# EVALUATING THE EFFICIENCY OF AIR SHOWER IN REMOVING LEAD FROM ARMY COMBAT UNIFORM SWATCHES LOADED WITH GUNSHOT RESIDUE

by

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Thesis submitted to the Faculty of the Department of Preventive Medicine and Biometrics Uniformed Services University of the Health Sciences In partial fulfillment of the requirements for the degree of Master of Science in Public Health, 2016



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## ABSTRACT

Evaluating the Efficiency of Air Shower in Removing Lead from Army Combat Uniform Swatches Loaded with Gunshot Residue

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INTRODUCTION. Lead is a well-known toxicant and the exposure on indoor firing ranges presents a health risk to both range employees and shooters. Contaminated clothing spreads lead outside the range and creates a risk of "take-home" lead exposure of family members. To reduce the spread of lead several US Army indoor firing ranges have employed a new control – air shower (AS), although its effectiveness in this particular application has not been examined. The purpose of this study is to evaluate the efficiency of an air shower in removing gunshot-residue-specific lead from Army Combat Uniform (ACU) swatches and to examine a potential lead breakthrough across ACU material during AS application.

METHODS. ACU swatches were loaded with 50-100  $\mu$ g of lead by firing leadcontaining ammunition inside a sealed chamber and allowing the gunshot residue to settle on swatches placed inside the chamber. The overall study design entailed the exposure of lead-loaded swatches inside the air shower to point air velocities of 6,900, 4,100, and 1,800 fpm at the 0-, 45-, and 90-degree angles of impact. Analysis of lead mass remaining on swatches after the exposure indicated the percent of lead removed. The breakthrough lead was isolated inside the breakthrough catchment chamber and was collected by pumping the chamber's air through 37mm filter cassettes and by wiping the inner surfaces of the chamber.

RESULTS. The observed lead reduction ranged from 8.2% to 56.1% and was positively correlated with the amount of lead load (r = 0.885, p < 0.000, 2-tailed  $\alpha =$ 0.01). The study found a positive correlation (r = 0.416, p < 0.000, 2-tailed  $\alpha = 0.01$ ) between selected point air velocities and observed lead reduction. The angles at which air velocities fell on swatches did not have a significant effect on lead reduction. The study confirmed the presence of lead breakthrough during air shower application but was unable to describe it quantitatively and statistically. The study results are limited to the air shower model, Army Combat Uniform material, characteristics of lead load, study approach, and methods.

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# ABBREVIATIONS

ABLES	Adult Blood Lead Epidemiology and Surveillance
ACGIH	American Conference of Government Industrial Hygienists
ACU	Army Combat Uniform
ALA	Delta-aminolevulinic acid
ANOVA	Analysis of Variance
AS	Air shower
Ba	Barium
BEI	Biological Exposure Index
BLL	Blood lead level
СР	Coproporphyrin
DoD	Department of Defense
EPA	United States Environmental Protection Agency
GSR	Gunshot residue
HEPA	High-efficiency particulate arrestance
IARC	International Agency for Research on Cancer
IFR	Indoor firing range
ISML	Isostearamidopropyl morpholine lactate
LOQ	Limit of quantification
NIOSH	National Institute of Occupational Safety and Health
NRD	Nanoparticle respiratory deposition
NTP	National Toxicology Program
OSHA	Occupational Safety and Health Administration
Pb	Lead
PEL	Permissible Exposure Limit
PPE	Personal protective equipment
PVC	Polyvinyl chloride
REL	Recommended Exposure Limit
Sb	Antimony
TLV	Threshold Limit Value
TWA	Time-weighted average
USASOC	United States Army Special Operations Command
ZP	Zinc protoporphyrin

## **CHAPTER 1: Introduction**

#### **BACKGROUND AND LITERATURE REVIEW**

Lead (Pb) is a well-known toxicant and the exposure on indoor firing ranges (IFR) presents a health risk to both range employees and shooters (5). An estimated 16,000-18,000 indoor ranges and 20 million shooters in the United States suggest a significant number of people potentially exposed to Pb (43; 46). New data showing toxic effects of Pb at much lower levels than previously found has intensified efforts in reducing the Pb exposure (42). In this attempt, some indoor firing ranges have employed a new control – air shower (AS), although its effectiveness in this particular application has not been examined. The purpose of this study is to evaluate the efficiency of ASs in removing Pb from Army Combat Uniform (ACU) swatches loaded with gunshot residue (GSR).

#### Lead Exposure on Indoor Firing Ranges

#### Source of Pb on Firing Ranges

Pb on firing ranges comes from an ammunition containing Pb in either the primer or projectile, or a combination of both. The primer commonly contains shock sensitive metallic compounds such as Pb styphnate and Pb peroxide, which could be substituted with non-Pb compounds (59). The projectile could entirely consist of metallic Pb or could be covered with other materials such as copper and nylon. Material wrapping (jacketing) the projectile may cover the Pb core partially or fully. Various combinations of a primer and projectile in ammunition differ from manufacture to manufacture and release different amounts of Pb to the surrounding environment during firearm discharge (67). Firing Pb-containing ammunition produces airborne Pb particles with unique characteristics.

#### Characteristics of Pb in Gunshot Residue

In the course of the firearm discharge, the combustion of ammunition priming compounds, burning of the propellant, and shearing of particles as bullet passes through the barrel produces a "plume" of particles generally called gunshot residue (GSR). Rapid increase in temperature and pressure within the ammunition cartridge vaporize compounds in the primer and exposed surfaces of the projectile. The combusted material escapes the weapon as vapors and solidifies as particulate by rapid cooling (9). This mechanism of particle formation determines the morphology and size distribution of particles in GSR (59).

Forensic studies of GSR morphology using electron microscopy determined that in most cases 70-100% of particles were spheroidal with a smooth, fuzzy, or scaly surface (58; 59). By count, anywhere between 65 and 95% of all GSR particles including Pbcontaining were less than 4  $\mu$ m in size (7; 8; 38). The study of nano characteristics of GSR revealed that even larger GSR particles represent an agglomeration of smaller, nano-size particles (38).

Similar particle size distribution was observed in occupational Pb exposure studies. The unpublished work of Young and Weber (70) showed that vast majority of Pb particles in GSR had aerodynamic diameter of  $\leq 4 \mu m$ . Size-selective air particulate sampling using respirable cyclone and nanoparticle respiratory deposition (NRD) samplers at two military firing ranges using Pb-containing ammunition revealed that 72-

78% of collected Pb mass was attributed to particles <4  $\mu$ m in size and 5-7% to particles <0.3  $\mu$ m (70).

Several other studies had similar results. Lach et al. conducted an evaluation of heavy metal exposure at indoor and outdoor ranges comparing various types of ammunition. The results demonstrated that between 60 and 74% of total Pb mass collected was credited to particles in size region 0.25-2  $\mu$ m (30). Examining various chemical element concentrations on an indoor range, Dams et al. concluded that over 60% of collected Pb mass was associated with particles with aerodynamic diameter of <3.5  $\mu$ m (10).

Both forensic and occupational exposure studies indicated that the majority of Pb particles generated during firearms discharge are  $\leq 4 \mu m$  in size. In addition to having relevance to health effects, particle size is an important aspect of AS efficiency discussed later in this work. Chemically, Pb in GSR is represented by its inorganic forms such as Pb oxides and various combinations of elemental Pb with antimony (Sb) and barium (Ba). Detailed description of chemical and elemental composition of GSR can be found elsewhere (9; 10; 38; 39; 53; 67).

#### Routes, Extent, and Assessment of Pb Exposure on Firing Ranges

Pb-containing GSR particles suspended in the air upon firearm discharge create a concentration of Pb in the air, which varies depending on type of ammunition and weapon used. Bullet fragmentation as it strikes a target or other hard surfaces on the range is another mechanism of Pb particle generation although is a lesser factor as it usually happens on a considerable distance from the shooter (4; 19). Resuspension of settled Pb dust may occur during shooters movement and range cleaning operations

further contributing to airborne Pb concentration. As particles settle with time, virtually all surfaces on the range become contaminated with Pb dust (31).

Inhalation of suspended GSR particles is the primary route of Pb exposure on indoor firing ranges. Inhalation usually occurs at the firing line during shooting and throughout the range during cleaning operations (49). Ingestion of Pb via contaminated hands is also a significant contributor to the overall Pb exposure (28). Ingestion can occur while smoking, handling food, beverages, and other items that contact the mouth (46). Contaminated hands and clothes also spread the Pb outside the range and can result in "take-home exposure" of family members (5; 25). Dermal absorption of Pb is considered negligible by most researchers (2).

Contamination of hands and clothing on indoor ranges could occur in several ways. During firearm discharge, kinetic energy of combustion propels GSR through openings in the weapon (e.g. muzzle and ejection port) and deposits Pb particles on shooter's hands and clothing in the close proximity to the weapon (8; 56). As airborne Pb spreads throughout the range and settles with time, Pb particles could attach to clothing of anyone present on the range (9). Physical contact with contaminated surfaces when the shooter fires from kneeling or prone positions can further contribute to hands and clothing contamination.

Firing range exposure studies showed that both range personnel and shooters are at risk of Pb exposure on firing ranges (1; 11; 20; 21; 66). Overall Pb exposure depends on many factors including the number of shooters on a range, type and caliber of the ammunition fired, rate of fire, and frequency of visits to a range (11; 23). The risk of Pb overexposure is present at both open (outdoor) and indoor firing ranges (30; 34; 64).

The extent of Pb exposure on enclosed firing ranges vary depending on facility design and the person's activities on the range. Indoor ranges such as "baffled" and "shoot house" ranges have higher Pb exposure than outdoor ranges due to a closed space environment (42). Baffled ranges are enclosed facilities designed with side and overhead bullet absorbing baffles and are the most common type of indoor ranges. Closed-space conditions on such ranges facilitate the retention of the airborne Pb around shooters although the exposure could be mitigated effectively with a proper ventilation design. "Shoot house" ranges resemble common close-combat scenarios that allow shooters to move through mock rooms while firing at targets as close as few meters away (34). On these ranges, bullet fragmentation on targets, resuspension of settled Pb dust during maneuvering, and traveling through GSR plumes while shooters move through a shoot house result in a higher Pb exposure, which is not as easily manageable with ventilation systems as on a traditional IFR (34; 42). Enclosed ranges also require more maintenance and cleaning, which increases the risk of exposure for range employees.

To assess Pb exposure on firing ranges, the National Institute for Occupational Safety and Health (NIOSH), the Occupational Safety and Health Administration (OSHA), and the United States Environmental Protection Agency (EPA) have developed several direct and indirect methods of exposure assessment. Direct methods include personal and area air monitoring and dermal sampling for Pb. Personal air monitoring involves collecting air samples within an individual's breathing zone and is the most accurate representation of individual's Pb inhalation exposure. Area sampling of air in close proximity of the shooter also can be used to estimate Pb exposure by integrating time and activity patterns. Collecting samples from shooters hands allows the assessment

of potential risk for lead ingestion. Although direct methods are time, labor, and resource consuming, these techniques produce the most accurate estimation of individuals' Pb exposure.

Indirect methods of Pb exposure assessment comprise various techniques assessing the design and operation of a firing range. These include the review of range design and materials, examination of ventilation system performance, and assessment of proper work practices and cleaning procedures. Although these methods do not directly measure the Pb on a range, they can identify conditions favorable for Pb exposure.

An examination of particular biological markers in a human body after the exposure is another way to estimate the extent of Pb exposure. Blood lead level (BLL) is a universal indicator of recent Pb absorption and widely used by regulatory agencies in establishing exposure limits and standards. Other biomarkers of Pb exposure, effect, and susceptibility such as delta-aminolevulinic acid in urine (ALA-U), blood (ALA-B) or plasma (ALA-P), coproporphyrin in urine (CP), and zinc protoporphyrin (ZP) in blood have been described in detail by Sakai (57).

#### Populations at Risk of Pb Exposure on Firing Ranges

The main population at risk of Pb exposure on firing ranges are range employees and other personnel that spend substantial amount of time on a range such as instructors (34). However, the state-based Adult Blood Lead Epidemiology and Surveillance (ABLES) program managed by NIOSH found that 56% of all firearms-related elevated BLLs during 2002-2012 were among recreational shooters indicating that this population has a potentially similar risk of Pb overexposure (5). In addition, family members especially children could be at risk due to Pb take-home exposure via contaminated hands and clothing of range employees and shooters as has been reported in some studies (25).

# Health Effects of Pb

Health effects of inorganic Pb have been studied extensively and are well-known. Once absorbed into the blood stream via inhalation or ingestion, Pb spreads throughout the body affecting nearly all systems or organs. The most sensitive targets are the developing nervous system, blood-forming and cardiovascular systems, and kidneys (2). The severity of toxic effects increases with duration and extent of exposure to Pb. Figure 1 illustrates health effects of Pb corresponding to certain BLLs.



Figure 1. Effects of inorganic lead on children and adults (63).

In adults, Pb exposure primarily affects peripheral and central nerve systems. Neurological and behavioral symptoms may include forgetfulness, irritability, impaired concentration, depression, increased nervousness, fatigue, decreased libido, dizziness, and weakness. Most common symptoms among range users and employees reported in firing range studies were metallic taste, intermittent headache and abdominal pain, and leg numbness (19; 31; 48; 66).

The most vulnerable populations are pregnant women and children. Some studies have observed associations between BLL and abortions and preterm deliveries among pregnant women. In children, Pb adversely affects the growth, sexual maturation, and cognitive and neurobehavioral development. Several studies found that an increase in BLL is associated with a decline in intelligence quotient (IQ) in children. Children's susceptibility to Pb exposure stems from Pb ingestion due to frequent hand-to-mouth contact and increased absorbance of Pb in gastrointestinal tract (2).

In 2006, the International Agency for Research on Cancer (IARC) placed inorganic Pb in Group 2A – "Probably cancerogenic to humans" based on animal studies. Pb caused cancer in rats and mice exposed orally, by injection, and perinatally (45). However, human studies did not produce adequate evidence of Pb cancerogenicity in humans (2).

In recent years, large number of studies on health effects at low-level BLLs have been published. The US National Toxicology Program (NTP) evaluated the new body of knowledge for evidence of health effects at BLLs below 10  $\mu$ g/dl. The NTP concluded

that there is sufficient evidence that BLLs <10  $\mu$ g/dL in adults and <5  $\mu$ g/dL in children are associated with adverse health effects.

#### **Regulatory Requirements for Occupational Exposure to Lead**

The primary occupational Pb exposure standards have been established by the Occupational Safety and Health Administration (OSHA). The current OSHA Pb standard for general industry 29 CFR 1910.1025, enacted in 1978, prescribes the permissible exposure limit (PEL) and action level applicable to occupational exposure at firing ranges. These exposure limits have been designed to ensure workers BLL does not exceed levels believed to cause negative health effects. Although studies published after 1978 have shown that Pb causes negative health effects at much lower BLLs than were previously adopted by OSHA, employers are still mandated to comply with OSHA standard but encouraged to follow more protective recommendations generated by other agencies.

The OSHA Pb standard sets the PEL at 50  $\mu$ g/m<sup>3</sup> meaning that no employee should be exposed to air Pb concentrations greater than the PEL averaged over an 8-hour period (52). If any employee is exposed to Pb concentrations above the PEL for more than 30 days per year, the employer is mandated to implement engineering and work practice controls to reduce the exposure and conduct exposure monitoring at least quarterly. If the exposure above the PEL is less than 30 day per year, the employer required to implement measures to ensure the exposure do not exceed 200  $\mu$ g/m<sup>3</sup> and continue control efforts toward reaching exposures below the PEL. If engineering and work practice controls do not reduce the exposure below the PEL, the employer shall supplement these controls with respirators and protective clothing. The rationale behind

the PEL is to ensure the employee is not exposed to air Pb concentrations resulting in BLLs of 40  $\mu$ g/dL of whole blood (46). The OSHA allows worker to have BLLs of up to 40  $\mu$ g/dL for the working lifetime of 40 years. For those who plan pregnancies, the OSHA recommends a BLL of under 30  $\mu$ g/dL.

The OSHA Pb standard also sets an action level of  $30 \ \mu\text{g/m}^3$ . If any employee is exposed to air Pb concentrations above the action level, the employee shall monitor the exposure at least semiannually. Once two consecutive observations drop below the action level, the employer may discontinue the monitoring. If the exposure above action level exists for more than 30 day per year, the employer shall enroll the employee in the medical surveillance program. Biological monitoring of BLLs must be performed every 6 months. If employee's BLL reaches or exceeds  $40 \ \mu\text{g/dL}$ , monitoring frequency must be increased to at least once every two months. The action level is used as an indicator of increased risk of reaching maximum permissible BLL and dictates more frequent biological monitoring for early diagnosis of Pb overexposure.

At any time during biological monitoring, if employee exhibits two consecutive BLLs at or above 60  $\mu$ g/dL, or three consecutive BLLs averaging at or above 50  $\mu$ g/dL, the employee must be temporarily removed from work. Upon removal, biological monitoring must continue at least monthly. Employee can return to work only after two consecutive BLLs are below 40  $\mu$ g/dL.

Currently there is no OSHA limit for Pb contamination of surfaces for general industry including firing ranges. However, under the provisions for construction industry the OSHA has established acceptable Pb surface dust level of up to 200  $\mu$ g/ft<sup>2</sup> or 21.52  $\mu$ g/100cm<sup>2</sup> for non-Pb work areas such as hallways, lunchrooms, restrooms, utility rooms

etc. This standard has been used on indoor firing ranges to assess the potential risk of Pb ingestion and the adequacy of ventilation system and housekeeping practices.

In addition to legally binding exposure limits established by OSHA, several other organizations have developed recommendations for occupational Pb exposure limits. Both, the NIOSH recommended exposure limit (REL) and the ACGIH threshold limit value (TLV<sup>®</sup>) for airborne Pb are also 50  $\mu$ g/m<sup>3</sup> as an 8-hour TWA. For the maximum level of Pb in blood, the NIOSH recommends less than 60  $\mu$ g/dL, where the ACGIH biological exposure index (BEI<sup>®</sup>) for Pb is 30  $\mu$ g Pb/dL of whole blood. In light of new knowledge about adverse health effects of Pb at lower BLLs than was previously known, Federal, State, and private organizations are encouraged to adopt more stringent Pb exposure limits.

The Department of Defense (DoD) in its Instruction 6055.01 "Safety and Occupational Health Program" outlines the general provisions of applying exposure limits to subordinate personnel and activities. The DoD directs subordinate Components to comply with the federal regulatory standards established by OSHA at all nonmilitaryunique operations and workplaces (12). Despite the fact that uniquely military equipment, systems, operations, and workplaces defined in part 1960.2(i) Title 29, Code of Federal Regulations are excluded from the federal regulatory standards, the DoD directs subordinate Components to apply the OSHA standards to such activities in whole or in part, as practicable. When such compliance is infeasible or inappropriate, the DoD Components must apply risk management procedures to mitigate the effects of exposure. The DoD also allows Components to develop and apply standards more stringent, alternate, and supplemental to OSHA standards in accordance with procedures outlined in the Instruction. Specific to occupational Pb exposure, the DoDI 6055.01 requires DoD Components to take further steps in implementing the OSHA's requirement of maintaining surfaces as clean of Pb as practicable. The Components must identify all operations generating airborne Pb dust and must establish, document, and integrate best practices engineering, housekeeping, containment, monitoring, and decontamination procedures to ensure surfaces are as clean of Pb as practicable "irrespective of measured airborne [Pb] exposure" (12).

Some DoD organizations such as the U.S. Army Special Operation Command (USASOC) have already adopted a more stringent Pb exposure standards. The USASOC Regulation 40-8 (65) requires the removal of personnel with repetitive, long-term Pb exposures (e.g. range instructors and maintenance workers) from work after two consecutive BLLs  $\geq 20 \ \mu\text{g/dL}$  or one BLL  $\geq 30 \ \mu\text{g/dL}$ . Such worker is not allowed to return to duties until BLL drops below 20  $\ \mu\text{g/dL}$ . For trainees or other personnel with shorter terms of Pb exposure the removal threshold is two consecutive BLLs  $\geq 30 \ \mu\text{g/dL}$  or one BLL  $\geq 40 \ \mu\text{g/dL}$  with return to duties after the BLL reaches levels below 30  $\ \mu\text{g/dL}$ .

#### Lead Exposure Controls on Indoor Firing Ranges

To reduce Pb exposure to levels that would not exceed permissible BLLs, OSHA lead standard directs facility owners to implement a range of control measures. Many Federal, State, and private organizations have developed and have been continuously improving various measures to satisfy OSHA requirements (13-15; 43; 47; 50). Controls can be placed in four major groups: 1) source reduction, 2) engineering controls, 3) administrative controls, housekeeping and hygiene practices, and 4) personnel protection.

