/MS) (Figure 14). The mass spectrometer was operated in dual Scan/Selective Ion Monitoring (SIM) mode.

Figure 14: Gas chromatograph/mass spectral (GC/MS) detector used in analyses.
Quantitation of the recovered analytes was done by external standardization. A series of standards containing bis(2-ethylhexyl)phthalate, pentachlorophenol, phenanthrene, naphthalene, octacosane, and m-xylene were prepared in methylene chloride that bracketed the expected range in the sample. The 2,2',4,4',5,5'-hexachlorobiphenyl standards were prepared in hexane and the 2,2',4,4'-tetrabromodiphenyl ether standard was prepared in octane. The square of correlation coefficient ($R^2$) for the calibration curves for each of the components was 0.994 or better.

**Inorganic Components**

The mechanism to deposit the arsenic and lead as the two representative heavy metals to the surface of the fabric utilized the nebulizer from the SF-ICP-MS instrument. A solution of arsenic and lead was prepared in 2-propanol. The solution contained 1000 nanograms of each element in 10 mL of the electronics grade isopropanol. The nebulizer created an aerosol from an argon gas stream. The nebulizer used is designed to allow for a flow of 0.4 mL/minute. The mist generated was directed onto fabric swatches of 3 inches square. The mist was directed to both sides of the cloth to deposit the majority of the solution. Three separate experiments were performed on 3 inch pieces of fabric. The fabrics were then allowed to dry in a hood for an overnight period.

The arsenic and lead were initially removed from the fabric by rinsing both sides of the fabric with 5 mL aliquots of dilute acid (nitric acid used for these experiments). A total of 50 mL acid was used for the rinse. The fabrics were also immersed in the 50 mL solution. The solutions were then analyzed using sector field inductively coupled plasma mass spectrometry (SF-ICP-MS).

**Figure 15:** Sector field inductively coupled plasma mass spectrometer (SF-ICP-MS) used in analyses.
Discussion and Observations

Given that precise amounts of organic chemicals were applied to the fabric, it was possible to determine the extent of chemical that could be extracted and quantified, known as percent recovery. Figure 16 lists each contaminant and the associated percent recovery.

**Figure 16: Recovery of organic contaminants.**

<table>
<thead>
<tr>
<th>Contaminant (Analyte)</th>
<th>Percent Recovery</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bis(2-ethylhexyl)phthalate</td>
<td>112</td>
</tr>
<tr>
<td>Pentachlorophenol</td>
<td>81</td>
</tr>
<tr>
<td>Phenanthrene</td>
<td>92</td>
</tr>
<tr>
<td>Naphthalene</td>
<td>119</td>
</tr>
<tr>
<td>Octacosane</td>
<td>75</td>
</tr>
<tr>
<td>m-xylene</td>
<td>65</td>
</tr>
<tr>
<td>2,2′,4,4′,5,5′-hexachlorobiphenyl</td>
<td>125</td>
</tr>
<tr>
<td>2,2′,4,4′-tetrabromodiphenyl ether</td>
<td>125</td>
</tr>
</tbody>
</table>

Only one of the xylene isomers was used because two of the isomers co-elute. M-Xylene has the lowest percent recovery because it was believed that some chemical evaporated under room temperature conditions. The reason that some percent recoveries are higher than 100% is due to the error associated with the relatively small quantities that were used in contaminating the fabrics. This aspect was combined with the fact that different samples were used in the analyses.

Figure 17 shows the SIM chromatogram for the methylene chloride fabric extract. A sample key follows to identify the numbered peaks. The 2,2′,4,4′,5,5′-hexachlorobiphenyl and 2,2′,4,4′-tetrabromodiphenyl ether peaks are much smaller than the others because these were purchased as 50 ppm and 100 ppm solutions respectively, while the other components were spiked at a higher level.

The m-xylene showed a low recovery compared to the rest of the components. This may be due to its higher volatility, as demonstrated by the fact that it has the earliest elution time. Some of the m-xylene also may have been lost during the spiking process due to its volatility.

The inorganic analysis initially targeted just the rinse of the fabric pieces. However, the recovery of selected metals from the rinse procedures was only approximately 30% for both arsenic and lead. It was speculated that the outer shell fabric hydrophobic properties (due to the finishes present) made it difficult for the rinse procedures to efficiently remove both heavy metals. Therefore, the rinsed fabrics were then placed in the original collection of dilute acid and extracted for an additional period of 30 minutes in an ultrasonic bath. The recovery on their removal then improved to > 90%. These recoveries are shown in Figure 18 for three sets of replicate samples.