### Source Reduction

Source reduction or elimination is the most effective exposure control measure. Source elimination can be achieved by using Pb-free ammunition, which has been shown to produce no airborne Pb concentrations (39). The use of jacketed ammunition, although does not completely eliminate the Pb, have shown to result in as much as 97% reduction of airborne Pb concentrations (64; 66). Some researchers, however, caution that switching to less toxic ammunition should also be accompanied with a thorough range cleaning. Otherwise, Pb dust on range surfaces from prior use are likely to contribute to elevated airborne Pb concentrations (60).

In recent years, manufactures and the military have developed several types of ammunition with reduced amount of Pb. In non-Pb primer ammunition, the Pb in the primer is substituted while the projectile is still made of Pb core but encapsulated in full metal jacket. Pb-free ammunition contains no Pb and the projectile's core is made off other materials such as tungsten or steel alloys. A special type of Pb-free ammunition is called frangible ammunition. It has the entire projectile made of soft material, usually copper alloys, which breaks into pieces on impact. Nowadays almost every manufacturer have added some type of reduced Pb ammunition to their line of products (43).

Although not required by OSHA, some indoor firing ranges have elected to use only Pb-free ammunition. This was found practical on some law enforcement and military training ranges where the owner has full control of the range and/or provides shooters with Pb-free ammunition. Owners of private or civilian ranges may be reluctant to implement such restriction as it may turn away some customers.

#### Engineering Controls

Engineering controls on indoor firing ranges consist of facility structural design aspects and various technological devices intended to reduce the exposure or to create conditions that would prevent exposure increase. Engineering controls are considered to be the second most effective means after source reduction. A health hazard evaluation conducted by NIOSH discovered a 95% reduction in air Pb concentrations in an indoor firing range following the installation of a new properly designed ventilation system (55). Inadequately designed indoor range could potentially create Pb exposures exceeding 40 times the OSHA PEL where shooters would reach the 8-hour PEL within 12 minutes of shooting (66).

Pertaining to ventilation, OSHA does not mandate system performance parameters but rather states that ventilation must be used if exposure exceeds the PEL. However, OSHA does require the range owner to assess the performance of installed ventilation system to demonstrate its effectiveness at least every 3 months. Another ventilation related provision explicitly stated in the OSHA Pb standard relates to systems that recirculate air on a range. Such systems must have a high efficiency filter, controls to monitor the concentration of Pb in the return air, and be able to bypass the recirculation system automatically if monitoring device fails (52).

Ventilation system design effective in controlling Pb exposure on IFRs has been developed and recommended by other organizations such as the NIOSH and ACGIH (3; 4). Below are some key components of the recommended ventilation system:

1) once-through (direct exhaust) ventilation system with laminar air flow of 75 ft/min at the firing line, 50 ft/min at shooters breathing zone, and 30 ft/min downrange of the firing line;

2) air supply distribution plenum with radial diffuser 10 feet behind the firing line to spread the flow evenly across the width of the shooting range;

3) 10% more air exhausted than supplied to keep negative pressure of 0.03 inches of water gage to ensure air moves downrange, away from the firing line.

Range exposure studies have shown that properly installed and operated ventilation system significantly reduces the exposure to airborne Pb. In one study, the correction of positive air pressure inside the range and creation of smoother (lamina) air flow across the firing line resulted in 62% reduction of air Pb concentration at the firing line. Creating negative air pressure also reduced the leakage of airborne Pb into adjacent rooms to below detectable levels (66).

Other engineering controls relate to the structural and operational design of a range and aim either to facilitate the performance of ventilation system or to reduce the spread of Pb dust outside the firing bay. Below are some aspects of the recommended range design:

1) 8 to 10-feet high ceilings to allow proper air flow;

2) two-door airlock between the firing bay and adjacent areas to prevent contaminated air leakage;

3) target retrieval system to prevent shooters from walking through lead contaminated areas downrange;

4) bullet trap that does not result in deformation or fragmentation of the projectile thus contributing to airborne Pb levels and surface contamination;

5) floor covered with water proof surface sealant to prevent Pb accumulation and to allow wet mopping.

#### Administrative Controls, Housekeeping and Hygiene Practices

Administrative controls encompass employee's training and education, establishing rotation schedules to reduce employee's time-weighted average exposure, and recordkeeping. Housekeeping and hygiene practices aim at minimizing Pb ingestion and prevention of Pb spread outside the facility.

If employee's exposure exceeds the PEL, the OSHA requires the employer to take measures preventing the spread of Pb dust to clean parts of the building such as break rooms, employee offices, and restrooms as well as outside the facility to personal vehicles and homes of range employees. In this case the OSHA requires the employer to:

1) ensure that food, beverages, tobacco products are not present or consumed, and that cosmetics are not applied except in designated areas;

2) provide clean change rooms with separate storage for protective and street clothing to avoid cross-contamination;

3) provide shower facilities and ensure employees shower at the end of the work shift and do not leave the workplace wearing any clothing used during work;

4) provide temperature controlled, positive pressure, filtered air supply lunchroom facilities and ensure employees wash their hands and face prior to eating, drinking, etc.; and ensure employees do not enter the lunchroom wearing work clothing unless it has been cleaned by vacuuming, down draft [AS] booth, or other cleaning method.

NIOSH scientists have recently developed a highly effective method of decontaminating the skin from Pb. A hand wiping method using a textured wipes with citric acid and isostearamidopropyl morpholine lactate (ISML) have shown a 99.9% Pb removal efficiency while washing hands with soap and water resulted in over 90% reduction of Pb of shooters hands (18).

About range cleaning, OSHA explicitly prohibits surface cleaning using compressed air. Instead, it directs to use vacuuming methods that minimize the reentry of Pb into the workplace (e.g. equipped with high-efficiency particulate arrestance (HEPA) filter). Shoveling, dry or wet sweeping is only allowed if vacuuming have been tried and found not to be effective.

Other commonly recommended practices include:

1) using wooden or plastic rakes for brass collecting (no brooms);

2) placing sticky mats at the range exits for wiping feet prior to leaving;

3) providing laundry accommodations.

#### Personnel Protection

The use of personal protective equipment (PPE) is a control measure of last resort. Although it is an effective control measure and studies have shown that respiratory protection can lower shooters BLL (21), every effort should be made to control the Pb exposure by means other than PPE. Whenever all other control measures have been implemented but failed to reduce Pb exposure to below the PEL, the OSHA requires the employer to provide workers with respirators and protective clothing to further lower employee's exposure.

In this case, the employer must establish a respiratory protection program in accordance with OSHA guidance containing provisions for employee training, medical examination, respirator selection and fit testing, repair, maintenance, and replacement. Protective clothing must consist of full-body coveralls, gloves, hats, and shoes or disposable shoe coverlets. The employer must provide all appropriate PPE to the employee at no cost. OSHA also outlines provisions for PPE repair, replacement,

laundering, and disposal, and explicitly prohibits clothing cleaning by "blowing, shaking, or any other means which disperse Pb into the air" (52).

#### **Air Showers**

In the continuous effort to reduce Pb exposure at firing ranges, several US Army IFRs recently have implemented a new control – the air shower (AS). However, ASs have never been used at indoor firing ranges before and their effectiveness in this application have never been studied. The literature search for studies about AS use in other areas produced limited results.

Most AS studies have been conducted by AS manufacturers and have not been published in peer-reviewed, scientific literature. The results were usually presented during cleanroom industry societies and conferences. Conference proceedings were not always available for review for this study. The obtained records lacked details on methods and materials and showed little statistical analysis of the reported data. The AS parameters, study designs, and analytical approaches varied among investigations, therefore did not always allow for comparison. Below is the information on ASs' design, operation, performance, and efficiency found in various sources reviewed for this study.

#### **Concept of Air Shower Operation**

The primary purpose of an AS is to remove gross particulate matter from personnel clothing. The concept of an AS is to blow high velocity air streams at employee's uniform to dislodge any particulate matter and then quickly remove the "dirty" air from the booth before the dust settles back on uniform (41). Placing AS on the border between "clean" and "dirty" environments allows an individual to clean an outer garment of undesired contaminant as he/she proceeds from one side to another. In addition to removing a contaminant of a garment, ASs also performs the function of an airlock, preventing contaminants suspended in the air from traveling from one side to the other. The AS units come in various shapes and sizes but all have the same key components:

1) an enclosure in the form of a booth or tunnel,

2) a powerful fan to create a high flow of air,

3) nozzles in walls and/or ceiling to direct airflow inside the booth, and

4) an air return vent near the bottom of the enclosure with HEPA filter to remove the contaminant from the air before it recirculates in the AS.

When an individual enters the AS and closes the door, either automatically or with a push on a button the AS activates and blows streams of air through the nozzles at high velocity toward the individual. Streams of air dislodge contaminants off the individual's outer garment while the same motor that delivered the air in the first place sucks the now dirty air through the return vent due to the close-loop system design. The cleaning cycle continues for a certain period and once it is over the individual opens the door on the other side of the AS and exists.

Depending on the size and shape of the AS, an individual may be asked to perform a certain movements during the cleaning cycle. In case of a small AS booth (Figure 2a) designed for one person, an individual usually raises hands and turns around to expose all surfaces of the garment to air jets. Long narrow AS tunnels (Figure 2b) do not require turning movements and an individual simply walks through the tunnel at normal pace. Some shorter but wider AS tunnels have rails protruding from the side walls

and require an individual to move through the tunnel in an "S"-shape pattern exposing all sides of the garment to air jets.



Figure 2. Air shower a) booth and b) tunnel.

Despite seemingly simple concept, currently there is no International or United States standard for ASs. The design and performance parameters of ASs vary from one manufacturer to another. In the literature, a brief mention of one foreign national standard was found in Japan but was not available for review in English translation (37).

### **Design and Parameters**

The lack of standardization perhaps is due to failure of industry experts to agree on optimum design and performance parameters. Nevertheless, the main parameters identified to affect ASs performance are the number and design of air nozzles, the air velocity, and the duration of the cleaning cycle period. Below is a brief description of several AS parameters and their influence on AS performance as it was found in AS studies.

#### Nozzle Air Velocity

The air velocity is considered to be the most important parameter determining AS's effectiveness. *Nozzle air velocity* is defined as a speed of air stream coming out of a nozzle measured at the face of the nozzle and expressed in feet per minute (fpm). Most AS manufacturers recommend nozzle air velocities between 4,500 and 7,500 fpm. Some AS models produce as little as 2,000 fpm while others offer as much as 9,000 fpm. Following the conception that the higher the velocity the better, some AS developers suggest that nozzles should produce air velocities of 12,000 fpm (33).

Studies have shown that AS units with higher air velocities offer more reduction of the contaminant. In a laboratory study with simulated nozzles Hirasawa et al. (26) observed a steady increase in dust removal efficiency when increasing air velocity. The results also suggested that increasing the nozzle air velocity above 4,000 fpm does not produce a relatively significant additional benefit.

The study by Ooi (51) observed similar results. A clean garment exposed to atmospheric environment for a week was put on a mannequin and placed inside a slim design AS. Application of various nozzle air velocities showed the increase in removal efficiency proportional to the increase in the velocity. The author concluded that although a 100% removal is possible as air velocities approach 6,000 fpm, the 4,900 fpm is the optimum nozzle air velocity from a practical point of view.

Instead of referencing AS's nozzle air velocity, some AS studies used a *point air velocity* measured at a certain distance (point) away from nozzles. In this case, researchers measured the velocity of an air at a distance at which it was expected to come into contact with a garment inside an AS. Although rarely used, a point air velocity
appears to be a more relevant measure of AS performance since it accounts for internal dimensions of an AS and the position of a body (garment) inside the AS.

#### Number and Design of Nozzles

Multiple points of impact during the cleaning process are believed to ensure a better garment agitation thus dislodging a greater amount of contaminant of the garment. The recommended "rule of thumb" for the amount is 20 to 26 nozzles for a single person AS booth (33). Some manufacturers produce AS units with as many as 36 nozzles, some with as few as 6 nozzles. Nozzles could be evenly distributed on walls and ceiling and directed toward the centerline. Some models have nozzles only on walls, others just on the ceiling. The effect of the number of nozzles on removal efficiency was examined by Maryuma et al. (37). The researcher concluded that increasing the number of nozzles results in better dust removal as long as the nozzles produce the same recommended nozzle air velocity.

The design of nozzles is another ASs' parameter varying significantly between different manufacturers. Most nozzles are round fittings between <sup>3</sup>/<sub>4</sub> and 1<sup>1</sup>/<sub>2</sub> inches in diameter protruding for up to 1 inch from a wall. Some nozzles are mounted flush with walls' surface. Less common nozzles design is a vertical or horizontal slot. Evaluation of ASs with slot-type nozzles showed inferior performance when compared to a round design (62). Most nozzles produce a constant flow of air, where some are design to create pulsating air jets. Shambreskis et al. (61) have found that pulsating nozzles removed 4-13% more dust particles of 0.3-5.0 µm in size than traditional nozzles. Following this discovery, the Airtech designed new AS units combining the two types of nozzles.

# Angle of Impact

Angle of impact is defined as an angle between the line of the air stream coming out of a nozzle and the surface of the garment. When air jets come in contact with a garment, the angle at which they fall on the surface of a garment is believed to affect the removal efficiency. Hirasawa et al. (26) have examined the effects of an angle of impact on removal efficiency of dust particles of various size. Simulating AS operation in the laboratory, researchers applied an air velocity of 4,000 fpm through a round nozzle for 60 seconds on the polyethtel cloth loaded with dust with a median particle size of 30 µm. The results suggested that angle of impact had effect on removal of particles  $\geq 20 \ \mu m$  in size and no effect on particles  $\leq 20 \ \mu m$ . Thus, nozzles in most ASs are pointed straight out at the 90-degree angle. However, some AS have nozzles pointed toward the bottom of the AS and others have adjustable nozzles that could be pointed in any direction.

#### Cleaning Cycle Duration

The cleaning cycle is considered the second most critical aspect of ASs' performance, although there is no agreement between AS experts on its duration. *Cleaning cycle* is the time period between the initiation of the fan and the moment an individual is allowed to leave. In some AS models it is limited to the actual garment-cleaning period, in others it also includes the time required to purge the AS booth after the cleaning is over. Purging is usually achieved by changing the airflow to slower vertical flow from the ceiling nozzles. Some AS do not have the purge function but rather allow the air flow to slowly wain away after the fan turns off. This time period some AS developers call the *dwell time*. To restrain an individual from exiting an AS prior to cycle

completion, some ASs have interlocking doors that lock when the entrance door opens and unlock once the cleaning cycle is over.

To examine the effects of cleaning duration in laboratory setting, Hirasawa et al. (26) selected the nozzle air velocity of 5,000 fpm and applied it at the 45-degree angle of impact. The results of dust removal at certain time periods varied for particles of various size but generally showed an increase in removal efficiency with time. The researches however did not suggest an optimum cleaning cycle duration.

Maruyama et al. (37) assessed the efficiency of the new AS unit combining traditional round and pulsating nozzles, a purge mode, and a built-in particle counter. While measuring the number of particles inside the AS during a continuous cleaning cycle, the researchers noted that the garment cleaning took the first 18 seconds, yet required an additional 12 seconds to purge the AS of contaminated air in order to reach the desired level of air cleanliness inside the AS. The results suggested that the cleaning cycle for this model should be around 30-40 seconds. The researchers concluded that the cleanup function is essential and that ASs' design should incorporate a particle counter to control the duration of the cleaning cycle.

Although most AS studies showed that prolonging the cleaning cycle resulted in higher removal efficiency, AS developers' goal is to keep the cleaning cycle short. The ASs with long cleaning cycles become chock points during shift changes, their use is less likely to get supported by management, and the AS protocol is more likely to get circumvented by users. Since there is no agreement on cycle duration, AS manufacturers ship their units tuned to a factory recommended setting but allow users to adjust the duration of the cleaning cycle.

## Airflow

*Airflow* is defined as a volume of air moving through an AS over a time period and usually expressed in cubic feet per minute (CFM). The airflow produced in AS primarily depends on the fan selection, but also could be affected by the size and number of air nozzles. The airflow is used in the AS manufacturing to assess how fast the air inside an AS would be purged. The greater the airflow into an AS, the faster the dirty air would be pushed out through the air return vent. However, to estimate the effect of the airflow on purging efficiency of a particular AS unit, one would also need to know the volume of the AS itself.

The air change is another parameter used by some AS developers to measure the purge efficiency. *Air change* indicates how many times the air within a defined space is replaced and usually expressed in air changers per hour (ACH). This parameter appears to be a better measure of AS's purging ability since the volume of the AS is already incorporated into it. Despite this advantage, all reviewed AS studies used the airflow instead.

The study by Maruyama et al. (37) mentioned earlier also assessed various purging modes of a new AS model. The combination of side and ceiling nozzles increased the overall airflow and purged the AS within 18 seconds. While the use of only side nozzles for AS purge required 50 seconds to reach the same drop in particle count. The study showed the benefit of using higher airflow and recommended this design of AS's purging function.

# **Application of Air Showers**

The first ASs were developed in the United States in the 1960s as a part of the cleanroom technology for the aerospace industry. The aviation industry long noticed that the dust was causing electronics to fail and were looking for ways to minimize dust presence in parts assembly areas. The purpose of ASs was to prevent the introduction of lint and dust particles on employee's outer garment into the controlled environment of a "clean room" where planes and rockets were assembled. In following years, the use of AS quickened beyond their initial place in aerospace and precision manufacturing to other dust-sensitive industries such as microelectronics, automobile painting, pharmaceutical, biomedical, optics, and food and drink production (27).

In some industries AS were put to use for purposes other than the one for which it was originally invented. The concept and operation remained the same: a worker with contaminated clothing used AS to remove contaminants prior to exiting the "dirty" site. However, instead of preventing product contamination, ASs were used to reduce occupational exposure to substances hazardous to human health.

The first AS use in the United States outside the cleanroom application found in literature was in secondary lead smelting operations in early 1980s (62). Following the enactment of OSHA lead standard of 1978, the ASs were used to satisfy the requirement in part 1910.1025(i)(4)(iv) stating employees must not enter lunchroom facilities while wearing work clothing "unless surface lead dust has been removed by vacuuming, down draft [AS] booth, or other cleaning method" (52). Since then, ASs have been employed or recommended for control of various occupational exposures such as animal allergens exposure among laboratory personnel (29), and silica exposure of construction and mining workers (24). Recently, ASs were incorporated into the design of several newly 27 built US Army indoor firing ranges to reduce Pb contamination of shooters' and range employees' garments.

#### Efficiency and Effectiveness of Air Showers

In the literature, the terms "efficiency" and "effectiveness" are used in AS studies interchangeably although with a different connotation. When using the term "efficiency", most authors referred to the fraction of particulate matter removed by an AS from a garment. The use of term "effectiveness" appeared to have a broader meaning encompassing several aspects of AS employment. Since there is no AS standards, the definitions of these terms were not found in literature. For the purpose of clarity, the two terms are delineated here.

*Efficiency* is a term describing how well an AS performs its primary task which is removing particulate matter of a garment. The two main measures of an AS' efficiency are the fraction of particulate matter removed and the time it took to achieve it.

*Effectiveness* is a term for usefulness of an AS in a particular application and answers the question whether an AS achieved the goal for which it was employed (e.g. did the employment of the AS decrease the amount of particulate matter introduced by an employee into a clean room). The effectiveness also encompasses other aspects of AS employment such as practicality.

As was discussed earlier, the efficiency of ASs depends on several parameters of its design and performance. Thus, the goal of most AS studies was to test various AS units to identify the design that would offer the highest reduction of contaminants in a shortest time possible. However, AS effectiveness also depends on the type of garment used and characteristics of the contaminant, specifically the particle size. While most

studies focused on a certain aspect of AS efficiency, several researchers examined the overall effectiveness of an AS in a particular application. Having different objectives in mind, different researchers selected different assessment approaches, materials, and methods for their study and therefore their findings do not always support each other.

#### Contamination Control Application

In a laboratory setting, Hirasawa et al. (26) examined the effects of select air velocities on removal of particles of various size. Clean polyethtel garments exposed to an ordinary office environment for 24 hours were used to study effects on particles  $\leq 5$  µm in size. The results showed insignificant difference in particle counts between select velocities and the control group. The findings lead researchers to conclude that ASs are inefficient in removing particles  $\leq 5$  µm.

Contrary to findings in Hirasawa et al. study, the study by Shambreskis et al. (61) found ASs very effective for the same size range particles. The study used an unspecified clothing textile loaded with a test dust to compare the performance of two types of nozzles. While testing the same continuous flow nozzle, air velocity, and cycle duration used in Hirasawa study, the researchers in Shambreskis et al. study observed a higher removal efficiency of particles  $\leq 5 \ \mu m$  ranging from 53 to 65 %. The contradicting results of the two studies perhaps are due to difference in garment material and test dust used during these tests.

To examine the effects of garment on AS removal efficiency, Hoenig (40) used six types of suites commonly used in cleanroom industry. The researcher loaded select areas on garments with fine Arizona road dust using gravity settling method and exposed them in the AS booth with round nozzles producing air velocity of 8,000 fpm. Comparing particle counts on garments before and after the 60-second cleaning cycle, the researcher determined that the removal efficiency for all garments ranged from 70 to 85% (no particle size reference).