Summary and Conclusions

Simple procedures involving the misting or dripping of known quantities of standard solutions onto swatches of fabric were found to be satisfactory for creating contaminated fabric for testing. Extractions in appropriate solvents (methylene chloride for organic species and dilute nitric acid for inorganics) followed by GC/MS or SF-ICP-MS showed acceptable recovery of all target analytes with the exception of xylene. m-Xylene recovery was low primarily because of losses that occurred during application of the standard contaminant mixture to the fabric due to its high relative volatility.
Augmentation of Contamination with Carbon Particles

In a fourth set of experiments, work from the previous research for developing contamination procedures was built upon. Prior efforts showed reasonable recoveries for nearly all contaminants using a technique for dripping solutions of organic contaminants onto fabric specimens and applying a mist of inorganic contaminants onto a different set of specimens. However, it was believed that these techniques did not account for the extensive particulate matter that accounts for a significant amount of fireground contamination and repository for specific contaminants. Thus, for this set of tests, a “soot-like” contaminated fabric was utilized as the foundation for the new experiments. To create a consistent “soot-like” contamination, a Martindale abrasion machine as seen in Figure 19 was utilized. This approach was chosen due to the ability of the machine to reproducibly abrade multiple fabric specimens simultaneously. The Martindale abrade rubs over a broad but precisely controlled pattern on the respective specimens. For this set of experiments, a set of laundered Fabric A outer shell fabric
specimens were mounted on the lower mounting surface as seen in the below Figure 20 and 0.05 gram of Lab-Grade 99.9% Acetylene-based Carbon Black (Figure 21) was applied in a small pile onto the surface beneath where the moving head contacts the lower surface where the fabric specimen was mounted. This carbon black was then ground into the material specimen for 80 complete abrasion cycles, and any loose material was lightly tapped off of the surface.

**Specimen Handling and Treatment**

Following being loaded with carbon, the fabric specimens were individually laid flat, wrapped in aluminum foil, and then stacked on top of each other within a plastic bag for shipping to Intertek Allentown for contamination with liquid chemicals.

Once received in Allentown, fabric specimens were placed in large mouth jars, and a known quantity of a standard solution containing all of the target compounds was applied by carefully dripping small amounts onto the fabric. After the spiked fabric had dried, some of the fabric specimens were laundered and others were not.

The nebulizer from an ICP instrument was used to deposit the arsenic and lead solution to the surface of the fabric specimen. The solution of arsenic and lead was prepared in 2-propanol. The solution contained 100 ng of each element per milliliter in electronics grade isopropanol.

Within 48 hours of the fabrics drying, specimens were split in sample groups according to the table below (Figure 22) and were then repackaged within new pieces of aluminum foil and sent to Intertek Cortland. Figures 23 and 24 show photographs for the respective manner for how the individual specimens were packaged, repackaged, and labeled.
Figure 20: Mounted Fabric A.

Figure 21: Lab-grade carbon black.

Figure 22: Specimen preparation plan. Each material evaluated in triplicate.

<table>
<thead>
<tr>
<th>Sample Group*</th>
<th>Analyte Group</th>
<th>Cortland Carbon</th>
<th>Allentown Chemical Loaded</th>
<th>Cortland Laundered</th>
<th>Allentown Chemical Analysis</th>
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</thead>
<tbody>
<tr>
<td>A</td>
<td>Organic</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>B</td>
<td>Organic</td>
<td>X</td>
<td>X</td>
<td></td>
<td>X</td>
</tr>
<tr>
<td>C</td>
<td>Organic</td>
<td>X</td>
<td>X</td>
<td></td>
<td></td>
</tr>
<tr>
<td>D</td>
<td>Organic</td>
<td>X</td>
<td>X</td>
<td></td>
<td></td>
</tr>
<tr>
<td>E</td>
<td>Inorganic</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>F</td>
<td>Inorganic</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>G</td>
<td>Inorganic</td>
<td>X</td>
<td></td>
<td></td>
<td>X</td>
</tr>
<tr>
<td>H</td>
<td>Inorganic</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td></td>
</tr>
</tbody>
</table>

* Equal sets of specimens were prepared for each sample group.
Figure 23: Complete contaminated sample package.

Figure 24: Individual package sample.
Intertek Cortland then removed specimens from their packaging and mounted the 12 specified specimens onto clean bunker coat as shown in Figures 25 and 26 at the positions shown and listed in Figure 27. Mounted specimens are shown in Figures 28 through 30.

**Figure 25:** Pre-laundered bunker gear coat front.

**Figure 26:** Pre-laundered bunker gear coat back.
**Figure 27:** Test specimen mounting locations.

<table>
<thead>
<tr>
<th>ID</th>
<th>Chemical</th>
<th>Carbon Loaded (Y/N)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A3</td>
<td>Organic</td>
<td>Y</td>
</tr>
<tr>
<td>A4</td>
<td>Organic</td>
<td>Y</td>
</tr>
<tr>
<td>A5</td>
<td>Organic</td>
<td>Y</td>
</tr>
<tr>
<td>D1</td>
<td>Organic</td>
<td>N</td>
</tr>
<tr>
<td>D2</td>
<td>Organic</td>
<td>N</td>
</tr>
<tr>
<td>D3</td>
<td>Organic</td>
<td>N</td>
</tr>
<tr>
<td>E1</td>
<td>Inorganic</td>
<td>Y</td>
</tr>
<tr>
<td>E2</td>
<td>Inorganic</td>
<td>Y</td>
</tr>
<tr>
<td>E3</td>
<td>Inorganic</td>
<td>Y</td>
</tr>
<tr>
<td>H7</td>
<td>Inorganic</td>
<td>N</td>
</tr>
<tr>
<td>H8</td>
<td>Inorganic</td>
<td>N</td>
</tr>
<tr>
<td>H9</td>
<td>Inorganic</td>
<td>N</td>
</tr>
</tbody>
</table>
**Figure 28:** Mounted test specimens.

**Figure 29:** Mounted organic test specimens.

**Figure 30:** Mounted inorganic test specimens.
Post mounting, the contaminated specimens were then washed one complete cycle in accordance with NFPA 1971, Section 8.1.11 utilizing the standard AATCC specified detergent, ballast loading, and temperatures and then dried also in accordance with the NFPA 1971 standard for 60 minutes. It was noted that the fabric specimens all felt fully dried to the touch and the carbon soot staining appeared much lighter than pre-laundering. Nevertheless, the specimens were still visibly darkened following laundering. Photos of the garments immediately after drying are provided in Figures 31 through 34. After drying, the specimens were then repackaged in aluminum foil and sent back to Allentown for analysis.

**Figure 31:** Post laundering specimens (full coat).
Figure 32: Post laundering inorganic specimens.

Figure 33: Post laundering organic specimens.

Figure 34: Close-up of laundered carbon and chemical loaded fabric.
Analysis of Organic Contaminant Removal

Upon receipt in Allentown, the specimens were treated with methylene chloride to extract the target organic compounds. The analysis was again performed by gas chromatography with mass spectrometry using a 30-meter Rtx-35 capillary column (0.25 mm ID with 0.25 µm film thickness) interfaced to a gas chromatograph combined with a mass spectral detector (GC/MS). The mass spectrometer was operated in dual Scan/Selective Ion Monitoring (SIM) mode.

Quantitation of the recovered analytes was done using external standardization. A series of standards containing each of the compounds of interest was prepared in methylene chloride. The square of correlation coefficient ($R^2$) for the calibration curves for each of the components was 0.995 or better.

For each group of specimens, the spike recoveries of the target analytes were determined. These values are reported in Figure 35 below as the average of the three replicate specimens. Data from the previous analysis of clean fabric and contaminated fabric are also included for comparison. None of the previous samples were laundered.

Any peaks present in the unspiked contaminated (control—Group C) specimens were taken into account when calculating the percent recovery of the spiked samples. Some of the peak recoveries in the non-laundered specimens (Group B) were not within the usual accepted range (75-125%)—these values appear in the first and third columns. It is suspected that the increased handling of the specimens in being transported between Allentown and Cortland (back and forth) combined with the relative volatility, contributed to these low recovery values.

Figure 35: Recovered percentages of analyte.