Professor Hoenig repeated the tests with the same types of garment but using a different cleaning procedure. An individual wearing a test garment was exposed to a standard cleaning procedure inside the AS with a hose attached to the garment at the hipline and blowing low-pressure air inside the suite. The resulting particle removal efficiency rose to 90-95% for all garments. The researcher suggested that some dust was trapped in seams and creases of the garment and that the air flowing inside the suite exited through the fabric removing some of the trapped dust.

Ernst (17), however, observed significantly lower removal efficiencies while comparing the performance of three different types of AS: booth, straight tunnel, and 90degree tunnel all equipped with nozzles producing low air velocities of 880, 600, and 790 fpm accordingly. The cleaning cycle period in the AS booth was 8-10 seconds and about 5 seconds for both tunnels. The results showed an average removal efficiency of 22, 20, and 13% for the booth, straight tunnel, and 90-degree tunnel accordingly, suggesting that the booth design was more efficient in removing particle from various environments.

Following the facility upgrade with new types of ASs, Ernst repeated the study assessing the efficiency of the three new AS tunnels: straight, 90-degree turn, and the Uturn tunnel. The results showed the removal efficiency of 19, 14, and 9% accordingly. Ernst also compared the effect of three cleaning cycle protocols recommended by different AS manufacturers. 1) 25-second and 2) 45-second cleaning cycles with standing in the middle of a tunnel while raising and lowering arms, and 3) walk-through at a

normal pace with no effort for extra movement such as turning or lifting arms. The results showed a 15, 19, and 12% removal efficiency accordingly, suggesting the longer cleaning cycle produces a higher removal efficiency.

Significantly different removal efficiencies observed in the above studies could be explained with differences in ASs' major design and operation parameters such as nozzle air velocity and cleaning cycle duration. Researchers also used different analytical methods potentially measuring particles not of the same size range. Again, since the above mentioned studies had different goals and objectives, a "one-to-one" comparison of the results would be inappropriate.

Instead of evaluating ASs efficiency in removing particles off a garment, Austin et al. (40) assessed ASs' overall effectiveness as a contamination control measure in a cleanroom application. The study examined two AS units incorporated into a cleanroom design: one AS unit connecting the general area with the garment change room and another AS located between the change room and the actual clean room. The researcher measured particle counts inside the clean room as employees were entering the room during certain AS use conditions:

Condition 1 – Both ASs off Condition 2 – First AS on, second off Condition 3 – Second AS on, first off Condition 4 – Both ASs on

Twenty employees processed into the clean room for each of the four AS use conditions. The differences in particle counts between conditions "Both off" and "Both on" showed a significant reduction in contamination as a result of AS use: 80% for particles  $\geq 0.5 \ \mu m$ , 95% for particles  $\geq 1.0 \ \mu m$ , and 61% for particles  $\geq 5.0 \ \mu m$ .

## Exposure Control Application

The ASs have been mostly used for contamination control in dust-sensitive industries. Their employment to control occupational exposures has been limited and therefore has not been well-studied or documented. The literature search found limited amount of studies for review.

A report produced by the Radian Corporation in 1983 under a contract from NIOSH presents an assessment of ASs effectiveness as an employee exposure control in secondary Pb smelters. The goal of the Simonson et al. (62) study was to evaluate two ASs installed at two smelting facilities on their efficiency in removing surface Pb dust from work clothing. The 2"x 3" swatches of two types of clothing worn by employees were each loaded with 40 mg of Pb oxide dust and several swatches were analyzed to verify the load. The size of Pb dust particles was not examined. Loaded and blank swatches were attached to the shoulders of the experimenter's jacket. The individual then walked into the AS and followed standard cleaning procedures. Cleaning cycle periods in both ASs were approximately 20 seconds.

The results showed that the AS with round nozzles producing average air velocities of 5,500 fpm removed 22-72% of Pb oxide dust while the other AS with horizontal slots producing average air velocities of 4,200 fpm removed 5-28% of Pb. During the swatch analysis, the blank swatches worn inside ASs during the test also showed Pb presence suggesting that Pb contamination of clean parts of a clothing can occur during an AS operation.

Simonson et al. also noted that the amount of Pb dust removed in ASs differed between the two fabrics used in the study; furthermore, some Pb oxide dust was blown

through one of the fabrics – the lighter weight shirting fabric. To further examine this finding, the researchers repeated the study in the laboratory using a simulated AS nozzle.

Simonson et al. repeated the study using three different types of fabric: 1) a plainweave shirting fabric, 2) a twill-weave coverall fabric, and 3) a non-woven disposable fabric (Tyvek<sup>®</sup>). Pre-weighted 50-mm swatches cut from the three fabrics were loaded with Pb oxide dust. The all swatches were reweighted to determine the net load. Loaded swatches were then pinned to a board. A 47-mm, 0.45 micron pore-size filter was placed underneath some swatches of every fabric type to determine the amount of Pb breakthrough. The AS nozzle was simulated with a glass tube connected to a source of compressed nitrogen gas and the air velocity was adjusted to 5,500 fpm to match that from the field study. The swatches were dusted off using back-and-forth movement of the nozzle repeated at distances of 1-10 inches from the nozzle. The simulated AS cleaning cycle was 20 seconds.

The results showed a 23% reduction for the twill-weave coverall fabric, 48% reduction for the plain-weave shirting, and 69% reduction for non-woven Tyvek<sup>®</sup> fabric. The lead breakthrough across fabric was registered for all three types of material tested and varied from 0.2 to 1.4% of the Pb load on the corresponding swatch. The results of this study suggest that 1) different fabrics have different "holding" properties for dust and affect the amount of particulate that can be removed by an AS, and 2) the application of high velocity air streams can result in the breakthrough of a contaminant across porous fabrics including tight-weave materials.

Norman (40) performed another evaluation of ASs effectiveness as an occupational exposure control measure. The researcher examined an AS tunnel installed between the work area and the lunchroom at a secondary Pb smelting facility. The AS was described as 32-foot long downdraft tunnel with adjustable nozzles producing 4,000 fpm air velocity. The researcher performed several tests aimed to assess the AS efficiency in removing Pb dust off employees work clothing and the overall effectiveness in preventing the spread of Pb from the work area into the lunchroom. The report provided little detail about materials and methods used in the study.

The wipe samples taking from work clothing of five employees before and after AS use showed a 60% removal of Pb averaged for all samples. In the second test, the researcher applied 50 grams of Pb [oxide] to the 2"x 2" pieces of work uniform material: cotton cloth, 65% / 35% [polyester/cotton], and nylon. The material swatches were carried through the AS and then soaked in distilled water to recover and measure the remaining Pb. The fraction of Pb removed from three swatches was 46, 62, and 74% accordingly. The third test consisted of wipe samples collected on tables and chairs in the lunchroom on days with and without use of the AS. The results showed an average of 27% reduction of Pb contamination on the day when the AS was used. Although the results suggest that the examined AS was effective in decreasing Pb exposure, the researcher did not make such conclusion.

Another study examining AS application to control occupational exposure was published in the Scandinavian Journal of Laboratory Animal Science in 2008. Krohn et al. (29) conducted a study to examine the benefit of AS use to reduce the exposure to animal allergens among laboratory employees. The aim of the study was to evaluate the effects of clothing type, air velocity, and cleaning cycle duration on the efficiency of allergen removal by ASs.

The 2"x 2" swatches of cotton lab coat and polyester suit were loaded with animal allergen powder collected from mice bedding (no particle size reference). Loaded swatches were attached to a holder, placed inside the AS, and exposed to various air velocities and cleaning cycle periods. Enzyme-linked immunosorbent assay was used to estimate the amount of allergen on swatches before and after AS application. The results showed that 1) polyester material retained [approximately 80%] less allergens than cotton (p < 0.001) and 2) the increase in nozzle air velocity and cleaning cycle duration were correlated with the observed reduction in allergens ( $R^2 = 0.115$ , p = 0.023 for air velocities and  $R^2 = 0.221$ , p = 0.004 for cleaning cycle).

The second part of study examined the allergen reduction produced by the AS with the manufacturer recommended 5,900 fpm air velocity. Allergen powder was applied to select areas of Tyvek<sup>®</sup> Pro-Tech Classic suit worn by a volunteer. Samples were collected before and after AS application, and then after the garment was removed by the volunteer. The results showed that AS's efficiency of allergen removal was 98.4% at the shoulder level and 87.4% at the thigh level. The removal of the garment resulted in additional reduction of allergen by 1.4 and 4.0% accordingly. The latter observation indicated that the AS removed the vast majority of loosely attached allergens and that garment handling following the AS application would not release a significant amount of allergens.

The results of Krohn et al. research once again demonstrated the relationship between major parameters of performance and efficiency of an AS described in cleanroom industry studies. The study also supported the findings of other evaluations suggesting that rough design fabrics such as cotton retain more contaminants that a slick materials such as polyester. High reduction efficiency demonstrated in this study could not be compared to other studies due to no particle size reference. Nevertheless, Krohn et al. obtained statistically significant data allowing researchers to conclude that the examined AS was an effective control for allergen exposure among animal laboratory employees.

Despite a general consensus among cleanroom industry experts that ASs exhibit some efficiency in removing particulate matter of a garment, there is lack of agreement on the overall effectiveness of AS in contamination control in cleanroom application. Although the focus of this study is not on AS application in the cleanroom industry, it is important to discuss the controversy about AS effectiveness. With a few exceptions, the body of knowledge about ASs consists of studies assessing its application in the cleanroom industry; thus, conclusion drawn in those studies could be viewed as applicable to AS use in general. Such assumptions, however, could be erroneous since the criteria of AS effectiveness in other applications could be different.

### The Air Shower Controversy

The controversy around the effectiveness of ASs has "plagued the design and operation of cleanrooms for many years" (68). The two sides often debating on this subject are 1) AS developers and suppliers, and 2) representatives of cleanroom industry

and academia. Several articles and white papers have been published on this subject leading to "Great Air Shower Debate" session during the CLEANROOMS'95 West conference (33). The proceedings of this debate were not available for review, nonetheless, below is the essence of the issue as it was found in the literature.

The position of AS supporters is based on the results of AS studies, which were mostly performed by AS developers and supplies. The results of the studies discussed earlier demonstrated that although depended on many aspects such as AS design and the type of garments used by employees, ASs do exhibit some removal efficiency and effectiveness as a contamination control measure. Their opponents, however, dispute the significance of AS effects compared to other contamination control efforts and question the practicality of AS employment.

Whyte et al. (69) disputed the notion that ASs play a significant role in controlling particle contamination in a clean room. Whyte et al. reasoned that cleanroom laundering practices result in very clean garments and therefore the garments are not the greatest source of contamination in the clean room. Thus, the ASs' particle removal efficiency would not produce any benefit when proper garment laundering and use protocols have been followed.

In the study, the researchers assessed the significance of AS use by measuring the dispersion of particles of various size coming from individuals wearing clean garments. The volunteers wore clean tightly-woven polyester coveralls that have been washed in the laundry and tested for compliance with the garment cleanness standard. After donning the garment, a volunteer either 1) walked around the "dirty" room for 45 seconds or 2)

entered the AS and walked up and down five times for a total of 45 seconds as well. The volunteer then entered a particle dispersion chamber. While in the chamber, the volunteer exercised to the beat of a metronome by marking time with his feet and moving arms across his chest. The particles dispersed were counted continuously at the chamber's exhaust vent and sized as  $\geq 0.5$ ,  $\geq 3$ ,  $\geq 5$ , and  $\geq 20 \ \mu\text{m}$ . The comparison of particle dispersal rates failed to show that AS had any effect (p > 0.2) on particle generation.

In the second part of the study, Whyte et al. compared particle counts inside the clean room when AS was used and when it was by-passed. For 16 days, regular clean room employees perform their daily duties it the cleanroom setting using AS only on alternate days. Particle counts were measured periodically throughout the work shift using two particle counters. The statistical analysis again failed to reveal any difference (p > 0.2) between particle counts on days when AS was used and when it was by-passed. The researchers also made a notion that the substantial reduction in particle counts observed in other studies are largely due to the AS's function as an airlock rather than a garment cleaning.

It is important to note, however, that the AS used in this study was not of a traditional design. The cleaning action in the AS was not provided by air jets from nozzles but rather by an airflow coming from a large supply vent on the bottom. The AS supporter reasonably argue that the results of this study should not be applied to all ASs in general since the AS design did not represent the best practices recommended by the AS developers (33)

Nevertheless, supporting evidence of White's notion about airlock function could be found in other studies. Maruyama et al. (37) examined ASs' garment cleaning efficiency by comparing particle counts inside an AS. Volunteers wore 1) clean garment, 2) garment exposed to a dirty environment for one hour, and 3) three types of general work clothing. No significant difference in particle count increase was registered between the types of the garment worn. Thus, the researcher concluded that the increase in particle count inside the AS did not originate from the outfit but was mostly due to the door opening and introduction of dirty air from the adjacent garment changing room

Another critical view of AS effectiveness was expressed by Wadkins et al. (68). The researches argued that data supporting the effectiveness of ASs should be provided by sources with no vested interest in selling these devices. The authors pointed out that the high removal efficiency shown is some studies are for particulate matter of  $\geq 5 \ \mu m$  in size, which is not the greatest concern in the microelectronics industry.

Wadkins et al. mentioned several AS studies pointing to erratic results and noting that there was no predictable performance, especially related to the recommended cleaning cycle durations. Although recognizing the mounting body of knowledge showing that AS do remove some particulate matter, Wadkins et al. argued that the time it takes to achieve desirable results makes ASs application impractical. To support their cause, the authors in their own study examined the cleaning cycle duration required to reach particle baseline levels in two types of ASs. The results showed that even when an individual wore a clean garment, it took an average of 30-40 seconds to reach the baseline. The time required for dirty garment was 40-50 seconds. The authors notion that such time requirements were greater than the management of a cleanroom would support and much greater than the employees would tolerate. However, the ASs used in this study produced air velocity of 1,000 fpm measured at one foot away from the nozzles, which is

more than twice below the recommended values. In addition, the study does not specify the airflow parameters, which were shown to be critical to AS purging efficiency and could potentially explain prolonged cleaning cycle durations.

Wadkins et al. also examined the two ASs on their removal efficiency of particles  $>0.3 \ \mu m$  in size. The surface of "clean" and "dirty" garments was evaluated for particulate matter using two commonly used methods. After garments were worn and exposed in the ASs, each garment was again examined for particulate matter using both methods. The comparison of averaged values before and after AS exposure showed the increase of particle concentration suggesting that ASs instead of cleaning the garments further contaminated them. Since the result was rather unexpected, the researchers did not incorporate a control group in their study to assess the effects of garment gowning on its contamination. Thus, the observed increase in contamination could not be positively attributed to AS exposure. Despite identifying this flaw in their study, the researchers include in the conclusion to their study that "clean garments become dirtier when exposed to air showers rather than being cleaned" (68).

#### Air Shower Summary

The review of available literature about AS performance and effectiveness in various applications lead to several conclusions applicable to this study:

1). ASs exhibit some efficiency as a garment-cleaning device.

2). ASs are efficient in removing particles  $\geq 5 \ \mu m$  in size while the effect on particles  $\leq 5 \ \mu m$  remains uncertain.

3). Air velocity is a major parameter of performance affecting AS's efficiency.

4). High velocity air streams produced by ASs may push a contaminant through garment.

5). Various types of clothing retain different amount of contaminant.

6). Each AS model requires testing to measure its efficiency and effectiveness in each application.

## Air Showers at Indoor Firing Ranges

In recent years, several newly built US Army IFRs have incorporated ASs to its design, although the basis for AS use at indoor ranges is unknown. The literature search for this study failed to find any prior studies assessing ASs' effectiveness in this particular application. Perhaps ASs' use on IFRs was based on a proposition that ASs would remove Pb particles off range employees' or shooters' garment as they exit the range, thus reducing lead ingestion, minimizing the spread of Pb outside the range, and preventing take-home exposure of family members. As was discussed earlier, the review of studies assessing ASs' effectiveness in other applications produced some evidence suggesting that ASs' use at IFRs is justifiable.

A brief examination of an AS installed on one of the US Army IFRs conducted by an industrial hygienist from the US Army Public Health Command indicated a potential flaw in the AS design (6). The smoke test performed inside the AS during the cleaning cycle discovered air movement patterns that could potentially increase the risk of Pb inhalation during AS use. Air currents brought the smoke up toward the AS ceiling and formed eddies at the corners. The observations indicated the air movement inside the AS could potentially blow the Pb toward individual's head and remain in the breathing zone, thus increasing the risk of Pb inhalation and facial skin and hair contamination. The investigator identified the need for AS design modification and recommended further evaluation of the AS's effectiveness as an occupational exposure control at indoor firing ranges.

### **STUDY OVERVIEW**

## **Goals and Objectives**

The goal of this research was to evaluate the efficiency of AS in removing Pb contamination from Army Combat Uniform (ACU).

The objectives of this research were:

- 1) Develop a method of loading uniform samples with Pb-containing GSR.
- Assess the effect of various air velocities and angles of impact on Pb removal from ACU.
- Examine the effects of various air velocities on the breakthrough of Pb through ACU during AS application.

## Hypotheses and Specific Aims

<u>**Hypothesis #1**</u>. Exposure of ACU swatches loaded with Pb to high air velocities will result in  $\geq$  50% reduction in Pb.

**Specific Aim #1**. Determine the percent reduction by comparing Pb mass on ACU swatches before and after application of point air velocities of 4,100 and 6,900 fpm at 0, 45, and 90-degree angles of impact.

**<u>Hypothesis #2</u>**. Exposure of ACU swatches loaded with Pb from GSR to high air velocities will result in the breakthrough of Pb through the fabric.

**Specific Aim #2**. Measure Pb mass pushed through ACU swatches by point air velocities of 4,100 and 6,900 fpm at 0, 45, and 90-degree angles of impact.

<u>**Hypothesis #3**</u>. Exposure of ACU swatches loaded with Pb to low air velocity will result in  $\geq$  50% reduction in Pb without causing the breakthrough of Pb through the fabric.

**Specific Aim #3**. Determine the percent reduction by comparing Pb mass on ACU swatches before and after application of point air velocity of 1,800 fpm at 0, 45, and 90-degree angles of impact.

**Specific Aim #4**. Measure Pb mass pushed through ACU swatches by point air velocity of 1,800 fpm at 0, 45, and 90-degree angles of impact.

## **Study Design**

The overall study design involves exposing ACU swatches loaded with Pbcontaining GSR to the cleaning action of an AS in order to determine the Pb removal efficiency of the device.

The study has three phases:

1) Loading ACU swatches with Pb.

2) The pilot.

3) The main study.

Phase 1. Loading ACU swatches with Pb.

During this phase, the investigators load ACU swatches with Pb-containing GSR. The method of loading is similar to a mechanism of GSR deposition to clothing that takes place at IFRs. The loading takes place on a military firing range on Aberdeen Proving Ground, MD. The investigators use a military-grade automatic weapon to fire Pbcontaining ammunition inside an environmental chamber. The resulting GSR is allowed to settle on ACU swatches placed inside the chamber. At the end of the settling period, the researches randomly select several swatches for Pb analysis to measure the load. The arithmetic mean of Pb mass on the selected swatches serves as the Pb load value for the remaining swatches. Chapter 2 of this manuscript describes the details of the developed loading procedure.

## <u>Phase 2</u>. The pilot.

The investigators conduct the pilot to obtain statistical parameters that would allow determination of the minimum sample size necessary to achieve at least 80% power at  $\alpha$ =0.05 in the main study. The pilot takes place in the same AS and in the same manner as the main study but has a limited scope. The investigators test three methods of capturing Pb breakthrough to select the most appropriate technique for the main study. In addition, the investigators assess whether the initial Pb load on ACU swatches is sufficient to produce subsequent Pb breakthrough amounts within limits of quantification of selected analytical methods. Chapter 3 of this manuscript provides further details on methods and results of the pilot and its effects on the main study design.

#### <u>Phase 3</u>. The main study.

The main study takes place at an IFR on Fort Bragg, NC. The range has an AS situated between the firing bay and the lobby. The investigators operate the AS at manufacture settings; no adjustment of AS performance parameters are made. The investigators attach Pb-loaded ACU swatches one at a time to a test assembly mounted on

a stand placed inside the AS. The investigators activate the AS exposing the swatch to the full cleaning cycle period. At the end of the cycle, the investigators collect the swatch and the Pb breakthrough for analysis. Since the AS is blowing air at a constant nozzle air velocity, to expose ACU swatches to point air velocities of interest the investigators are placing the stand inside the AS at certain distances away from the nozzle-bearing wall to achieve desired air velocities. All samples are delivered for analysis to a certified lab. The investigators use the amounts of Pb mass on swatches before and after AS application to determine the removal efficiency of the device. Chapter 4 of this manuscript provides further details on methods and results of the main study.

The study design does not involve an intentional exposure of human subjects to Pb. To prevent accidental exposure during sample handling, the investigators wear PPE consisting of disposable full coveralls, shoe covers, and N95 mask. All materials potentially contaminated with Pb are marked and disposed in accordance with federal, state and local regulations.

#### SIGNIFICANCE

This research is the first to assess the efficiency of ASs in removing GSR-specific Pb particles from ACU. Examining the hypotheses of this study would provide some basic information about ASs ability to reduce Pb contamination on ACU resulting from activities on IFRs. The results of this study could impact the use of ASs at IFRs by identifying AS parameter settings that offer a better removal of Pb. This information could also help AS developers to select the parameter setting best suited for application at indoor range, therefore decreasing the exposure to Pb among range employees and users.

If this study demonstrates high efficiency of ASs in Pb removal, this engineering control device could become widely employed on all indoor ranges with Pb exposure.

This study is the first to examine a potential breakthrough of Pb-containing GSR particles through ACU as a result of AS use. This information could help public health professionals to identify potentially hazardous exposure. The extent of the breakthrough could also signal whether ACU is appropriate protective clothing for use with ASs, which could lead to an Army-wide change in policies on the use of IFRs.

This study will also develop a method of loading samples with a GSR-specific Pb particles. The method closely resembling the mechanism of clothing contamination with Pb taking place on IFRs could open new research opportunities in occupational exposure studies.

# CHAPTER 2: Loading Army Combat Uniform Swatches with Pbcontaining Gunshot Residue

## ABSTRACT

This study is the first to load fabric swatches with a desired amount of a gunshotresidue-specific lead in the order of micrograms per square centimeter. Swatches of Army Combat Uniform material loaded with lead in the range of 50-100  $\mu$ g were needed for a follow-on study. The loading procedure developed in this study entailed firing of a lead-containing ammunition inside a sealed chamber and allowing the gunshot residue to settle on swatches placed inside the chamber. At the end of the 7-hour settling period, the investigators collected several swatches for mass analysis to estimate the load of lead. The arithmetic mean of lead mass on analyzed swatches served as the lead load value for the remaining swatches. In six separate loading events, the lead load ranged from 67.6 to 111.7  $\mu$ g with a mean of 88.2  $\mu$ g/swatch. The relative standard error for the reported means was from 0.84 to 2.93% with an average of 1.72%. The relative standard deviation for the lead mass on swatches ranged from 3.49 to 10.16% with an average of 6.18%. The loading procedure offers an accurate and reproducible method of loading fabric swatches with small amounts of lead originated from gunshot residue.

#### **INTRODUCTION**

Lead (Pb) exposure at indoor firing ranges (IFRs) remains to be an issue (5). Firing Pb-containing ammunition produces airborne Pb particles creating an inhalation hazard. Contamination of hands and clothing contributes to Pb ingestion and spreads the Pb outside firing ranges creating a risk of Pb "take-home exposure" for family members (5; 25). In recent years, several US Army IFRs have implemented a new control measure

– the air shower (AS) – to reduce Pb contamination on clothing of service members and range employees.

ASs are booth- or tunnel-type enclosures designed to remove particulate matter off a garment by applying high velocity air streams. These devices have been widely used for contamination control purposes in various dust sensitive industries such as microelectronics, automobile painting, pharmaceutical, biomedical, optics, and food and drink (27). To a lesser extent, ASs have been used or recommended for control of various occupational exposures such as Pb exposure in secondary Pb smelters (62), animal allergens exposure among laboratory personnel (29), and silica exposure of construction and mining workers (24). A number of studies evaluated ASs' ability to remove certain reference materials loaded on several types of garments. The size of a particle and type of a garment fabric have been shown to affect the removal efficiency of ASs (26; 40). However, the efficiency of ASs in removing Pb particles with unique morphology and size found in a gunshot residue (GSR) on IFRs, as well as the effects of particle retention by garments used on US Army's IFRs has not been examined.

The Pb-containing particles in GSR have unique characteristics and differ from referenced materials used in previous studies in terms of shape, size, and density. Molten at high temperatures during combustion of ammunition priming compounds (9), most GSR particles are spheroidal with a smooth, fuzzy, or scaly surface (58) and represent various combinations of Pb, antimony (Sb), and barium (Ba) with estimated particle density ranging between 7.22 and 11.34 g/cm<sup>3</sup> (7). By count and by mass, anywhere between 60 and 95% of Pb-containing GSR particles are <4  $\mu$ m in size (8; 10; 30; 38).

Different types and brands of ammunition produce different amounts and size distributions of Pb-containing GSR particles (9).

Contamination of clothing with Pb on IFRs occurs in several ways. During firearm discharge, kinetic energy of combustion propels GSR through openings in the weapon (e.g. muzzle and ejection port) and deposits Pb particles on shooter's clothing in the close proximity to the weapon (8; 56). As GSR spreads throughout the range and settles with time, Pb particles attach to clothing of anyone present on the range (9). Physical contact with contaminated surfaces when a shooter fires from kneeling or prone positions can also contribute to clothing contamination. The prevalence of one mechanism of contamination over another, as well as the quantitative description of Pb contamination on clothing on IFRs have not been found in the literature reviewed for the study. A number of studies measured Pb on various surfaces inside and outside IFRs and qualitatively examined Pb contamination of shooters' and range employees' clothing to assess the adequacy of hygiene and housekeeping practices. The results of these evaluations indicate that the degree of clothing contamination at IFRs could be in the order of micrograms per square centimeter (54; 55).

Prior studies used various sample loading techniques and amounts of reference materials to examine the efficiency of ASs. Most studies used the Arizona test dust or Japanese Industrial Standard (JIS) Z8901 dust loaded on garment samples in the order of milligrams per square centimeter using a gravitational settling method (40; 61). Some studies exposed clean garments for hours or days to various environments such as outdoors, offices, metal shops, production areas, smoking rooms, etc. to accumulate dust

particles present in those settings (17; 37; 51). The amount of load in these studies was measured with various particle-counting techniques.

The literature review for this study found one evaluation of AS efficiency that used Pb as a reference material. Simonson and Mecham loaded fabric samples by distributing Pb oxide dust on the surface of a swatch with a cotton swab. The resulting load was measured with a gravimetric technique and averaged at 1.4 mg of Pb per square centimeter of the swatch surface (62).

The purpose of this study was to load Army Combat Uniform (ACU) swatches with Pb-containing GSR particles using the gravity settling mechanism of clothing contamination on IFRs. The target range for the Pb load was 50-100  $\mu$ g of Pb per a 9.6cm<sup>2</sup> ACU swatch. GSR-loaded swatches were needed for a follow-on study of ASs' efficiency.

#### **METHODS**

The overall design of the developed loading procedure entailed firing a Pbcontaining ammunition inside a sealed chamber and allowing the produced GSR to settle on ACU swatches placed inside the chamber. At the end of the settling period, the investigators randomly sampled ACU swatches for Pb mass analysis to estimate the load. The arithmetic mean of Pb mass on analyzed swatches served as the Pb load value for the remaining swatches.

## **ACU Swatches Design**

The design of ACU swatches was dictated by the requirements of the subsequent study. To prepare ACU swatches, the investigators used a permethrin treated 50% cotton, 50% nylon fabric meeting the requirements of MIL-STD-44436 (Class 8). A new ACU coat (lot number SECS-0028, UNICOR, Seagoville, TX) was stripped into pieces, machine washed and dried once. The investigators wrapped the fabric around a 1½-inch plastic flanged tailpiece washer (part #50879K, Keeney Manufacturing Co., Newington, CT) and fixed it with a 3-mm wide 18-lb rated zip tie (item #366601, Craft Smart by Michaels Stores Inc., Irving, TX). The excess material was cut off along the edge of the zip tie with a precision knife. The parts of fabric with holes from seams or with other visible damage were excluded from swatch preparation. The resulting ACU swatch was approximately 45 mm in diameter with the exposed surface of 35 mm in diameter and an area of 9.6 cm<sup>2</sup>. Figure 3 illustrates the steps of ACU swatch preparation.



Figure 3. Steps of ACU swatch preparation.

Sixteen ACU swatches were arranged in a 4x4 manner on a cardboard as depicted on Figure 4. Swatches' positions on each board were identified with a number between 1 and 16. Prior to placing swatches on a board, the investigators identified positions of four random swatches to be collected for analysis at the end of the loading event to estimate the Pb load. The random numbers were generated using the RANDBETWEEN function in MS Excel<sup>®</sup> and the positions of selected swatches were marked on a board. Once swatches were placed on a board, the investigators secured the swatches to the board with a <sup>1</sup>/<sub>2</sub>-in (12 mm) wide PVC electrical tape (Gardner Bender, Milwaukee, WI). Each board was marked with a unique identification number. Each board with swatches was kept inside a two-inch tall cake box for storage and transportation.



Figure 4. Arrangement of ACU swatches on a loading board.

## Weapon and Ammunition Used in the Loading Procedure

The study used one Machine Gun, 7.62mm, M240 (FN Manufacturing LLC, Columbia, SC) – a belt-fed, gas-operated medium machine gun firing the 7.62×51mm NATO cartridge. The weapon selection for this study was driven by the necessity of firing it remotely in order to avoid Pb exposure of the investigators. Selecting a belt-fed over a magazine-fed weapon offered an ability to fire any number of rounds without reloading the weapon. The design of the M240 machine gun also allowed to remediate weapon malfunctions remotely if such would arise and to continue firing without restarting the procedure.

The investigators used CTG M80 NATO BALL ammunition (lot number LC-03B204-003, LCAAP, Independence, MO) containing Pb in both the primer and projectile. The primer mix FA956 in this ammunition contained Pb styphnate 37% by weight. The bullet consisted of a 114.5-grain (7.4 g) Pb core covered by bi-metallic full jacket. The type of ammunition selected for the study were based on the results of a previous study. In the ammunition comparison test conducted by the Aberdeen Test Center (ATC) in the same chamber, the M80 ammunition produced the highest air Pb concentrations compared to other types of 7.62-mm ammunition.

#### Loading Chamber Setup

The loading of ACU swatches took place inside the Military Operations in Urban Terrain (MOUT) Chamber (Figure 5) belonging to the US Army Aberdeen Test Center (ATC) and located on an outdoor small arms firing range on Aberdeen Proving Ground, MD. The chamber represented a metal frame container with walls and ceiling covered with plastic sheeting. The internal W x L x H dimensions of the chamber were 98 x 110 x

98 inches respectively. In the center of the chamber, a 4-foot tall stand with a weapon mount was permanently fixed to the metal floor. An electrical box outside the chamber allowed control of light fixtures and power outlets inside the chamber. Two side and one rear windows ca. 20 x 20in covered with removable metal sheeting allowed the introduction of analytical instrument probes, hoses, etc. The chamber also had a front window ca. 30 x 50 inches allowing a fired projectile a clear path to leave the chamber.



Figure 5. The loading chamber.

The machine gun was mounted on the stand in the center of the chamber and a lanyard attached to the weapon's trigger was routed through the back window to allow remote firing from outside the chamber. A triangle-shaped wooden block was placed underneath the weapon breach to deflect the expended brass to the right side of the weapon during the firing. Rubber mats were placed on the floor on the right side of the weapon to soften the brass landing and localize brass accumulation, thus preventing brass interference with ACU swatches.

The boards with ACU swatches were placed inside the loading chamber nine inches off the floor behind the machine gun along the back wall of the chamber. A maximum of seven boards were placed in the chamber during a loading procedure. Boards' loading locations in the chamber were numbered 1 thru 7. Figure 6 depicts the placement of the boards in the chamber.



Figure 6. The back part of the loading chamber with loading boards placed along the back wall.

Two 8-inch in diameter office fans model LF-8TB (Shanghai Limach

Manufacturing Co. Ltd, Shanghai, China) were placed on the chamber's floor, one in

each of the two front corners (Figure 7). The fans were directed upward and toward the center of the chamber and were used during the firing to thoroughly mix produced GSR in the chamber. The fans were plugged into power outlets and were controlled from outside the chamber. The front window facing the weapon was covered with a cardboard to contain the GSR exiting from the weapon's muzzle. At the same time, the cardboard allowed the fired projectiles to penetrate it and exit the chamber intact. All other openings in the chamber were taped to trap the GSR inside the chamber and to limit the interference from the outside environment. The overall setup of the chamber is depicted on Figure 8.



Figure 7. The front part of the loading chamber with fans and front window.



Figure 8. The overall setup of the loading chamber.

## Loading, Collection, Transportation, and Analysis of ACU Swatches

## Theoretical Estimations

The amount of ammunition required for the study was determined using the data from ATC's ammunition comparison test and the estimated air Pb concentration necessary to produce the target load of 50-100 µg of Pb mass per ACU swatch. Assuming that an entire amount of Pb in the air settles over time, the Pb mass  $(m_{Pb})$  that would settle on each swatch can be calculated by multiplying the air Pb concentration  $(C_{Pb})$ , the height  $(h_a)$  of the air column between the ceiling of the chamber and the ACU swatches, and the surface area  $(A_s)$  of a swatch. Thus:

$$m_{Pb} = C_{Pb} \times h_a \times A_s \tag{1}$$

When the above assumption does not hold and only some amount of Pb in the air settles over a time period, a fraction  $(f_{sd})$  of Pb mass in the air estimated to settled has to be factored into formula [1], therefore:

$$m_{Pb} = C_{Pb} \times h_a \times A_s \times f_{sd} \tag{2}$$

In this study, the Pb mass on a swatch is a desired value and the goal is to calculate the concentration of Pb in the air necessary to produce the desired Pb mass on swatches, i.e. Pb load. Therefore, the formula [2] was solved for  $C_{Pb}$ :

$$C_{Pb} = \frac{m_{Pb}}{h_a \times A_s \times f_{sd}}$$
[3]

where:  $C_{Pb}$  – concentration of Pb in the air,  $\mu g/m^3$ 

 $m_{Pb}$  – desired Pb mass on swatches, µg

 $A_s$  – area of the swatch, m<sup>3</sup>

 $h_a$  – height of the air column above swatches in the chamber, m

 $f_{sd}$  – fraction of Pb mass in the air estimated to settled over settling period.

To estimate the fraction  $(f_{sd})$  of Pb mass in the air that would settle over a

settling period, the investigators used a Pb mass distribution and terminal settling velocities for Pb-containing particles in GSR. The data for Pb mass distribution by particle aerodynamic size was extracted from Dams et al. study (10). The terminal settling velocities for particles of the reported aerodynamic sizes were determined using the on-line calculator offered by the Harvard University (35).

Using terminal settling velocities ( $V_{TS}$ ), the investigators calculated the distance (y) particles of each size would travel in a selected time period. Then, divided the
traveled distances (y) by the height (h<sub>a</sub>) of the air column above the swatch equal to 2.26 meters to determine the fraction (f<sub>p</sub>) of particles of each size in the air column that would have reached swatches within the selected time period. The resulting values are then multiplied by the particle mass fraction (f<sub>Pb</sub>) each particle size contributes to the total Pb mass in GSR. The sum of the resulting fractions of total Pb mass by particle size (f<sub>pm</sub>) expected to settle within the selected time period signifies the fraction of total Pb mass in the air expected to settle ( $f_{sd}$ ) on swatches within the selected time period. Table 1 summarizes the parameters used in estimating the  $f_{sd}$  for a 7-hr settling period. The investigators determined the  $f_{sd}$  values for the 5, 10, 15, 30, and 45-min settling periods, and then for 1 thru 8-hr time periods by an hour. Figure 9 shows the changes in  $f_{sd}$  values over settling time periods.

Pb particle aerodynamic diameter, μm	Particle terminal settling velocity, m/s (V <sub>TS</sub> )	Distance particles travel in 7 hrs, m (y)	Fraction of particles in 2.26- m air column expected to settle in 7 hrs $(f_p)$	Fraction of Pb particle mass in GSR by particle size (Dams et al.) (f <sub>Pb</sub> )	Fraction of total Pb mass expected to settle in 7 hrs by particle size (f <sub>pm</sub> )
>9.0	>2.47E-03	>62.24	1.00	0.05	0.05
9.0	2.47E-03	62.24	1.00	0.06	0.06
5.8	1.04E-03	26.08	1.00	0.09	0.09
4.7	6.84E-04	17.23	1.00	0.17	0.17
3.3	3.42E-04	8.61	1.00	0.20	0.20
2.1	1.42E-04	3.57	1.00	0.17	0.17
1.1	4.13E-05	1.04	0.46	0.13	0.06
0.7	1.79E-05	0.45	0.20	0.07	0.01
0.4	6.66E-06	0.17	0.07	0.06	0.00

Table 1. Parameters used in estimating the fraction of total Pb mass in the air column expected to settle on swatches within a 7-hr settling period.

Fraction of total Pb mass in the air expected to settle in 7 hrs  $(f_{sd})$ : 0.82



Figure 9. Changes in the fraction of total Pb mass settling over time in a 2.26-m air column.

Based on the results, the investigators estimated that within a 7-hour settling period ca. 82% of Pb mass in the air column above swatches would settle on ACU swatches. Seven hours was the longest time the supporting firing range could facilitate for the study. Using the formula [3] with  $f_{sd} = 0.82$ ,  $A_s = 9.6 \times 10^{-4} \text{ m}^2$ ,  $h_a = 2.26 \text{ m}$ , and  $m_{Pb} = 75 \text{ µg}$  (middle value of the desired load range of 50-100 µg per swatch), the investigators determined that the air Pb concentration necessary to produce the desired load should be ca. 42,200 µg/m<sup>3</sup> or 42.2 mg/m<sup>3</sup>.

Firing of 30 rounds of M80 ammunition during the ammunition comparison test conducted by the ATC in the same chamber resulted in air Pb concentration of 6.62 mg/m<sup>3</sup> (67). Based on the results of the comparison test, the investigators determined that creating an air Pb concentration of 42.2 mg/m<sup>3</sup> would require firing of 191 rounds. To account for potential misfires and weapon malfunctions, the investigators decided to use 200 rounds, which is estimated to result in air Pb concentration of 44.1 mg/m<sup>3</sup>.

### Loading Procedure

The weapon loaded with 200 linked rounds of ammunition and boards with ACU swatches were placed inside the chamber. The chamber doors were closed and the office fans inside the chamber were turned on remotely. The investigator fired the weapon by pulling on the lanyard from outside of the chamber. Ammunition was fired in short 6-7-round bursts with one-second interval between bursts. The firing took approximately two minutes. After the last round of ammunition was expended, the fans continued mixing the air inside the chamber for three additional minutes and were then turned off. The projectiles' exit hole in the front window's cardboard was covered with tape and the chamber with all of its content was left undisturbed for a total GSR settling period of seven hours. At the end of the 7-hour period, the chamber doors were open and the boards with loaded ACU swatches were collected and placed back in paper boxes.

### Collection and Analysis of ACU Swatches

In a wind-protected location adjacent to the chamber, four randomly pre-selected ACU swatches from each board were collected for analysis to estimate Pb load. Each of the selected swatches was removed of a board, the zip tie holding the swatch to the washer was cut with a decorative knife, and the swatch folded in half with forceps was placed inside a 50-ml plastic vial. Each swatch was analyzed for Pb mass in a certified lab using the modified EPA 3052 method of digestion and the EPA 200.8 (ICP-MS) method of analysis. A number of uncut ACU jacket pieces was submitted for analysis as media controls. Two blank ACU swatches were submitted with every batch to examine a potential Pb contamination of swatches during ACU cutting and mounting on washers.

# Transportation of Loaded ACU Swatches

The boxes with the remaining ACU swatches were transported in a vehicle to the follow-on study site located 350 miles away from the loading site. To minimize a potential loss of Pb load on ACU swatches due to sudden agitation during transportation, the boxes with ACU swatches were transported inside a plastic container suspended with elastic bands inside the vehicle as depicted on Figure 10. Upon the arrival to the study site, the investigators collected one randomly selected swatch from each box for analysis to examine the effect of transportation on Pb load.



Figure 10. Securing loaded ACU swatches inside a vehicle for transportation.

## Statistical Analysis

The investigators analyzed the Pb mass-per-swatch data using SPSS<sup>®</sup> Statistics version 22 software. The Kolmogorov-Smirnov test and visual examination of frequencies histograms were used to assess the normality of data distribution. One-way analysis of variance (ANOVA) with multiple comparisons between boards of the same loading event was used to examine spatial variation (homogeneity) of Pb distribution among loading locations in the chamber. The investigators verified the underlying assumption of equal sample variances between boards using the Levene test. Descriptive statistics for a sample of swatches from all boards of the same loading event were obtained. The estimated mean of Pb mass on analyzed swatches was used as the value of "Pb load" for the remaining ACU swatches. To examine the effects of transportation on Pb load, the investigators conducted a t-test for a sample of calculated differences between the assigned Pb load and Pb mass measured on transportation control swatches.