<table>
<thead>
<tr>
<th>Contaminant</th>
<th>Previously Reported Clean Fabric LIMS 116913</th>
<th>Previously Reported Samples Contaminated with Soot LIMS 118545</th>
<th>Group B Contaminated with Carbon, Not Laundered</th>
<th>Group A Contaminated with Carbon, Laundered</th>
<th>Group D Contaminated (no Carbon), Laundered</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pentachlorophenol</td>
<td>81</td>
<td>90</td>
<td>96</td>
<td>26</td>
<td>25</td>
</tr>
<tr>
<td>Phenanthrene</td>
<td>92</td>
<td>131</td>
<td>117</td>
<td>54</td>
<td>67</td>
</tr>
<tr>
<td>Octacosane</td>
<td>75</td>
<td>124</td>
<td>54</td>
<td>50</td>
<td>51</td>
</tr>
<tr>
<td>Naphthalene</td>
<td>119</td>
<td>NA</td>
<td>1</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Diethylhexyl phthalate (DEHP)</td>
<td>112</td>
<td>NA</td>
<td>68</td>
<td>50</td>
<td>50</td>
</tr>
<tr>
<td>m-Xylene</td>
<td>65</td>
<td>NA</td>
<td>10</td>
<td>10</td>
<td>9</td>
</tr>
<tr>
<td>2,2’,4,4’,5,5’-Hexachlorobiphenyl (PCB 153)</td>
<td>125</td>
<td>NA</td>
<td>209</td>
<td>177</td>
<td>203</td>
</tr>
<tr>
<td>2,2’,4,4’,5,5’-Tetra-bromo-diphenyl ether (BDE-47)</td>
<td>125</td>
<td>NA</td>
<td>156</td>
<td>131</td>
<td>264</td>
</tr>
</tbody>
</table>
The spike recoveries for the laundered samples were low, which led to the conclusion that the laundering of the samples assisted in removing the components of interest. However, it was clear contaminant removal was not complete, and in most cases the compounds were still detected even if at lower levels. To more clearly illustrate these findings, these analytical results were also presented on the basis of decontamination efficiencies taking into account the ability to recover specific analytes by comparing the raw specimen chemical concentration values for Specimen Set B (contaminated but not laundered) with both Set A (contaminated with carbon, laundered) and Set D (contaminated without carbon, laundered). These values are presented in Figure 37. The results are also shown graphically in Figure 38.

**Figure 36:** Overlaid SIM chromatogram of contaminated fabric with and without a spike.

**Figure 37:** Calculated decontamination efficiencies for carbon and non-carbon specimens.

<table>
<thead>
<tr>
<th>Contaminant</th>
<th>Concentration (ppm)</th>
<th>Decontamination Efficiency</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Contaminated</td>
<td>Contaminated</td>
</tr>
<tr>
<td></td>
<td>Carbon-loaded</td>
<td>Laundered</td>
</tr>
<tr>
<td>Pentachlorophenol</td>
<td>57.8</td>
<td>14.0</td>
</tr>
<tr>
<td>Phenanthrene</td>
<td>61.8</td>
<td>25.3</td>
</tr>
<tr>
<td>Octacosane</td>
<td>46.7</td>
<td>39.4</td>
</tr>
<tr>
<td>Naphthalene</td>
<td>0.5</td>
<td>0.0</td>
</tr>
<tr>
<td>Diethylhexyl phthalate (DEHP)</td>
<td>82.8</td>
<td>56.6</td>
</tr>
<tr>
<td>m-Xylene</td>
<td>5.9</td>
<td>5.8</td>
</tr>
<tr>
<td>2,2',4,4'-Tetra-bromo-diphenyl ether (BDE-47)</td>
<td>1.0</td>
<td>0.8</td>
</tr>
<tr>
<td>2,2',4,4',5,5'-Hexachlorobiphenyl (PCB 153)</td>
<td>1.8</td>
<td>1.6</td>
</tr>
</tbody>
</table>
A number of interesting observations can be made with respect to the reported decontamination efficiencies.

- Reasonably high levels of decontamination (70 to 100%) took place for pentachlorophenol and naphthalene, although as previously pointed out, the recovery for naphthalene based on the applied amount was low.
- Intermediate to low levels of decontamination (30 to 70%) were found for phenanthrene and DEHP.
- Relatively low levels of decontamination occurred for all of the other chemicals.
- Xylene decontamination was practically nil and it is believed that this chemical simply evaporated with any residue indicative of an insignificant part of the chemical that was retained by the fabric.