## Air Sampling for Pb Inside the Chamber during the Loading Procedure

The investigators followed the NIOSH method 7300 air sample collection guidance using 37-mm closed face polyurethane cassettes with MCE 0.8 µm pore size filters, GilAir5<sup>®</sup> pumps (Sensidyne, Clearwater, FL), and ¼-inch nylon tubing. Three cassettes were placed inside the chamber each above loading location #'s 1, 4, and 7 and collected 15-min air samples after the ammunition was expended and the fans were turned off. The registered value represented the air Pb concentration in the chamber at the beginning (start) of the loading procedure. The second cassette placed above the loading location #4 collected a 15-min air sample during the last 15 minutes of the 7-hour settling period and represented the air Pb concentration at the end of the loading procedure. All

cassettes were placed 58 inches of the chamber's floor. Figure 11 illustrates the location of cassettes in the chamber. Nylon tubing routed through the chamber's side window connected the cassettes to the pumps located outside the chamber. The pumps sampled air at 3.8 L/min flow rate and were calibrated with DryCal<sup>®</sup> DC-Lite model H (BIOS, Butler, NJ) on site before and after the loading procedure using a nylon tubing of the same length as was used for air sampling. The average of the two readings was used to calculate a total air volume sampled: the average flow rate was multiplied by the sampling time.





Since the cassettes had to be left inside the chamber for the entire duration of the loading procedure, the effect of a potential Pb migration into cassettes was assessed with

a field blank. A cassette with the pump-side plug in place and the air-side plug removed was taped to the nylon tubing four inches above the second cassette at the loading location #4. The Pb mass discovered in the field blank was subtracted from the Pb mass in collected air samples prior to calculating air concentrations. Media blanks were submitted per method guidance.

The air samples were analyzed for Pb by a certified lab using modified NIOSH method 7300 (ICP-MS) and included the wiping of the cassettes' interior surfaces. The investigators used the MS Excel<sup>®</sup> software to determine the average air Pb concentration in the chamber between the three samples at the beginning of the loading procedure and to calculate a relative decrease in air Pb concentration over the GSR settling period.

The investigators intended to use the observed relative decrease in air Pb concentration over the settling period to assess the accuracy of the theoretical approach in estimating the fraction  $(f_{sd})$  of Pb mass estimated to settled. In its essence, a relative decrease in air Pb concentration is the change in Pb mass per air volume and, therefore, is the fraction  $(f_{sd})$  of Pb mass settled from the air column above the air sampling cassettes. The investigators calculated the 7-hour  $f_{sd}$  for the 1-meter air column above the cassettes using the same theoretical approach. The resulting  $f_{sd} = 0.91$  suggested that air sampling cassettes should register a 91% decrease in air Pb concentration over the 7-hour settling period.

# **Loading Events**

In the course of developing the loading procedure and producing GSR-loaded ACU swatches, the investigators conducted six loading events, one per day, over a 3month period. During each event, the investigators followed the loading procedure described earlier but with a different number of boards and ACU swatches. The difference between loading events is depicted on Figure 12 and explained below.



Figure 12. Number and position of loading boards and swatches during loading events. The first loading event (Day1) was investigatory in nature and aimed at confirming the adequacy of the selected type and amount of ammunition, duration of

GSR settling period, and limits of quantification of selected analytical methods. Thus, only one board with four swatches was placed in the loading location #4 and two single swatches were placed in loading locations #1 and #7.

The goal of the following two loading events (Day2 and Day3) was to examine the spatial distribution of Pb among the loading locations and to confirm the reproducibility of the desired load over several loading events. Three boards were placed inside the chamber one at each of the loading locations #1, 4, and 7. Each board contained only four ACU swatches attached in positions randomly pre-selected for analysis.

During the next loading event (Day4), the investigators produced a small batch of GSR-loaded ACU swatches to pilot the follow-on study. Three boards containing all 16 swatches were placed in the loading locations #1, 4, and 7. The investigators analyzed four swatches from each board to estimate the Pb load and transported the remaining swatches to the follow-on study site to be used in the pilot. Once the sample size for the follow-on study has been determined based on the results of the pilot, the investigators conducted two full-scale loading events (Day5 and Day6) to produce the required amount of GSR-loaded ACU swatches.

## RESULTS

The results of the six loading events are summarized in Table 2.

### Pb Load on ACU Swatches

The visual examination of frequency distribution of Pb mass on analyzed swatches during each loading event discovered no significant deviations from normality. The Kolmogorov-Smirnov test of normality for the two full-scale loading events (Day5

and Day6) with the largest sample sizes (n=28 and n=24) suggested the normal

distribution of the data (p>0.200 and p=0.127 respectfully). Based on these findings, the

researchers assumed the normal distribution of the data for all loading events.

		Day1	Day2	Day3	Day4	Day5	Day6
Loading event description	Amount of ammunition, rnd	199	200	200	200	200	200
	Settling duration, min	405 <sup>A</sup>	375 <sup>a</sup>	420	420	420	420
	Number of boards	1	3	3	3	7	6
	Board loading locations	4	1,4,7	1,4,7	1,4,7	1-7	1-5,7
	Number of swatches loaded	6	12	12	48	112	96
	Start, mean (n=3)	38.3	47.3	44.3	42.0	45.7	48.7
	End (n=1)	0.5	2.2	0.8	0.6	2.3	3.9
Air Pb	Abs. decrease	37.8	45.1	43.6	41.4	43.3	44.8
$(mg/m^3)$	Rel. decrease, %	98.7	95.3	98.3	98.6	94.9	92.0
	Field blank, µg/sample	na	20.0	17.0	23.0	17.0	16.0
	Media blank, µg/sample	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5
	Sample size	6	12	12	12	28	24
	Mean	80.3	67.6	111.7	109.2	77.9	82.7
	Std. error	1.994	1.983	1.124	1.486	0.657	1.382
	Rel. std. error, %	2.48	2.93	1.01	1.36	0.84	1.67
	Median	79.0	66.0	110.0	110.0	77.9	83.3
Pb mass	Variance	23.87	47.17	15.15	26.52	12.07	45.85
on ACU	Std. deviation	4.89	6.87	3.89	5.15	3.47	6.77
swatches	Rel. std. deviation, %	6.08	10.16	3.49	4.72	4.46	8.19
(µg/swatch)	Minimum	77	51	110	100	70	61
	Maximum	90	77	120	120	84	93
	Range	13	26	10	20	14	32
	Interquartile range	6	9	0	0	4	8
	Media blank, µg/sample	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0
	Blank swatch, µg/sample	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0
Statistical	Kolmogorov-Smirnov	0.014	0.016	0.000	0.000	0.200	0.127
	Levene	na	0.489	0.000	0.002	0.876	0.166
(p-value)	ANOVA between boards	na	0.746	0.100	0.100	0.531	0.488
×	t-test, trans. (2-tailed)	na	na	na	0.220	0.063	0.909
<sup>A</sup> The settling duration on Day1 and Day2 were short of the desired 7 hours (420 min) due to impeding range operations.							

The ANOVA of Pb mass on swatches with multiple comparison between boards of the same loading event did not show statistically significant difference suggesting that Pb was equally distributed among all loading locations. The underlying assumption of equal variances between boards of the same loading event was confirmed with statistically significant results of the Levene test for Day2, Day5, and Day6 (p-values of 0.489, 0.876, and 0.166 respectfully). The Levene test for Day3 and Day4 showed inequality of variances between boards with p-values of 0.000 and 0.002 respectfully. Because the supporting laboratory was reporting the results of Pb mass analysis rounded to two significant figures, at least one board during Day3 and Day4 loading events had all four values of Pb mass equal to 110  $\mu$ g/swatch. This resulted in a zero variance in data for that board and the Levene test detected a significant difference in variances between boards. Following this observation, the investigators requested that laboratory report the results of subsequent tests with three significant figures.

The mean Pb mass per swatch varied between loading event from 67.6 and 111.7  $\mu$ g with an average of 88.2  $\mu$ g/swatch. The relative standard error for the reported means ranged from 0.84 to 2.93% with an average of 1.72%. The coefficient of variation ranged from 3.49 to 10.16% with an average of 6.18%. Other statistical parameters for each loading event can be found in Table 2. All media controls and blank swatches were negative. The transportation was not found to have a statistically significant effect on Pb load on swatches.

## Air Pb Concentrations in the Chamber

Field blanks present inside the chamber during the entire duration of the settling period passively accumulated on average 18.6 µg of Pb per sample ranging from 16 to 23

µg/sample. These amounts were substracted from Pb mass found in air samples prior to calculating the concentrations of Pb at the beginning (start) and at the end of each loading event. The average air Pb concentration in the chamber at the beginning of loading events was 44.4 mg/m<sup>3</sup> ranging from 38.3 to 48.7 mg/m<sup>3</sup>. The avarage decrease in Pb concentration over a settling period was 96.3% ranging between 92.0 and 98.7%. All media controls were negative.

### DISCUSSION

The study has succeeded by achieving four out of six Pb loads within the target range of 50-100  $\mu$ g. Two loading events produced Pb loads above 100  $\mu$ g; however, going above the range was not detrimental for the follow-on study's objective. The target range for the load was based on the investigators' desire to create a Pb load in the amount close to the suspected levels of Pb clothing contamination on IFRs, i.e. in the order of micrograms per square centimeter. At the same time, the load had to be large enough that the subsequent amounts anticipated from manipulations in the follow-on study would be measurable with the selected analytical methods. The investigators anticipated the subsequent amounts as low as 1% of the initial Pb load, therefore, to be measurable by an analytical method with a limit of quantification at 0.5  $\mu$ g per sample, the initial Pb load had to be above 50  $\mu$ g per swatch.

Although the study did not evaluate the case-effect or correlation of various aspects of the suggested loading procedure and characteristics of the resulting Pb loads, the investigators believe that procedure's key features made certain the results of the study. Mixing the GSR with fans have ensured a homogenous distribution of Pb on swatches with an average relative standard deviation of 6.18%. Sufficient sample sizes

and small variation in Pb mass between swatches allowed an accurate estimation of the Pb load with relative standard errors between 0.84 and 2.93%. The suggested packaging and transportation technique ensured Pb load preservation during transportation. The results of this study, however, are limited to the type of weapon, ammunition, and swatches' material.

Even though achieving Pb loads of the same value in separate loading events was not an objective of this study, the difference in Pb loads is worth discussing. Despite using the ammunition of the same lot and following the same loading procedure protocol, the difference of Pb loads between some loading events was statistically significant. The Pb loads produced on Day3 and Day4 were on average 25-39% higher compared to other loading events ( $\alpha = 0.05$ , p = 0.854). The only difference between these two and other events that the investigators had noticed was environmental conditions inside the chamber during the loading. During Day3 and Day4, the air temperature was higher and the relative humidity was lower compared to other events. The study did not aim at investigating the effects of environmental conditions on Pb loads and therefore did not collect sufficient environmental data allowing any meaningful correlation. The effects of air temperature and relative humidity specific to GSR particles' generation and behavior in the air following a firearm's discharge have not been found in the literature reviewed for this study and is a subject for future research.

The theoretical approach used in this study to predict the Pb load appeared to be relatively accurate. The amount of ammunition selected for the study produced on average 44.4  $\mu$ g/m<sup>3</sup> of airborne Pb in the chamber, which is less than 1% off the predicted value of 44.1  $\mu$ g/m<sup>3</sup>. Such a high accuracy can be explained with the fact that the

prediction was based on the data from Veety study that used the same type of weapon and ammunition and in the same chamber as was used in this study. Veety (67) fired several types of 7.62mm ammunition and compared the levels of produced various toxic substances including Pb. Various types of ammunition have been shown to produce different amounts of airborne Pb; therefore, prior to loading samples in the future, the investigators recommend firing of a designated ammunition and sampling air for Pb to enable accurate prediction of a desired air Pb concentration.

The predicted fraction  $(f_{sd})$  of Pb mass estimated to settled within 7 hours from the air column above the air sampling cassettes was slightly underestimated. The observed average relative decrease in air Pb concentration was 96.3%, which is slightly higher than the predicted value of 91%. The predicted value was based on a Pb mass distribution reported in Dams et al. study that used a different type of weapon, ammunition, and different air sampling methods, which could explain the disagreement between the predicted and observed  $f_{sd}$  values of this study. In addition, the predicted  $f_{sd}$  was estimated based on terminal settling velocities calculated for the standard temperature and pressure whereas the temperature inside the chamber was different and changing throughout the loading events. As was stated earlier, air temperature and relative humidity could have also affected the settling of GSR particles.

Regardless of a small disagreement between the predicted and observed  $f_{sd}$  and the resulting average Pb load within the predicted range, the investigators cannot conclude that the Pb mass distribution produced by the ammunition used in this study was similar to the one reported in Dams et al. study. The 7-hour settling period used in this study allowed for 100% of particles with aerodynamic size  $\geq 2.1$  µm to settle on

swatches as shown in Table 1. This means that any distribution of Pb mass between particles of that size range could result in the same Pb load. Therefore, an agreement between predicted and observed values in this study should not lead to a conclusion of similar Pb mass distributions.

Despite the fact that many studies have examined Pb exposure at firing ranges, it appears that only a few had conducted an analysis of particle size-specific Pb mass distribution. Besides the study by Dams et al., the literature review for this study revealed one additional published study and one unpublished work on this subject. Further research of Pb mass distribution produced by various types of ammunition would enhance the accuracy of theoretical estimates for loading techniques such as reported in this study as well as for assessments of Pb exposures taking place on firing ranges.

Using an appropriate Pb mass distribution data would also allow to determine the optimum or desired settling period duration for the loading procedure depending on a study objective. The predicted  $f_{sd}$  values based on data from Dams et al. (Figure 9) suggest that during the first five hours ca. 80% of Pb would settle on swatches. For every additional hour, the Pb load would increase by 1%. Therefore, if a study is not concerned with loading samples with a full spectrum of particle sizes but rather with a Pb mass in general, an investigator may choose to shorten the settling duration period and increase the air Pb concentration to achieve a desired Pb load in a shorter settling period. In this study, the investigators opted for the longest possible settling period since the particles' size is of a particular interest for the follow-on study.

# CONCLUSION

This study is the first to load fabric swatches with a desired amount of a GSRspecific Pb in the order of micrograms per square centimeter. The developed loading procedure was accurate and reproducible but limited to the type of weapon, ammunition, and swatch material used in this study.

### RECOMMENDATIONS

Occupational and environmental hygiene practitioners and researchers could use the loading procedure developed in this study to produce GSR-loaded fabric samples to examine the efficiency of clothing cleaning techniques such as air showering, vacuuming, and laundering. Prior to using the loading procedure, the investigators recommend conducting a firing of a designated ammunition and performing air sampling to determine the amount of Pb and mass distribution by particle size produced by the selected ammunition. The effects of air temperature and humidity should be further examined and addressed during future loading events.

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# CHAPTER 3: The Pilot: Evaluating Materials and Methods Developed for the Main Study

### INTRODUCTION

The purpose of the main study was to evaluate the efficiency of an AS in removing Pb from ACU swatches and to examine a potential breakthrough of Pb across ACU material. The main study design entailed placing ACU swatches loaded with a known amount of Pb inside an AS and exposing the swatches to the cleaning action of air streams inside the AS selected for the study. Analysis of Pb mass remaining on swatches after the exposure in the AS would indicate the percent of Pb reduction. By placing swatches at selected distances (points) away from a nozzle-bearing wall and at selected angles to air streams produced by the nozzles, the investigators examine the effects of the select point air velocities and angles of impact on Pb removal. To examine the Pb breakthrough, the investigators envisioned isolating and collecting the Pb particles pushed by air streams across ACU swatches during the cleaning action of the AS. While designing materials and methods for the main study the investigators faced several challenges.

The greatest challenge was the development of a method to isolate the breakthrough Pb and collecting it for analysis. The Pb load on ACU swatches used in this study was in the range of ca. 50-100  $\mu$ g of Pb per swatch. Based on the results of the study by Simonson et al., the investigators anticipated the subsequent amounts of the breakthrough Pb as low as 1% of the initial Pb load, which could result in a breakthrough amounts in this study as low as 0.5-1.0  $\mu$ g of Pb. Therefore, to be measurable by the available analytical method with the limit of quantification (LOQ) of 0.5  $\mu$ g per sample, a

breakthrough collection technique used the study must be very efficient at collecting the breakthrough Pb. Another option is to use a technique allowing the collection of the breakthrough Pb from several swatches to increase the amount of Pb per sample above the LOQ. In addition, a selected technique must be able to isolate the breakthrough Pb from the Pb removed off a swatch and present in the air inside the AS during the cleaning action.

A search of the peer-reviewed literature identified one study suggesting a method of a breakthrough Pb collection. Simonson et al. collected the breakthrough Pb by placing a filter underneath each of the Pb-loaded fabric swatches (62). Although the researchers detected the Pb on filters in amounts above the LOQ, the Pb load used by Simonson et al. was three orders of magnitude higher than the amount used in this study. In addition, the breakthrough collection procedure used by Simonson et al. would not allow the collection of a breakthrough Pb from several swatches.

Securing swatches during the test also had to be addressed. During the exposure to the cleaning action of the AS, swatches would be exposed to air velocities in excess of 6,000 feet per minute. Such high air velocities pose a threat of displacing a swatch during the exposure inside the AS and necessitate securing the swatch in place. The challenge here was to secure a swatch during the test in a manner that would not interfere with the cleaning action of the air streams affecting the retention of Pb on a swatch or the Pb breakthrough across it. A swatch attachment technique reported in Simonson et al. entailed using a double-sided adhesive tape; however, such method could obstruct the airflow across a swatch potentially affecting the breakthrough of Pb. Other studies did not provide details on swatch securing methods. The

Another aspect of consideration for the main study design was selecting the air velocities of interest. A common practice among AS manufacturers and distributors when describing their devices is to report a nozzle air velocity produced by an AS. Nozzle air velocity is the linear velocity of air measured at the face of a nozzle. However, this technical characteristic can be hardly used as a reference point for selecting air velocities that would be relevant in assessing an AS's efficiency. Since the air velocity decreases with distance, the velocity of air streams reaching the surface of an individual's garment would depend on internal dimensions of the AS as well as the number and position of nozzles. In addition, there is no standard on AS design and performance; ASs made by different manufacturers vary in size and nozzle parameters, thus a nozzle air velocity is not a good measure to compare ASs' performance.

In prior AS studies, the researchers did not elaborate on methodology of selecting air velocities of interest for their studies. In studies assessing the effects of various air velocities on a substance removal off a fabric swatch, the investigators usually put fabric swatches at a distance away from a nozzle and then adjusted the nozzle air velocity to achieve various values (26; 36). Most studies assessed the efficiency of an AS as a whole unit, where the investigators either contaminated the entire garment or attached contaminant-loaded swatches to various parts of an individual's garment and exposed the subject to the cleaning action of the AS at a fixed nozzle air velocity recommended by the manufacture (29; 37; 68; 69).

A literature search for standard methods to address the identified challenges did not discover any techniques appropriate for this study. Most AS studies have been conducted by AS manufacturers and have not been published in peer-reviewed, scientific

literature. The results were usually presented during cleanroom industry society meetings and conferences and their proceedings were not always available for review for this study. The studies available for review lacked details on methods and materials and presented little statistical data.

The purpose of the pilot study was to test the materials and methods developed for the main study and to obtain statistical parameters for determination of the minimum sample size necessary to detect the difference between Pb mass on swatches "before" and "after" AS exposure with at least 0.8 power and 95% confidence ( $\alpha = 0.05$ ). The main goal of the pilot was to test three proposed methods of capturing the breakthrough Pb in order to select the most appropriate technique and to ensure the initial Pb load on ACU swatches was sufficient to produce subsequent breakthrough Pb amounts within LOQs of selected analytical methods.

### MATERIALS AND METHODS

The activities of the pilot took place in a laboratory and at the site of the main study.

### Air Shower Description

The investigators used the same AS for both the pilot and main study. The AS model CAP701KD-ST-4954 (Clean Air Products, Minneapolis, Minnesota) was situated on an indoor firing range. The booth internal L x W x H dimensions were  $52 \times 40 \times 86$  inches. The AS had 36 anodizes aluminum nozzles with 17 nozzles on both sides and 2 nozzles on the ceiling. The nozzles were 1¼ inches (30 mm) in diameter protruding inside the booth for 1½ inches (38 mm) at a 90-degree angle to the walls' surface. An average nozzle air velocity reported by the manufacture for this model was 7,800 fpm.

The air return was in the floor of the AS covered with a steel grate to protect a pre-filter followed by a high-efficiency particulate arrestance (HEPA) filter. Figure 13 illustrates the AS used in the study. The AS was set for a 15-second cleaning cycle activated with a push of the button located inside the AS.



Figure 13. The view of the air shower used in the study.

## **Nozzle and Point Air Velocities**

The investigators used a multi-functional ventilation meter VelociCalc<sup>®</sup> model 9565-P with a heated wire anemometer probe 966 (TSI Inc., Shoreview, Minnesota) to measure air velocities in the AS. The nozzle air velocities were measure to ensure the AS was performing at the manufacture recommended settings. The tip of the probe was placed at the surface and in the middle of a nozzle. For each nozzle, the investigators took one 5-second averaged air velocity reading in the middle of the 15-second cleaning cycle. The mean of all nozzle air velocities was 7,807 fpm, which indicated that the AS was performing as designed.

The point air velocities of interest selected for the main study were based on estimated distances "points" between the AS's nozzle-bearing walls and an ACU that are likely to take place inside the booth during a standard cleaning procedure. The investigators placed an ACU-dressed volunteer of an average Soldier's size (69-in statue and 46-in shoulder circumference (22)) inside the AS and directed to follow a recommended cleaning protocol. While the volunteer was turning around inside the AS with hands raised above the head, the investigators measured distances between the booth's wall and volunteer's ACU at chest-, beltline-, and knee-levels during various moments of the turning motion. Throughout several repetitions of the above procedure, the distances measured between the wall and various regions of ACU ranged between 8 and 19 inches. The 8 and 19 inches were selected as a minimum and maximum, and 13 inches as the mid-value for distances that were estimated to take place inside the booth.

The investigators then measured point air velocities inside the AS during the cleaning cycle at the selected distances away from the nozzle wall. The heated wire anemometer probe was fixed with a clamp on a stand and placed inside the booth as shown on Figure 14. The tip of the probe was placed 8 inches off the nozzle wall and was centered in the booth 26 inches away from each door and 43 inches away from each the floor and the ceiling. The investigators took a 5-second averaged air velocity reading in the middle of the 15-second cleaning cycle. The mean of 10 readings was rounded to two

significant figures and was used as the indicator of the air velocity at the selected point away from the wall. The above procedure was repeated for distances of 13 and 19 inches away from the wall. Table 3 summarizes the results of measuring nozzle and point air velocities. The resulting point air velocities at 8, 13, and 19 inches off the wall were 6,900, 4,100, and 1,800 fpm accordingly.



Figure 14. Measuring point air velocity inside the AS at 8 inches away from the wall.

			Point Air Velocities			
		Velocity	8 in	13 in	19 in	
Readings (n)		36	10	10	10	
Mean air velocity, fpm		7807.3	6911.8	4117.0	1799.8	
Std. error		41.4	122.7	26.4	12.3	
Rel. std. error, %		0.5	1.8	0.6	0.7	
95% CI	Lower bound	7723.3	6634.2	4057.4	1772.0	
	Upper bound	7891.3	7189.4	4176.6	1827.6	
Std. deviation		248.2	388.1	83.4	38.8	
Min		7358	5869	4006	1734	
Max		8668	7212	4243	1879	
Range		1310	1343	237	145	
Rounded	mean	7,800	6,900	4,100	1,800	

Table 3. The results of measuring nozzle and point air velocities inside the AS.

## **Angles of Impact**

The angles of impact used in this study were selected based on the observation of a body position and movement during the standard cleaning procedure inside the AS. Since the volunteer stood in an up-right position inside the AS, majority of the ACU surface was in the vertical plane during the cleaning process. Due to the individual's rotating movement in the AS during the cleaning cycle, air streams coming out of the air nozzles fell on vertical surfaces of ACU at angles changing from 0 to 90 to 180 degrees. The investigators selected the 0-, 45-, and 90-degree angles of impact for the study; a use of angles from 90 to 180 degrees would result in an air stream force of the same magnitude but in an opposite direction and therefore were not considered. Thus, during the study, the investigators placed ACU swatches inside the AS vertically and at the selected angles to air streams coming from the nozzles (Figure 15).



Figure 15. Positioning ACU swatch 8 inches off the wall at 45-degree angle of impact. The selected three point air velocities of 1,800, 4,100, and 6,900 fpm and threeangles of impact of 0, 45, and 90 degrees formed nine combinations of the two variablesof interest. The ACU swatches exposed to each combination of variables were annotated

as sub-samples  $n_{ij}$  of the total sample size n as depicted in Figure 16.

Variables		Point Air Velocity (fpm)				
		1,800	4,100	6,900		
act (°)	0	n <sub>11</sub>	n <sub>21</sub>	n <sub>31</sub>		
t of Imp	45	n <sub>12</sub>	n <sub>22</sub>	n <sub>32</sub>		
Angle	90	n <sub>13</sub>	n <sub>23</sub>	n <sub>33</sub>		

Figure 16. Combination of variables and corresponding sub-samples  $n_{ij}$  of ACU swatches.

# **Pb-Loaded ACU Swatches**

The investigators used a permethrin treated 50% cotton, 50% nylon fabric meeting the requirements of MIL-STD-44436 (Class 8) to prepare ACU swatches. The resulting ACU swatches (Figure 17) were approximately 45 mm in diameter with the exposed surface of 35 mm in diameter and an area of 9.6 cm<sup>2</sup>. The investigators loaded ACU swatches with GSR-specific Pb by firing a Pb-containing ammunition inside a sealed chamber and allowing the produced GSR to settle on ACU swatches placed inside the chamber. The investigators prepared a batch of 36 ACU swatches with the Pb load of 109.2  $\mu$ g of Pb mass per swatch to be used in the pilot. The swatches were packaged in three paper boxes each containing 12 swatches. The details of ACU swatch preparation, loading, packaging, and transportation to the test site have been described in Chapter 2.



Figure 17. The ACU swatch.

### **Test Assembly**

The main purpose of the test assembly was to secure an ACU swatch during the exposure to high air velocities inside the AS and to facilitate the collection of the breakthrough Pb. The assembly consisted of a breakthrough catchment chamber, filtered air supply system, and breakthrough collection pump. The major components of the test assembly are depicted on Figure 6 and their design and purpose are explained below.



Figure 18. Major components of the test assembly.

# **Breakthrough Catchment Chamber**

The main purpose of the chamber was to secure a swatch and isolate the breakthrough Pb particles pushed across a swatch into the chamber by point air velocities in the AS. The investigators devised the breakthrough catchment chamber (the "chamber") shown on Figures 19 and 20 using a chrome plated brass outlet tee (P/N 540TTK, Keeney Mfg. Co., Newington, Connecticut). A latching clamp from a hinged glass jar was placed around the chamber's top orifice and was used to secure an ACU swatch to the chamber during the test. To prevent high velocity air streams in the AS from pushing a swatch inside the chamber during the test, the investigators fabricated a swatch-supporting base. The base was made from a piece of a 24-gage chrome plated brass tubing (P/N 30304CCP, Keeney Mfg. Co., Newington, Connecticut) and a stainless steel mesh from a sink strainer basket (P/N SF3511, BrassCraft Mfg. Co., Novi, MI). To ensure a tight seal between the orifice's rim, supporting base, and a swatch, the investigators used two rubber gaskets cut out of the rubber [gasket] sheet (P/N 59849,

Danco Inc., Irving, Texas). One gasket was placed between the orifice and the base and another between the base and a swatch washer. To enable latching clamp's firm grip on a swatch, the investigators used a rigid galvanized steel conduit-reducing washer (P/N 68510, Halex Co., Cleveland, OH). All together the washer, swatch, supporting base, and rubber gaskets formed a "stack" referenced later in this study.



Figure 19. View of the assembled breakthrough catchment chamber.



Figure 20. Components of the breakthrough catchment chamber.

The chamber's side orifice was utilized as the attachment site for a 37-mm polypropylene air sampling filter cassette used in one of the proposed methods of breakthrough collection. Breakthrough collection techniques are described in detail below. To facilitate a quick and tight fitting of cassettes to the chamber, the investigators used a mid-section of a three-part 37-mm polyurethane cassette as a connecting link. The mid-section was inserted inside the chamber's side orifice and secured with a nut. The third end of the chamber was fitted with a threaded polyvinyl chloride (PVC) coupling to enable the attachment of the filtered air supply system.

The investigators acknowledge that to eliminate potential effects on study results, the materials comprising the chamber should be Pb-free or should contain insignificant amounts of Pb. Due to funding limitations of this study, the investigators were unable to determine the extent of Pb presence in materials used in the construction of the chamber. The materials and parts chosen for this study were based on availability, low price, and the need to minimize additional fabrications.

### **Breakthrough Collection Techniques**

<u>Method A</u>. The first technique involved placing an MCE 0.8 µm pore size filters underneath a swatch. The filter rested on top of the swatch support base and passively collected the breakthrough Pb particles pushed across the swatch by air streams during the cleaning action of the AS. One MCE filter collected the breakthrough Pb from one ACU swatch. The investigators sealed one chamber's side orifice with a PVC cap and connected a vacuum cleaner hose RIDGID<sup>®</sup> VT1720 (Ridgid, Inc., Newark, Delaware) to the other side orifice in order to isolate the chamber from air currents inside the AS while allowing an unrestricted passage of the breakthrough airflow across a swatch and filter. The open end of the hose was routed outside the AS. Figure 21 illustrates the chamber configured for a breakthrough collection Method A.



Figure 21. Chamber configuration for a breakthrough collection Method A.

Method A offered several advantages but also had potential drawbacks. This technique allowed the collection of the breakthrough Pb from each swatch individually while minimizing a potential loss of some breakthrough amounts due to Pb particles' adhesion to the chamber's walls. However, placing a filter underneath a swatch could create a resistance to the breakthrough airflow across the swatch thus potentially decreasing the amount of the breakthrough Pb passing through the fabric. In addition, this technique did not allow for collection of the breakthrough Pb from several swatches in case the breakthrough amount from a single swatch would be below the LOQ of the selected analytical method.

<u>Method B</u>. The second technique called for pumping the chamber's air during the cleaning action of the AS through a filter to collect the breakthrough Pb particles while they are still airborne inside the chamber. In Method B, a 37-mm closed face polyurethane cassette with MCE  $0.8 \mu m$  pore size filter was attached to the chamber's

side orifice as was described earlier. The air was collected by the GilAir5<sup>®</sup> pump (Sensidyne, Clearwater, FL) connected to the cassette using a <sup>1</sup>/<sub>4</sub>-inch nylon tubing. One filter cassette collected the breakthrough Pb from one swatch. Figure 22 illustrates the chamber configured for a breakthrough collection Method B.



Figure 22. Chamber configuration for a breakthrough collection Method B.

<u>Method C</u>. The third techniques was a modification of the Method B in which one filter cassette was used to collect the breakthrough Pb from five swatches exposed in the AS one after another. This method addressed a potential issue of achieving a below-LOQ breakthrough amounts collected from single swatches. A step-by-step description of breakthrough collection process is described in the Test Procedure section of this Chapter.

### Estimating the Breakthrough Collection Flow Rate

Breakthrough collection Methods B and C would not create a resistance to the breakthrough airflow as would the filter in Method A, but presented a potential drawback of losing some amount of the breakthrough Pb due to Pb particles' adhesion to the chamber's walls. To minimize the resting time of the breakthrough Pb particles inside the chamber potentially causing Pb particles' settling and/or adhesion to chamber's walls, the breakthrough collection flow rate had to be equal or greater than the rate of the breakthrough airflow across a swatch. To estimate the breakthrough airflow across a swatch caused by air velocities selected for the study, the investigators conducted a test using the chamber, a fan, and an air flow meter.

In a laboratory setting, the investigators exposed the chamber with attached blank swatch to various air velocities produced by a fan and measured the airflow passing across the swatch. Both side orifices of the chamber were covered with PVC caps equipped with air tube connectors. One connector was sealed and another was connected with a nylon tubing to the flow meter DryCal<sup>®</sup> Defender 510-M (Mesa Labs Inc., Butler, New Jersey). The investigators placed the chamber at a 90-degree angle to the fan's exhaust and measured the airflow coming across the swatch (see Figure 23). Placing the chamber at various distances away from the fan, the investigators achieved various point air velocities, which were measured with the heated wire anemometer mentioned earlier. To examine the effects of the swatch supporting base on the breakthrough air flow, the investigators conducted tests with and without the base. Using the results shown of

Figure 24, the investigators estimated that exposing a swatch to the highest air velocity selected for the study (6,900 fpm) would result in ca. 1.2-L/min breakthrough airflow across a swatch resting on the supporting base. To account for a potential underestimation and to ensure the airflow across a swatch had been overcome, the investigators decided to collect the breakthrough at a flow rate of 3.8 L/min.



Figure 23. Determining the breakthrough airflow in the laboratory.



Figure 24. Breakthrough airflow rates across an ACU swatch at various point air velocities.

Throughout the pilot, the pump's flow rate was periodically verified with a flow meter DryCal<sup>®</sup> Defender 510-M, and as long as the flow rate was within  $\pm$  5 % of the intended value of 3.8 L/min, no adjustments were made. The flow rate was periodically verified to ensure it overcomes the estimated breakthrough flow and for consistency in procedures throughout the pilot. The total volume of air pumped through a cassette had no relevance for the breakthrough collection techniques and therefore was not reported.

However, collecting the chamber's air at a rate exceeding the breakthrough airflow across a swatch would cause a negative pressure inside the chamber and would create a backpressure on a swatch potentially increasing the airflow across a swatch and the breakthrough of Pb. To minimize the effects of the pumping action on Pb breakthrough, the investigators incorporated a filtered air supply system into the test assembly designed to eliminate the negative pressure inside the chamber during the breakthrough Pb collection.

# Filtered Air Supply System

The major components of the filtered air supply system (the "system") are the vacuum pump, air tank, and hose connecting the system to the chamber. The vacuum pump available for the study was model 1531-107B-G557X (GAST Mfg. Corp., Benton Harbor, Michigan). The investigators fabricated an air tank using a 3.8L Sterilite<sup>®</sup> Ultra-Latch<sup>®</sup> plastic food container (P/N 0425, Sterilite Co., Townsend, Massachusetts). A vacuum HEPA filter Arm & Hammer<sup>®</sup> Dirt Devil<sup>®</sup> F2 (Church & Dwight Co. Inc., York Township, Pennsylvania) was installed inside the container with the filter's inlet connected to the outside through the container's wall with a PVC elbow, threaded fitting, two steel washers, and two rubber gaskets. The components of the air tank are depicted

on Figure 25. The vacuum pump's output vent was connected to the air tank's filter inlet with a ¼-in braided PVC hose. To connect the air tank to the chamber, the investigators used a 1-7/8" x 7' universal vacuum cleaner hose RIDGID<sup>®</sup> (P/N VT1720, Ridgid, Inc., Newark, Delaware) fitted on one side with a threaded PVC pipe connector. The investigators fitted one of the tank's walls with a connector for the RIDGID<sup>®</sup> hose. On the opposite wall, the investigators cut out an air vent ca. 2 inches in diameter.





The purpose of the system was to provide a supply of air to the chamber during the breakthrough Pb collection in order to minimize the backpressure on a swatch. The vacuum pump supplied the air at ca. 25 L/min flow rate to provide abundant air supply for the breakthrough Pb collection flow rate. Since the vacuum pump was taking the air
inside the firing range, the HEPA filter was incorporated into the system to ensure the supplied air does not introduce Pb into the chamber, thus affecting the amount of the breakthrough Pb collected by a filter cassette. The vent was incorporated into the air tank's design to allow the excess of supplied air to escape the tank preventing the creation of a positive pressure inside the chamber.

#### Grounding the Chamber

Presence of an electrical charge on the chamber's walls could cause electrostatic attraction of the breakthrough Pb particles to the chamber's walls, thus affecting the amount of the collected breakthrough Pb. To prevent the loss of Pb particles due to electrostatic forces, the investigators used a copper wire to ground the chamber during the test. The investigators attached one end of the wire to the steel clamp holding the chamber and connected the other end to the ground terminal of an electrical outlet.

#### Validating the Test Assembly Design

The purpose of the validation was to ensure the developed test assembly design allows the collection of the breakthrough without causing a backpressure on a swatch. The investigators assembled the experimental setup in a laboratory setting and measured the changes in the air pressure across a swatch while running vacuum and breakthrough collection pumps separately and then conjointly. The chamber's top orifice with a blank swatch in place was sealed with a PVC cap connected to a pressure port on the multifunctional ventilation meter VelociCalc<sup>®</sup> model 9565-P as shown on Figure 26. With the vacuum pump off and the breakthrough collection pump sampling at ca. 3.8 L/min rate, the investigators registered a backpressure across the swatch of -0.014 inches of water gauge (in wg). In a reverse mode, with the breakthrough collection pump off and the air

supply vacuum pump pushing air into the air tank at ca. 25 L/min rate, the investigators registered a positive air pressure across the swatch of 0.001 in wg. When both pumps were operating at the desired flow rates, the multi-functional ventilation meter did not register air pressure across the swatch displaying 0.000 in wg. These results indicated that the test assembly design would allow for the breakthrough Pb collection without causing a significant backpressure on a swatch. To confirm that a potentially very small airflow across a swatch not registered during the validation did not have a significant effect on the collected amount of the breakthrough Pb, the investigators incorporated a backpressure control described in Controls section of this Chapter.



Figure 26. Validating the test assembly design.

# **Test Procedure**

The pilot took place at an indoor firing range equipped with the AS described earlier. The AS was located in a hallway connecting the lobby and the range as depicted on Figure 27. In the lobby, the investigators set up an administrative area where the equipment and swatches were stored and handled. The co-located restroom was used for equipment cleaning. At the time of the pilot, the range was undergoing a renovation and no shooting took place during the test.



Range

Figure 27. Sketch of the test site setup.

From the nine combinations of two variables selected for the main study as described earlier, the investigators selected two combinations to be used in the pilot. The selected combinations were 1,800 fpm at 0° angle and 6,900 fpm at 90° angle corresponding to the sub-samples of ACU swatches  $n_{11}$  and  $n_{33}$  respectfully. Based on

results of prior AS studies, the investigators estimated that the selected sub-samples  $n_{11}$  and  $n_{33}$  would exhibition the lowest and the highest Pb reduction and Pb breakthrough. Descriptive statistics of these two sub-samples would allow the estimation of the minimum required sample size necessary to detect the difference between Pb mass on swatches "before" and "after" AS exposure with at least 80% power and 95% confidence ( $\alpha = 0.05$ ) in the main study.

The Pb-loaded ACU swatches used in the pilot were randomly assigned to the two sub-samples allocating swatches to the three breakthrough collection techniques; some swatches were allocated to be used as various controls (see Table 4). Random numbers were generated using the RANDBETWEEN function in MS Excel<sup>®</sup>. A set of two random numbers were used to first identify the box and then the position of a swatch in the box. Table 4. Swatches allocation in the pilot.

Sub- sample	Variables	Breakthrough collection method	ACU swatches	37mm cassettes	Filters
		А	4		4
n <sub>11</sub>	1,800 fpm 0° angle	В	4	4	
		С	5	1	
			13		
n <sub>33</sub>	6,900 fpm 90° angle	А	4		4
		В	4	4	
		С	5	1	
			13	-	
Controls					
Transportation			3		
Handling		А	3		3
Filtered air supply				1	
Backpre	ssure	sure C 4 1			

Due to limited time availability at the range, the investigators sequenced the test in a manner minimizing the time spent to configure and re-configure the experimental setup for the three breakthrough collection techniques. First, the investigators configured the test assembly for the breakthrough collection Methods B and C and conducted tests with swatches allocated to these techniques from both sub-samples. Then the investigators re-configured the experimental setup for Method A and conducted tests with swatches from both sub-samples allocated to this breakthrough collection technique.

# Protocol for Tests Using the Breakthrough Collection Methods B and C

The test assembly setup inside the AS is shown on Figures 28 and explained below. The chamber with attached blank ACU swatch was mounted on a tripod. The investigators placed the tripod inside the AS in a manner to position the swatch at the point in space inside the AS corresponding to the selected point air velocity of 6,900 fpm, i.e. 8 inches off the wall, 26 inches away from each door, and 43 inches off the floor. Using a Johnson rafter angle square, the investigators rotated the chamber to achieve the selected 90-degree angle of impact. The vacuum pump and air tank of the air supply system were taken through the AS's back door and placed outside the AS on the backside facing the range. The investigators connected the air tank to the chamber with the RIGID<sup>®</sup> hose routed through the back door of the AS. To enable the vacuum pump control, the investigators ran an electric extension cord from the administrative area through the AS to the backside of the device where the vacuum pump was located. The back door was closed and the gap caused by the hose was sealed with a plastic sheeting and adhesive tape. The grounding wire attached to the chamber was routed through the front door to the electrical outlet in the lobby. The investigators then attached a blank

37mm filter cassette to the chamber and routed the nylon tubing through the AS's front door to the breakthrough collection pump located in the administrative area.



Figure 28. The test assembly setup inside the AS for the breakthrough collection Methods B and C.

Before the test began, the air supply system and the chamber were purged off potentially Pb-containing air that entered the assembly during its installation in the AS. The blank swatch was removed and the chamber's orifice was sealed with adhesive tape. Both vacuum and breakthrough collection pumps were turned on for ca. 5 min to purge the air in the chamber, hose, and air tank. The total volume of the assembly was estimated at 7.8L (0.2L + 3.8L + 3.8L respectfully). With the flow rate of the breakthrough collection pump at 3.8 L/min, the assembly purging was estimated to take under 3 minutes. Once the purging was complete, the investigators verified the position and angle of chamber, replaced the blank 37mm cassettes used during purging was with a new cassette, and proceeded with the test.