Some differences were noted between fabric specimens that included carbon versus those that did not include carbon. For most chemicals, the decontamination efficiency was higher for non-carbon loaded specimens. This suggests that the carbon acts to retain some contaminant. This is a potentially important finding that would support that increased removal of soot ensures greater cleaning.

It is noted that the NFPA 1971 laundering procedures used in this study for demonstration purposes are not optimized for soil removal, but rather to create conditions to remove finishes. Many independent service providers use more extensive laundering procedures that are coupled with presoak steps and longer suds or detergent use in the washing process.

**Inorganic Components**

Calibration standards were prepared from 10 parts per million single element solutions acquired from Inorganic Ventures. Standards ranged from 0.05 to 50 parts per billion. The solutions were then analyzed using a Thermo Scientific Element 2 SF-ICP-MS. The correlation coefficient ($R^2$) for the calibration curves were greater than 0.9999. The 10 ppb QC check standard for arsenic and lead was 9 ppb in both cases. Results are reported in nanograms (ng) in Figure 39. The percent recoveries are reported based on the amount of analyte applied to the fabrics. Note that the recoveries only apply to the amount of the arsenic and lead loaded, and do not take into account levels of the analyte from the fabric or the carbon loading.
The levels of arsenic and lead in the starting fabric were measured in the original work in August 2016. (Allentown request number 116913; CSA IntertekCortland072216av1-MM) This work included blanks consisting of clean fabric with no carbon or analyte loading. The concentration of arsenic extracted from these blanks was determined to be below measureable levels, while lead levels were 20 to 50 ng. The carbon-loaded blank in the current work (Group G) shows significant lead concentrations in the extract even though no metals were applied to the fabric. It therefore appears that the carbon black contains lead, which will skew the recovery data for Groups E, F, and G. It should also be noted that carbon loading of Group G Sample 1 was done at a different time from the carbon loading for Groups E and F and Group G Samples 2 and 3. This could explain the higher lead levels detected in Group G Sample 1 versus the other two samples in that group.

As with the organic analysis, the recovery data were repositioned to permit the calculation of decontamination efficiency. This required using the Group F data to constitute the normal levels of contamination through the misting process and then determining the removal by laundering with carbon either present or not present on the specimens (Groups E and H, respectively). The results of these calculations are shown in Figure 40.

The decontamination efficiency for arsenic appeared to be at a relatively high level though removal of lead was much less efficient. For both heavy metals, the decontamination efficiency was lower for fabric specimens without carbon than it was for carbon-loaded fabric specimens. Surprisingly, given the high background levels of lead in the carbon, this did not affect the decontamination efficiency since it would have been expected that the carbon-loaded fabrics would have had a lower decontamination efficiency, which they did not. Instead, it appears that some adsorption of heavy metals on the carbon and the removal of carbon provided for some improvement of cleaning effectiveness. The mechanisms for large differences between arsenic and lead removal are not yet understood but could be affected by relative solubilities of the heavy metal salts as applied to the fabric specimens.

**Summary and Conclusions**

The previously developed methods detailed within the second set of the experiment for applying the target compounds were utilized on the carbon-loaded and clean fabrics. Extraction in methylene chloride followed by GC/MS analysis showed variable recovery of the organic target compounds before laundering that may be due to chemical volatility and the extended period of time for analysis. The laundering of the specimens shows widely varying levels of contaminant removal. The results show...
suggested that the presence of carbon allowed greater fabric specimen retention of contaminants though the differences were small for several chemicals. Recoveries of the inorganic components were also acceptable; however, it was noted that the carbon black had background levels of lead. Lower levels of decontamination efficiency were noted for lead as compared to arsenic. Decontamination efficiencies were higher for carbon-loaded fabrics as compared to non-carbon specimens.

**Overall Study Findings and Recommendations**

A preliminary investigation was undertaken to evaluate chemical contaminants in used, exposed firefighter clothing and developed methodology that could be used to assess cleaning effectiveness.

The ability to analyze clothing materials for contaminants is fraught with multiple issues for discerning contamination levels. The specific identification and quantification of contaminants is strongly affected by the extraction and analytical procedures that are used. In this study, several substances were found at higher levels that may have more to do with the fabric composition and general use of the product than fireground contamination such as fabric dyes/finishes and fatty acids from skin contact. Therefore, any analytical work to discern contaminated levels over a broad range of chemical substances must include relatively low levels of sensitivity and specifically target certain groups of chemical substances of interest, e.g., phthalates and polynuclear aromatic compounds. Detailed extraction and analytical procedures should be standardized to enable consistent forms of determining contamination.

It is recognized that in order to understand cleaning effectiveness, specific techniques are needed to provide uniform and repeatable contaminate specimens that can be subjected to a specific cleaning or laundering process and then evaluated for residual chemicals. Ideally, the contamination process should emulate fireground exposures and in particular the deposition of carbon particles via soot onto clothing materials.

Intertek attempted to create a soot deposition technique by the burning of a known fuel in a tube furnace and directing the off gassing particles and chemicals onto fabric specimens in the effluent path. While this approach appeared to provide samples that simulate the appearance of field contaminated clothing, the method proved to be inconsistent and not yield a full range of contaminants of interest for general firefighter clothing use in structural fires. While additional efforts could have been spent optimizing fuel load and exposure conditions, it was believed that the technique simply could not reliably contaminate specimens for evaluating cleaning process effectiveness.

Intertek was able to develop a hybrid technique though a stepwise development process that entailed the use of carbon black as a soot surrogate in combination with more conventional chemical doping methods. The specific approach use was
to grind a known amount of carbon black into the material and then either drip or mist a specific quantity of either organic or inorganic contaminants onto the carbon loaded specimens. It was observed that direct chemical addition to fabric specimens provided high rates of recovery, i.e., the amounts of chemical found from extraction analysis closely matched the applied levels of chemical. When carbon was added to the material and a larger period of time transpired between carbon loading, chemical contamination, and analysis, lower recoveries were found. This finding suggests that specific care must be applied in the selection of chemicals such that the chosen chemical do not evaporate or are not affected by long-term storage.

The technique for contaminating fabric specimens with carbon and chemicals did prove useful for evaluating cleaning effectiveness. For the range of organic chemicals and representative inorganic heavy metals, specific decontamination efficiencies were measured. These efficiencies were compared for both carbon-loaded and non-carbon-loaded specimens. As a result of this analysis, it was learned that carbon-loaded fabrics retain more organic chemical during laundering than fabric specimens without the additional carbon. However, the reverse was true for the inorganic heavy metals. It was further learned that the carbon itself could be a source of lead contamination. Therefore, adjustments are needed in the types of soils to ensure relatively clean backgrounds.

Based on the visual appearance of the fabrics following laundering, it was recognized that some adjustment of the amount of carbon added to the fabric must be made since the selected cleaning process still indicated the presence of carbon-based soils. It is expected that with refinement of the process for adding carbon to the fabric specimens, improvements for the realism of the contamination levels and general effectiveness for visual soiling removal as now achieved by current laundering practices can be mimicked. Therefore, with the optimization of carbon deposition levels, careful selection of chemical contaminants (based on relevance, physical properties, and analytical capabilities), and appropriate application of chemicals, a reliable contamination technique can be established.

**Recommendations to Standard Development**

Based upon a significant amount of anticipated changes within NFPA 1851, 2017 edition, specific sections and organization of the below method will likely be different in the final accepted version of the test method. Below are suggested high-level test method details for the consideration by the committee:

**11.3.7** For verification of an organization’s advanced cleaning services, the certification organization shall evaluate the organization’s procedures in accordance with Section 7.3 of this standard. For verification of an ISP’s advanced cleaning services, the certification organization shall evaluate the ISP’s procedures in accordance with Section 7.3 of this standard and evaluate the effectiveness of its cleaning processes against the requirements specified in 11.3.7.1 and 11.3.7.2.

**11.3.7.1** When tested for removal of selected products of combustion as specified in 12.4, Chemical Decontamination Efficacy Test, the ISP cleaning process shall provide for a 90% or greater decontamination efficiency for each of the specified surrogate contaminants.

**12.4 Chemical Decontamination Efficacy Test**

**12.4.1 Application.** This test method shall apply to the decontamination of outer shell material that is subjected to ISP cleaning procedures for advanced cleaning employed for the removal of products of combustion from structural firefighting protective garments.