In the admin area, the investigators stacked the rubber gaskets, the swatch support base, and a Pb-loaded swatch and carried the stack to the AS. The adhesive tape on chamber's top orifice was removed and the stack with a Pb-loaded swatch was secured immediately to the top orifice of the chamber. The AS's front door was closed and both pumps were turned on simultaneously. The investigators then activated the AS for the full 15-second cleaning cycle. At the end of the cleaning cycle, the assembly's pumps were left operating for additional 10 seconds while air streams fading for ca. 5-7 seconds after AS's de-activation were potentially still producing cleaning and breakthrough effects. Once both pumps were turned off simultaneously, the investigators opened the front door, removed the stack with the swatch, and covered the orifice immediately with adhesive tape. The stack was then taken to the admin area where the investigators cut the zip tie holding the swatch to the washer, folded the swatch in half with forceps, and placed it inside a 50-ml plastic vial for analysis. The investigators then formed a stack with the next Pb-loaded swatch randomly assigned to sub-sample  $n_{33}$ , attached the stack to the chamber, and exposed the swatch to the cleaning action of the AS.

The investigators continued the sequence described above exposing all nine swatches allocated to Methods B and C in sub-sample  $n_{33}$ . The breakthrough Pb from the first four swatches was collected using Method B, in which one 37mm cassette collected a breakthrough from one swatch and was replaced with a new cassette at the end of each swatch exposure. The breakthrough from the last five swatches was collected on one

37mm cassette (Method C) that remained attached to the chamber during swatches sequential exposure.

Once all nine swatches have been exposed, the investigators conducted a qualitative Pb test to confirm the assumption that grounding the chamber and overcoming the estimated breakthrough airflow had prevented the breakthrough Pb particles' adhesion to the chamber's walls. The investigators used the EPA-approved for wood and metal (16) LeadCheck<sup>®</sup> swabs (lot# AABBA50, 3M Co., St. Paul, Minnesota), which when rubbed against a surface produce a pink-to-red color indicator if the amount of Pb picked up by a swab exceeded a threshold of 1-2  $\mu$ g. The investigators disconnected the air supply system from the chamber, detached the chamber from the tripod, and further disassembled the chamber in the admin area. Following the LeadCheck<sup>®</sup> instructions, the investigators rubbed a swab against internal surfaces of the chamber and then assessed the color change. After the qualitative Pb test, the investigators washed the chamber and its components with soap and water and dried it with paper towels.

The investigators then re-assembled the test assembly, attached the chamber to the tripod, and re-located the tripod in the AS to position it at the point in space corresponding to the point air velocity of 1,800 fpm, i.e. 19 inches off the wall, 26 inches away from each door, and 43 inches off the floor. Using a Johnson rafter angle square, the investigators rotated the chamber to achieve the selected 0-degree angle of impact. After purging the test assembly, the investigators exposed nine swatches from the subsample  $n_{11}$  to the cleaning action of the AS, collected the breakthrough Pb using Methods B and C, and conducted the qualitative Pb test in the same manner as described above.

# Protocol for Tests Using the Breakthrough Collection Method A

The investigators re-configured the test assembly and experimental setup to enable the collection of the breakthrough Pb using the Method A. The chamber's side orifice used as the attachment site for 37mm cassettes was sealed with a PVC cap and a new vacuum cleaner hose RIDGID<sup>®</sup> VT1720 was connected the other side orifice equipped with the PVC connector. The investigators attached the chamber to the tripod and positioned the tripod inside the AS at the 6,900 fpm and 90° combination as was described earlier. The RIDGIT<sup>®</sup> hose was routed outside the AS through the back door, the back door was closed, and the gap caused by the hose was sealed with a plastic sheeting and adhesive tape. The air supply system, breakthrough collection pump, and grounding wire were not used as they did not pertain to the design of the Method A collection technique.

In the admin area, the investigators stacked the rubber gaskets, the swatch support base, and a Pb-loaded swatch adding a filter underneath the swatch. Using a pair of tweezers, the investigators placed an MCE 0.8 µm pore size filter on top of the support base and covered it with a Pb-loaded swatch. The stack was taken to the AS and attached to the chamber's top orifice. The AS's front door was closed and the AS was activated. After a full 15-second cleaning cycle, the investigators waited for additional 10 seconds and then opened the front door. The stack was detached from the chamber and moved to the admin area where the swatch and filter were placed in two separate 50-ml plastic vials for analysis.

The above sequence was performed for each of the four swatches allocated to Method A in the sub-sample  $n_{33}$ , after which the investigators moved the tripod with chamber into position corresponding to 1,800 fpm at 0° combination and conducted tests

in the same manner with the four Method A swatches from the sub-sample  $n_{11}$ . The qualitative Pb tests were not performed.

## **Controls**

<u>Transportation</u>. The three boxes with Pb-loaded ACU swatches used in the pilot were transported in a vehicle from the loading site to the pilot site located 350 miles away. Upon the arrival to the pilot site, the investigators collected one randomly selected swatch from each box for analysis to examine the effect of transportation on Pb load.

Handling. The investigators were concerned that extensive handling of swatches during the test procedure (i.e. removal from a box, building a stack, carrying a stack to the AS, attaching and detaching a stack to the chamber, carrying it back to the admin area, and then packaging a swatch for analysis) could potentially result in reduction of Pb amount on swatches independent from the cleaning action of the AS. To examine the effect of swatch handling, the investigators exposed three randomly selected Pb-loaded swatches to the full test protocol with exception of the AS's cleaning action. During the tests using the breakthrough collection Method A, the first handling control swatch was collected at the beginning and the second swatch at the end of the sub-sample  $n_{33}$  sequence. The third control swatch was collected in the middle of the sub-sample  $n_{11}$  sequence. Collecting handling controls during tests with Method A technique also allowed the investigators to examine whether the breakthrough collection filters placed underneath a swatch could have been contaminated in the process of building stacks thus affecting the resulting breakthrough Pb amounts measured on filters.

<u>Filtered air supply</u>. The purpose of collecting the filtered air supply control was to confirm the assumption that the air supply system equipped with a HEPA filter would

produce an air supply containing amounts of Pb negligible for this study. Although the indoor firing range was closed for renovation and no shooting took place on the range during the pilot, the investigators needed to ensure that the air supplied to the chamber to facilitate the breakthrough collection with Methods B and C did not contribute significantly to the amounts of Pb collected on 37mm cassettes. The air sample was collected in the same manner as a breakthrough Pb in Methods B and C. After purging the test assembly for the Methods B and C tests with swatches from the sub-sample *n*<sub>33</sub>, the investigators attached a new 37mm cassette to the chamber, turned on both the vacuum and breakthrough collection pumps, and sampled the air supplied to the chamber at a flow rate of 3.8 L/min for 15 minutes. The flow rate was verified with the flow meter DryCal<sup>®</sup> Defender 510-M before and after the sampling. The average of the two readings was used to calculate a total air volume sampled.

Backpressure. The purpose of the control was to ensure the developed test assembly allowed the collection of the breakthrough without causing a backpressure on a swatch. Although the test assembly was validated in laboratory setting as described earlier, the investigators needed to ensure that a potentially very small breakthrough airflow across a swatch not registered during the validation test did not have a significant effect on the collected amount of the breakthrough Pb. In the experimental setup configured for Methods B and C tests with swatches from the sub-sample  $n_{33}$ , the investigators conducted the breakthrough Pb collection from four Pb-loaded swatches to one 37mm cassette without activating the AS. Each of the four swatches remained attached to the chamber for a breakthrough collection duration of 30 seconds (5s before AS activation + 15s AS cleaning cycle + 10s waiting period). The investigators used four

swatches instead of five as required for Method C technique due to insufficient number of Pb-loaded swatches.

#### **Analytical Methods**

The investigators delivered all samples to a certified lab for analysis. ACU swatches were analyzed for Pb mass using the modified EPA 3052 method of digestion and the EPA 200.8 (ICP-MS) method of analysis (LOQ =  $1.0 \mu g$ /sample). The 37mm cassettes and single MCE filters were analyzed for Pb mass using modified NIOSH method 7300 (ICP-MS) and included the wiping of the cassettes' interior surfaces (LOQ =  $0.5 \mu g$ /sample). A detailed description of all modifications of analytical methods can be found in Appendix A. Two blank 37mm cassettes and single MCE filters was submitted with every batch for analysis as media and field controls. All results were reported in mass per sample units of measure. The investigators administered qualitative LeadCheck<sup>®</sup> tests (threshold =  $1 \mu g$  on hard surfaces) and assessed the results of on-site following the test instructions.

#### **Statistical Analysis**

The investigators used MS Excel to calculate the percent reduction of Pb for each swatch in both sub-samples using the formula:

$$d = \frac{X_B - X_A}{X_B} \times 100\%$$

where *d* - Pb reduction, %;

 $X_B$  - Pb mass on swatches before exposure in AS, which is equal to Pb load;  $X_A$  - Pb mass on swatches after exposure in AS.

The investigators analyzed the Pb reduction data using SPSS<sup>®</sup> Statistics version 22 software. The Kolmogorov-Smirnov test and visual examination of frequencies

histograms were used to assess the normality of data distribution. Descriptive statistics for values of Pb reduction on swatches in both sub-samples of swatches were obtained. To examine the effects of transportation and handling on Pb load, the investigators conducted two-tailed ( $\alpha = 0.05$ ) t-tests for samples of calculated differences between the Pb load and Pb mass measured on transportation and handling control swatches.

The Piface version 1.76 application (32) and the greatest standard deviation (SD) and the lowest reduction from the two sub-samples were used to determine the minimum sample size for the main study.

## RESULTS

The visual examination of frequency distribution of Pb reduction in each subsample discovered no significant deviations from normality (Figure 29). Both the Kolmogorov-Smirnov and Shapiro-Wilco tests of normality suggested a normal distribution of data (Figure 30). The mean Pb reduction for sub-samples  $n_{11}$  and  $n_{33}$  was 48.9% and 56.1% respectfully (Figure 31). Descriptive statistics for both sub-samples are summarized in Table 5. Swatches transportation and handling were not found to have a statistically significant effect on Pb load (p-values of 0.220 for both t-tests).



Figure 29. Frequency distribution histograms for sub-samples  $n_{11}$  and  $n_{33}$  in the pilot.

		Kolmogorov-Smirnov <sup>a</sup>			Shapiro-Wilk		
	Sub-samples	Statistic	df	Sig.	Statistic	df	Sig.
Reduction	n11	.137	13	.200*	.959	13	.746
	n33	.173	13	.200*	.922	13	.269

\*. This is a lower bound of the true significance.

a. Lilliefors Significance Correction

Figure 30. Tests of normality for the data in the pilot.



Figure 31. Boxplots for sub-samples  $n_{11}$  and  $n_{33}$  in the pilot.

			n11	n33
Esse avies a setal	Distance from wal	13	8	
experimental	Point air velocity,	1,800	6,900	
setup	Angle of impact, d	0	90	
	Sub-sample size	13	13	
	Mean	48.86	56.11	
	Std. error	0.830	0.660	
	Rel. std. error, %	1.70	1.18	
<b>D</b>	95% CI for mean	lower	47.05	54.67
Descriptive	95% CI 101 mean	upper	50.67	57.55
nercent Ph	Median	48.7	56.0	
reduction	Variance	8.95	5.67	
	SD	2.991	2.381	
	RSD, %	6.12	4.24	
	Minimum	43.2	53.3	
	Maximum	53.3	60.6	
	Range		10.1	7.3

Table 5. Descriptive statistics for sub-samples  $n_{11}$  and  $n_{33}$  in the pilot.

The results of the breakthrough Pb collection are summarized in Table 6. With exception of one sample, all breakthrough Pb amounts collected on 37mm cassettes and filters were below LOQ of 0.5  $\mu$ g/sample. The 37mm cassette that collected the breakthrough Pb from five swatches in sub-sample  $n_{33}$  (Method C) contained 0.75  $\mu$ g of Pb. Backpressure and filtered air supply controls were below LOQ. Both qualitative Pb tests were positive.

Sub- sample	Variables	Breakthrough collection method	ACU swatches	37mm cassettes	Filters	Collected Pb breakthrough µg/sample
n <sub>11</sub>	1,800 fpm 0° angle	А	4		4	all < 0.5
		В	4	4		all < 0.5
		С	5	1		< 0.5
n <sub>33</sub>	6,900 fpm 90° angle	А	4		4	all < 0.5
		В	4	4		all < 0.5
		С	5	1		0.75
Controls						
Filtered air supply				1		< 0.5
Backpressure		С	4	1		< 0.5

Table 6. Results of the breakthrough Pb collection in the pilot.

The investigators used the largest observed SD of 3.0 (rounded SD of 2.991 from the sub-sample  $n_{11}$ ) and the lowest observed effect size (Pb reduction) of 48% to determine the minimum sample size for the main study. Using Piface application, the investigators determined that to detect the observed effect size using a two-tailed t-test with at least 0.8 power and a 95% confidence, the minimum sample size of each sub-sample  $n_{ij}$  could be as low as 3 (Figure 32a), which is the lowest sample size recommended for statistical analysis in general. Assuming the main study data will have a higher variance and smaller effect size, the investigators calculated the minimum sample sizes for effect sizes of 25% and 10% with SDs of 6.0 and 9.0. Figures 32b-e show the resulting sample sizes. The investigators decided to use the largest sample size the main study could fund, which was 10 swatches for each sub-sample. Assuming the SD in the main study will be four times the SD observed in the pilot (i.e. SD = 12.0), with n = 10 the study could detect an effect size (Pb reduction) as low as 12% with at least 0.8 power and 95% confidence (Figure 32f).



Figure 32. Calculating the sample size for the main study.

#### DISCUSSION

The pilot has successfully tested the materials and methods developed for the main study and produced statistical parameters necessary for determination of the sample size for the main study. The main challenges have been addressed in the design of the test assembly and protocol. Underlying assumptions have been validated during simulated tests in a laboratory and by the use of controls throughout the study.

The resulting Pb reductions of 48.9 and 56.1 percent were statistically significant and were not affected by transportation and handling during the pilot. Low variability in the resulting data suggests that the procedure of placing swatches in the AS at a point in space corresponding to a selected point air velocity and angle of impact was consistent. Although the investigators anticipated a 48.9% Pb reduction in the sub-sample  $n_{11}$ , a small difference (7.2 %) in Pb reduction between the two sub-samples was rather unexpected. The investigators believed that the two sub-samples used in the pilot would represent the highest and the lowest Pb reduction among all nine sub-samples identified for the main study. If that is the case and 7.2 is the range for Pb reduction in all nine subsamples, then determining a statistically significant difference between sub-samples in the main study could be problematic. Therefore, for each sub-sample in the main study the investigators decided to use the maximum sample size the study could fund, which was 10. With the same SD = 3.0 and the sub-sample size 10, the main study could determine a statistically significant (power 0.8,  $\alpha = 0.05$ ) difference between sub-samples as low as 3 percent.

The results of the breakthrough Pb collection were unexpected. All samples with the exception of one were below the LOQ of 0.5 µg. Based on a previous study, the investigators expected the breakthrough to be ca. 1% of the Pb load, which should have produced breakthrough samples in the range of 1 µg of Pb with Methods A and B, and 5 µg of Pb with Method C. Low breakthrough results could be explained with potentially overestimated breakthrough expectation, which was based on a study with Pb loads three orders of magnitude greater than the load used in this pilot. In addition, filters placed underneath swatches in Method A may have created a resistance to the breakthrough airflow thus reducing the amount of Pb breakthrough. Positive qualitative Pb tests on interior surfaces of the chamber clearly demonstrated that some Pb particles had adhered to chamber's walls thus resulting in lower than expected breakthrough amounts collected with Methods B and C.

The only breakthrough Pb sample above LOQ was collected on one 37mm cassette from 5 swatches in the sub-sample  $n_{33}$  using Method C and contained 0.75 µg of Pb. Although one positive sample did not allow describing the breakthrough quantitatively with any statistical significance, this result together with positive qualitative Pb tests on chamber's walls allowed the investigators to conclude that the Pb breakthrough did take place during the pilot. To further examine the Pb breakthrough during the main study, out of three techniques tested in the pilot the investigators selected Method C as the most capable of collecting breakthrough samples with amounts of Pb quantifiable with the selected analytical methods. To increase the chance of collecting breakthrough Pb amounts above the LOQ, the investigators decided to modify the method by increasing the number of swatches from 5 to 10. A modified Method C should

also include a quantitative wipe test to account for Pb particles settled/attached to interior surfaces of the chamber.

## CONCLUSION

The materials and methods tested in the pilot enabled the exposure of Pb loaded ACU swatches to the cleaning action of the AS in a consistent manner resulting in statistically significant data with low variability. Based on descriptive statistics of the two sub-samples examined in the pilot, the investigators selected the sample size of 10 swatches for each sub-sample in the main study. Results of the breakthrough collection confirmed the presence of the breakthrough during the pilot but were limited and did not support a quantitative description. Potential effects of the backpressure and filtered air supply on Pb breakthrough collection were examined and found negligible with appropriate controls. The method of collecting the breakthrough Pb from multiple swatches to one 37mm cassette was selected for the main study as the most viable of collecting breakthrough Pb samples above the LOQ of the selected analytical method. Some amounts of the breakthrough Pb were unaccounted during collection due to adhesion of Pb particles to the chamber's interior surfaces and this issue should be addressed in the main study.

#### RECOMMENDATIONS

The investigators recommend increasing the number of swatches used in the breakthrough collection Method C from 5 to 10 to ensure the resulting breakthrough Pb samples are above the LOQ of the selected analytical method. In addition, a quantitative wipe test should be incorporated into Method C technique to account for Pb particles settled/attached to interior surfaces of the chamber.

# CHAPTER 4: The Effects of Select Air Velocities and Angles of Impact on Lead Removal from Army Combat Uniform Swatches inside an Air Shower

# ABSTRACT

This is the first study to examine the efficiency of air showers in removing a gunshot-residue-specific lead from Army Combat Uniform swatches loaded with amounts of lead in the order of micrograms per square centimeter. The study also examined the breakthrough of lead across Army Combat Uniform swatches. The overall study design entailed the exposure of lead-loaded swatches inside the air shower to point air velocities of 6,900, 4,100, and 1,800 fpm at the 0-, 45-, and 90-degree angles of impact. Analysis of lead mass remaining on swatches after the exposure indicated the percent of lead removed. The breakthrough lead was isolated inside the constructed breakthrough catchment chamber and was collected by pumping the chamber's air through 37mm filter cassettes and by wiping the inner surfaces of the chamber. The observed lead reduction ranged from 8.2% to 56.1% and was positively correlated with the amount of lead load (r = 0.885, p < 0.000, 2-tailed  $\alpha$  = 0.01). The study found a positive correlation (r = 0.416, p < 0.000, 2-tailed  $\alpha$  = 0.01) between selected point air velocities and observed lead reduction. The angles at which air velocities fell on swatches did not have a significant effect on lead reduction. The study confirmed the presence of lead breakthrough during air shower application but was unable to describe it quantitatively and statistically. The study results are limited to the air shower model, Army Combat Uniform material, characteristics of lead load, study approach, and methods.

## INTRODUCTION

Lead (Pb) is a well-known toxicant and the exposure to it on indoor firing ranges (IFR) presents a health risk to both employees and shooters (5). Firing Pb-containing ammunition produces airborne Pb particles creating an inhalation hazard. Contamination of hands and clothing contributes to Pb ingestion and spreads the Pb outside firing ranges creating a risk of Pb "take-home exposure" for family members (5; 25). An estimated 16,000-18,000 indoor ranges and 20 million shooters in the United States suggest a significant number of people potentially exposed to Pb (43; 46). New data showing toxic effects of Pb at much lower levels than previously found (44) has indicated the need to intensify efforts in reducing the Pb exposure. In recent years, several US Army IFRs have implemented a new control measure – the air shower (AS) – intended to reduce Pb contamination on clothing of service members and range employees.

Since AS's first appearance as a part of the cleanroom industry in 1960s, these devices have been widely used for contamination control purposes in various dust sensitive industries such as microelectronics, automobile painting, pharmaceutical, biomedical, optics, and food and drink (27). To a lesser extent, ASs have been used or recommended for control of various occupational exposures such as Pb exposure in secondary Pb smelters (62), animal allergens exposure among laboratory personnel (29), and silica exposure of construction and mining workers (24).

The concept of an AS is to blow high velocity air streams at employee's uniform to dislodge any particulate matter and then quickly remove the "dirty" air from the booth before the dust settles back on uniform. The major components of an AS are an enclosure in the form of a booth or tunnel, a powerful fan to create a high flow of air, nozzles in walls and/or ceiling to direct airflow inside the booth, and an air return vent with HEPA

filter to remove the contaminant from the air before it recirculates in the AS. Despite seemingly simple concept and long history of application, currently there is no international or United States standard for ASs. The design and performance parameters of ASs vary from one manufacturer to another.