**12.4.2 Samples.** Pristine 100 mm (4.0 in.) diameter surrogate outer shell material samples shall be laundered ten times in accordance with Section 8.1.2 of NFPA 1971, 2013 edition.

**12.4.2 Specimens.** A total of 12 pieces of surrogate outer shell material shall be tested.

**12.4.3 Procedure.**

**12.4.3.1** Six specimens shall be mounted to the lower mounting stage of a Martindale abrasion machine.
12.4.3.2 Specimens shall be loaded with 0.05 g ± 0.005 g each of 99.9% Acetylene based Carbon black for 80 cycles at utilizing 9kPa head pressure. The top abradant head shall be the 30 mm (1.2 in.) diameter stainless steel head.

12.4.3.3 All 12 specimens shall be contaminated with the following chemical quantities as specified in Table 12.4.3.3:

<table>
<thead>
<tr>
<th>Specimens</th>
<th>Chemical</th>
<th>Amount</th>
</tr>
</thead>
<tbody>
<tr>
<td>1–6</td>
<td>Pentachlorophenol</td>
<td>2.5 g of a 625 ppm standard</td>
</tr>
<tr>
<td>1–6</td>
<td>Phenanthrene</td>
<td>2.5 g of a 625 ppm standard</td>
</tr>
<tr>
<td>1–6</td>
<td>Octacosane</td>
<td>2.5 g of a 625 ppm standard</td>
</tr>
<tr>
<td>1–6</td>
<td>Naphthalene</td>
<td>2.5 g of a 625 ppm standard</td>
</tr>
<tr>
<td>1–6</td>
<td>Diethylhexyl phthalate</td>
<td>2.5 g of a 625 ppm standard</td>
</tr>
<tr>
<td>1–6</td>
<td>Xylene</td>
<td>2.5 g of a 625 ppm standard</td>
</tr>
<tr>
<td>1–6</td>
<td>2,2′,4,4′-Tetrabromodiphenyl ether</td>
<td>2.5 g of a 625 ppm standard</td>
</tr>
<tr>
<td>1–6</td>
<td>2,2′,4,4′,5,5′-Hexachlorobiphenyl</td>
<td>2.5 g of a 625 ppm standard</td>
</tr>
<tr>
<td>7–12</td>
<td>Arsenic</td>
<td>600+/–50 ng</td>
</tr>
<tr>
<td>7–12</td>
<td>Lead</td>
<td>600+/–50 ng</td>
</tr>
</tbody>
</table>

12.4.3.4 Organic chemical contamination shall be performed by placing fabric samples in large mouth jars and dripping from above specified quantity of a standard solution containing all of the target compounds on the carbon loaded portion of the fabric.

12.4.3.5 Inorganic chemical contamination shall be performed utilizing the nebulizer from an ICP instrument to deposit the arsenic and lead to the carbon loaded surface of the fabric. A solution of arsenic and lead was prepared in 2-propanol. The solution contained 100 ng of each element per milliliter in electronics grade isopropanol.

12.4.3.6 All specimens shall be allowed to fully dry within a chemical hood for 24 hours.

12.4.3.7 Specimens shall be mounted in the locations as seen in Figure 12.4.3.7 to a standard protective coat that is certified to NFPA 1971-2013 by fastening with a safety pin through the top and bottom of the circles to the outer shell.

12.4.3.8 The protective coat and surrogate specimen assembly shall be cleaned in accordance with the procedures of the ISP. Sufficient ballast of suitable type shall be provided to make up a full load.

12.4.3.9 All 6 organic specimens shall be extracted using methylene chloride or another suitable methodology.

12.4.3.10 All 6 inorganic specimens shall be extracted using a suitable methodology.
12.4.4 Report.

12.4.4.1 The recovered quantity of each chemical in each individual specimen shall be reported.

12.4.4.2 Specimens 1–3 recovered quantities shall be averaged and compared by chemical versus the non-laundered specimens 4–6 averages.

12.4.4.3 Specimens 7–9 recovered quantities shall be averaged and compared by chemical versus the non-laundered specimens 10–12 averages.

12.4.4.4 The average percent reduction for each chemical shall be reported as the decontamination efficiency of the ISP process.

12.4.5 Interpretation. Compliance with the pass/fail requirements shall be determined on the basis of each individual decontamination efficiency for all 8 chemical contaminants.