The body of knowledge on AS performance and effectiveness mostly consists of studies conducted by AS manufacturers, which have not been published in peerreviewed, scientific literature. The results of these studies were usually presented during cleanroom industry society meetings and conferences and their proceedings were not always available for review for this study. The reviewed studies lacked details on methods and materials and presented little statistical data. The AS parameters, study designs, and analytical approaches varied among investigations and therefore produced erratic results and did not allow for comparison.

The main AS performance parameter identified by manufacturers was the produced air velocity measured at the face of a nozzle. Most AS manufacturers recommend nozzle air velocities between 4,500 and 7,500 fpm. Some AS models produce as little as 2,000 fpm while others offer as much as 9,000 fpm. AS studies have shown that higher air velocities result in greater reduction of the contaminant (29; 51), although some studies indicated that increasing the nozzle air velocity above 4,000 fpm did not produce a significant additional benefit (26). Other performance parameters found to affect AS's efficiency were the angle of air [impact] application, design and number of nozzles, and duration of the cleaning cycle (26; 36; 61).

The efficiency of an AS in removing a contaminant from clothing also depended on the type of garment and characteristics of a contaminant used in a study. Slick fabrics

such as polyester retained 80% less of contaminant and resulted in greater removal efficiency than rough materials such as cotton (29). Studies assessing AS efficiency in removing contaminants by various particle size showed that some AS models caused between 63% and 97% reduction in particles larger than 5  $\mu$ m (37; 51; 61), but as low as 0% reduction for particles < 2  $\mu$ m in size (26).

Prior studies assessed the efficiency of ASs using various garment loading techniques and amounts of reference materials. Most studies used the Arizona test dust or Japanese Industrial Standard (JIS) Z8901 dust loaded on garment samples in the order of milligrams per square centimeter using a gravitational settling method (40; 61). Some studies exposed clean garments for hours or days to various environments such as outdoors, offices, metal shops, production arears, smoking rooms, etc. to accumulate dust particles present in those settings (17; 37; 51). However, the efficiency of ASs in removing Pb particles with unique morphology and size found in a gunshot residue (GSR) on IFRs, as well as the effects of particle retention by garments used on US Army's IFRs have not been examined.

The characteristics of Pb particles in GSR and the amount of contamination on clothing at IFRs differ from parameters used in previous AS studies. Most GSR particles are spheroidal with a smooth, fuzzy, or scaly surface (58) and represent various combinations of Pb, antimony (Sb), and barium (Ba) with estimated particle density ranging between 7.22 and 11.34 g/cm<sup>3</sup> (7), which is several times higher than density of dust used in prior AS studies. By count and by mass, anywhere between 60 and 95% of Pb-containing GSR particles are <4  $\mu$ m in size (8; 10; 30; 38). The quantitative description of Pb contamination on clothing on IFRs have not been found in the literature

reviewed for the study. However, the results of several studies measuring Pb on various surfaces inside and outside IFRs and qualitatively examining Pb contamination of shooters' and range employees' clothing suggest that the degree of clothing contamination at IFRs could be in the order of micrograms per square centimeter (54; 55).

The literature review for this study found one evaluation of AS efficiency that used Pb as a reference material. Simonson and Mecham (62) loaded three types of fabric samples with an average of 1.4 mg of Pb oxide per square centimeter of the swatch surface and exposed them to air velocity of 5,500 fpm. The results showed the Pb reduction of 23%, 48%, and 69% for a twill-weave coverall fabric, plain-weave shirting fabric, and non-woven disposable fabric (Tyvek<sup>®</sup>) respectfully. The study also registered a Pb breakthrough across all three fabrics in amounts ranging from 0.2% to 1.4% of the initial Pb load on swatches.

The purpose of this study was to examine the effects of various air velocities and angles of impact on removal of GSR-specific Pb from ACU swatches inside an AS and to examine a potential breakthrough of Pb across the ACU swatches. The study intended to be the first in a series of studies aimed at evaluating the overall effectiveness of ASs as a Pb exposure reduction measure on IFRs.

## METHODS

The overall study design entailed placing ACU swatches loaded with a known amount of Pb inside an AS and exposing the swatches to the cleaning action of the device. Analysis of Pb mass remaining on swatches after the exposure in the AS indicated the percent of Pb removed. By placing swatches at selected distances (points)

away from a nozzle-bearing wall and at selected angles to air streams produced by the nozzles, the investigators examined the effects of the select point air velocities and angles of impact on Pb removal. To examine the Pb breakthrough, the investigators constructed a test assembly designed to isolate and capture the breakthrough Pb particles pushed by the air streams across ACU swatches during the cleaning cycle.

The study was conducted in two phases: the pilot and main study. The investigators conducted the pilot to test the developed materials and methods and to obtain statistical parameters for determination of the minimum sample for the main study. For the purpose of clarity, the materials and methods reported here describe the parameters and procedures used in the main study. The full description of the pilot can be found in Chapter 3. Some aspects and results of the pilot are reported here for reference and to facilitate the discussion.

### **Air Shower Description**

The study used an AS model CAP701KD-ST-4954 (Clean Air Products, Minneapolis, Minnesota) installed on an indoor firing range. The booth internal L x W x H dimensions were 52 x 40 x 86 inches. The AS had 36 anodizes aluminum nozzles with 17 nozzles on both sides and 2 nozzles on the ceiling. The nozzles were 1¼ inches (30 mm) in diameter protruding inside the booth for 1½ inches (38 mm) at a 90-degree angle to the walls' surface. An average nozzle air velocity reported by the manufacture for this model was 7,800 fpm, which was verified during the pilot (Table 3, Chapter 3) to ensure the AS performs at manufacturer-recommended settings. The air return was in the floor of the AS covered with a steel grate to protect a pre-filter followed by a HEPA filter. The AS was set for a 15-second cleaning cycle activated with a push of the button located inside the AS booth. Figure 13, Chapter 3 illustrates the AS used in the study.

#### **Point Air Velocities**

The investigators used a multi-functional ventilation meter VelociCalc<sup>®</sup> model 9565-P with a heated wire anemometer probe 966 (TSI Inc., Shoreview, Minnesota) to measure air velocities in this study. The air velocities of interest for this study were identified during the pilot based on estimated distances "points" between the AS's nozzle-bearing walls and an ACU that are likely to take place inside the booth during a standard cleaning procedure. The resulting point air velocities selected for the study were 6,900, 4,100, and 1,800 fpm, which were measured at 8, 13, and 19 inches off the AS's wall respectfully.

## **Angles of Impact**

The angles of impact used in this study were selected during the pilot based on the observation of a body position and movement during the standard cleaning procedure inside the AS. As a volunteer dressed in ACU stood in an up-right position inside the AS, majority of the ACU surface was in the vertical plane during the cleaning process. Due to the individual's rotating movement in the AS during the cleaning cycle, air streams coming out of the air nozzles fell on vertical surfaces of ACU at angles changing from 0 to 90 to 180 degrees. The investigators selected the 0-, 45-, and 90-degree angles of impact for the study; a use of angles from 90 to 180 degrees would result in an air stream force of the same magnitude but in an opposite direction and therefore were not considered. Thus, during the study, the investigators placed ACU swatches inside the AS

vertically and at the selected angles to air streams coming from the nozzles as shown in the example in Figure 15, Chapter 3.

#### **Sample Size Estimation**

The selected three point air velocities of 1,700, 4,200, and 6,900 fpm and three angles of impact of 0, 45, and 90 degrees formed nine combinations of the two variables used in this study. The ACU swatches exposed to each combination of variables were annotated as sub-samples  $n_{ij}$  of the total sample size n as depicted in Figure 16, Chapter 3. In the pilot, the investigators used two combinations corresponding to the sub-samples of ACU swatches  $n_{11}$  and  $n_{33}$ , which were expected to result in the lowest and highest Pb reduction and Pb breakthrough. Based on descriptive statistics of these two sub-samples (Table 5, Chapter 3) and a conservative assumption that a standard deviation (SD) in the main study will be four times the SD observed in the pilot, the investigators selected the sample size of 10 swatches for each sub-sample  $n_{ij}$ . This sample size would allow detecting an effect size (Pb reduction) in the main study as low as 12% with at least 0.8 power and 95% confidence.

#### **Pb-Loaded ACU Swatches**

The investigators used a permethrin treated 50% cotton, 50% nylon fabric meeting the requirements of MIL-STD-44436 (Class 8) to prepare ACU swatches. The resulting ACU swatches (Figure 17, Chapter 3) were approximately 45 mm in diameter with the exposed surface of 35 mm in diameter and an area of 9.6 cm<sup>2</sup>. The investigators loaded ACU swatches with GSR-specific Pb by firing a Pb-containing ammunition inside a sealed chamber and allowing the produced GSR to settle on ACU swatches placed inside the chamber. The investigators prepared two batches of 84 and 72 swatches to be

used in this study with Pb loads of 77.9 and 82.7  $\mu$ g of Pb mass per swatch respectfully. The swatches were packaged in 13 paper boxes each containing 12 swatches. The details of ACU swatch preparation, loading, packaging, and transportation to the test site have been described in Chapter 2.

## **Test Assembly**

The main purpose of the test assembly was to secure an ACU swatch during exposure to high air velocities inside the AS and to facilitate the collection of the breakthrough Pb. The assembly consisted of a breakthrough catchment chamber, filtered air supply system, and breakthrough collection pump. The major components of the test assembly are depicted on Figure 18, Chapter 3 and their design and purpose are explained below.

## **Breakthrough Catchment Chamber**

The main purpose of the chamber was to secure a swatch and to isolate the breakthrough Pb particles pushed across a swatch into the chamber by point air velocities in the AS. The investigators devised the breakthrough catchment chamber (the "chamber") shown on Figures 19 and 20, Chapter 3 using a chrome plated brass outlet tee (P/N 540TTK, Keeney Mfg. Co., Newington, Connecticut). A latching clamp from a hinged glass jar was placed around the chamber's top orifice and was used to secure an ACU swatch to the chamber during the test. To prevent high velocity air streams in the AS from pushing a swatch inside the chamber during the test, the investigators fabricated a swatch-supporting base. The base was made from a piece of a 24-gage chrome plated brass tubing (P/N 30304CCP, Keeney Mfg. Co., Newington, Connecticut) and a stainless steel mesh from a sink strainer basket (P/N SF3511, BrassCraft Mfg. Co., Novi, MI). To

ensure a tight seal between the orifice's rim, supporting base, and a swatch, the investigators used two rubber gaskets cut out of the rubber [gasket] sheet (P/N 59849, Danco Inc., Irving, Texas). One gasket was placed between the orifice and the base and another between the base and a swatch washer. To enable latching clamp's firm grip on a swatch, the investigators used a rigid galvanized steel conduit-reducing washer (P/N 68510, Halex Co., Cleveland, OH). All together the washer, swatch, supporting base, and rubber gaskets formed a "stack" referenced later in this study.

The chamber's side orifice was utilized as the attachment site for a 37-mm polypropylene air sampling filter cassette. To facilitate a quick and tight fitting of cassettes to the chamber, the investigators used a mid-section of a three-part 37-mm polyurethane cassette as a connecting link. The mid-section was inserted inside the chamber's side orifice and secured with a nut. The third end of the chamber was fitted with a threaded polyvinyl chloride (PVC) coupling to enable the attachment of the filtered air supply system.

## Breakthrough Collection

Unable to find a standard method of a breakthrough collection applicable to the design of this study, the investigators developed and tested three potential techniques during the pilot to select the optimum one for the main study. Two techniques involving the collection of breakthrough Pb particles from single swatches were rejected because the collected Pb amounts were below the limits of quantification (LOQ) of analytical methods available for this study. The third method involving the collection of one breakthrough sample from several swatches produced an above-LOQ result and was selected for the main study with some modifications. Based on recommendations of the

pilot, the selected technique entailed the collection of both the airborne and adhered fractions of breakthrough Pb particles.

The airborne fraction of breakthrough Pb particles was collected by pumping the chamber's air through a filter during the cleaning action of the AS while particles were still suspended inside the chamber. The investigators attached a 37-mm closed face polyurethane cassette with MCE 0.8  $\mu$ m pore size filter to the chamber's side orifice as was described earlier. One filter cassette collected the airborne breakthrough Pb from all 10 swatches allocated for each sub-sample  $n_{ij}$ . The air was collected by the GilAir5<sup>®</sup> pump (Sensidyne, Clearwater, FL) connected to the cassette using a <sup>1</sup>/<sub>4</sub>-inch nylon tubing. The pumping rate was selected at 3.8 L/min to overcome the breakthrough airflow across a swatch, which was estimated during the pilot. The breakthrough collection pump was turned on for the entire duration of the AS's cleaning cycle (15 seconds) and an additional 10 seconds after the AS shut off while air streams fading for ca. 5-7 seconds after AS's de-activation were potentially still causing the breakthrough effect. Throughout the pilot, the pump's flow rate was periodically verified with a flow meter DryCal<sup>®</sup> Defender 510-M, and as long as the flow rate was within  $\pm$  5 % of the intended value of 3.8 L/min, no adjustments were made. The total volume of air pumped through a cassette had no relevance for the breakthrough collection techniques and therefore was not reported. However, collecting the chamber's air at a flow rate exceeding the breakthrough airflow across a swatch would cause a negative pressure inside the chamber and would create a backpressure on a swatch potentially increasing the airflow across a swatch and the breakthrough of Pb. To minimize the effects of the pumping action on Pb breakthrough, the investigators incorporated a filtered air supply system described later in

this study. Figure 22, Chapter 3 depicts the collection of the airborne fraction of the breakthrough Pb particles.

During the pilot, the investigators discovered that some amount of breakthrough Pb particles had adhered to the chamber's walls and was not collected by the filter cassette. In the main study, the adhered fraction of breakthrough Pb particles was collected by wiping the internal surfaces of the chamber after the exposure to a cleaning action in the AS. After all 10 swatches from a sub-sample  $n_{ii}$  have been exposed to a corresponding combination of variables, the chamber was separated from the test assembly and taken to the admin area. There the investigators took the chamber apart and wiped the chamber's internal surfaces and select parts with a Ghost Wipe (Environmental Express, Charleston, SC). The investigators unfolded the wipe and using one side of it wiped those surfaces of swatch support base, mid-section of 37mm polyurethane cassette, and the top section of the 37mm polypropylene cassette that were inside the chamber and could have accumulated breakthrough Pb particles. Then, the investigators folded the wipe in half (always on a side that was previously used) and pushed the wipe from the chamber's one side orifice through the other using the Sponge<sup>®</sup> bottle brush (Munchkin Inc., Van Nuys, CA) as shown on Figure 33. [Brush's side bristles were previously cut off to prevent the contact with the chamber's walls.] Then, the investigators folded the wipe again and repeated the wiping from one orifice to the other. The investigators then folded the wipe again and while resting it on two fingers wiped the "neck" of the top orifice. Wiping of the chamber's neck was performed twice, after which the investigators placed the wipe inside a 50ml plastic vial for analysis.



Figure 33. Collecting Pb breakthrough on the chamber's walls using a GhostWipe. *Filtered Air Supply System* 

The filtered air supply system was designed to minimize the effects of the pumping action on Pb breakthrough by eliminating the negative pressure inside the chamber during the breakthrough Pb collection and was validated during the pilot. The major components of the filtered air supply system are the vacuum pump, air tank, and hose connecting the system to the chamber. The vacuum pump available for the study was model 1531-107B-G557X (GAST Mfg. Corp., Benton Harbor, Michigan). The investigators fabricated an air tank using a 3.8L Sterilite<sup>®</sup> Ultra-Latch<sup>®</sup> plastic food container (P/N 0425, Sterilite Co., Townsend, Massachusetts). A vacuum HEPA filter Arm & Hammer<sup>®</sup> Dirt Devil<sup>®</sup> F2 (Church & Dwight Co. Inc., York Township, Pennsylvania) was installed inside the container with the filter's inlet connected to the outside through the container's wall with a PVC elbow, threaded fitting, two steel washers, and two rubber gaskets. The components of the air tank are depicted on Figure 25, Chapter 3. The vacuum pump's output vent was connected to the air tank's filter inlet with a ¼-in braided PVC hose. To connect the air tank to the chamber, the investigators used a 1-7/8" x 7' universal vacuum cleaner hose RIDGID<sup>®</sup> (P/N VT1720, Ridgid, Inc., Newark, Delaware) fitted on one side with a threaded PVC pipe connector. The investigators fitted one of the tank's walls with a connector for the RIDGID<sup>®</sup> hose. On the opposite wall, the investigators cut out an air vent ca. 2 inches in diameter.

## Grounding the Chamber

The investigators used a copper wire to ground the chamber during the test reducing the amount of breakthrough Pb particle adhered to the chamber's walls due to electrostatic force. The investigators attached one end of the wire to the chamber and connected the other end to the ground terminal of a wall electrical outlet.

# **Test Procedure**

The main study took place at an indoor firing range equipped with the AS described earlier. The AS was located in a hallway connecting the lobby and the range as shown on Figure 27, Chapter 3. In the lobby, the investigators set up an administrative area where the equipment and swatches were stored and handled. The co-located restroom was used for equipment cleaning. At the time of the pilot, the range was undergoing a renovation and no shooting took place during the study.

# Test Assembly Setup inside the Air Shower

The test assembly setup inside the AS is shown on Figures 28, Chapter 3 and explained below. The chamber with attached blank ACU swatch was mounted on a tripod. The investigators placed the tripod inside the AS in a manner to position the swatch in the center of the booth (26 inches away from each door and 43 inches away from each the floor and the ceiling) and at a distance from the nozzle-bearing wall

corresponding to a selected point air velocity, e.g. 8 inches for the point air velocity of 6,900 fpm. Using a Johnson rafter angle square, the investigators rotated the chamber to achieve a desired angle of impact. The vacuum pump and air tank of the air supply system were taken through the AS's back door and placed outside the AS on the backside facing the range. The investigators connected the air tank to the chamber with the RIGID<sup>®</sup> hose routed through the back door of the AS. To enable the vacuum pump control, the investigators ran an electric extension cord from the administrative area through the AS to the backside of the device where the vacuum pump was located. The back door was closed and the gap caused by the hose was sealed with a plastic sheeting and adhesive tape. The grounding wire attached to the chamber was routed through the front door to the electrical outlet in the lobby. The investigators then attached a blank 37mm filter cassette to the chamber and routed the nylon tubing through the AS's front door to the breakthrough collection pump located in the administrative area.

## **Test Protocol**

The testing began by purging the air supply system and the chamber off potentially Pb-contaminated air that entered the assembly during its installation in the AS. The purging was performed every time the assembly was taken apart for the wipe test and cleaning. The blank swatch was removed and the chamber's orifice was sealed with adhesive tape. Both vacuum and breakthrough collection pumps were turned on for ca. 5 min to purge the air in the chamber, hose, and air tank. Once the purging was complete, the position and angle of the chamber inside the AS were again verified and the investigators proceeded with the test.

The blank 37mm cassettes used during purging was replaced with a new cassette intended to collect the airborne fraction of the breakthrough Pb during the test. In the admin area, the investigators stacked the rubber gaskets, the swatch support base, and a Pb-loaded swatch and carried the stack to the AS. The adhesive tape on chamber's top orifice was removed and the stack with a Pb-loaded swatch was secured immediately to the top orifice of the chamber. The AS's front door was closed and both pumps were turned on simultaneously. The investigators then activated the AS for the full 15-second cleaning cycle. At the end of the cleaning cycle, the assembly's pumps were left operating for additional 10 seconds while air streams fading for ca. 5-7 seconds after AS's de-activation were potentially still producing cleaning and breakthrough effects. Once both pumps were turned off simultaneously, the investigators opened the front door, removed the stack with the swatch, and covered the orifice immediately with adhesive tape. The stack was then taken to the admin area where the investigators cut the zip tie holding the swatch to the washer, folded the swatch in half with forceps, and placed it inside a 50-ml plastic vial for analysis. The investigators then randomly selected the next Pb-loaded swatch from the batch, formed the stack, attached it to the chamber, and exposed the swatch to the cleaning action of the AS in the same manner.

The investigators continued the sequence described above exposing all 10 swatches in a sub-sample  $n_{ij}$ . The same 37mm cassette remained attached to the chamber for the entire duration of a sub-sample  $n_{ij}$  test collecting the airborne breakthrough Pb particles from all 10 swatches in the sub-sample. Once all 10 swatches in a sub-sample  $n_{ij}$ have been exposed to the corresponding combination of point air velocity and angle of impact, the investigators disconnected the air supply hose from the chamber, detached the
chamber from the tripod and carried it to the administrative area for the collection of the adhered breakthrough Pb particles. As was described earlier, one wipe was used to collect the adhered breakthrough Pb particles from all 10 swatches in a sub-sample.

Following the breakthrough collection, the chamber with all its components was washed using LeadOff<sup>®</sup> cleaning solution (Hygenall Corp., Huntsville, AL) and dried with paper towels. The investigators then re-assembled the chamber with a blank swatch, attached the chamber to the tripod, and rotated the chamber to achieve another angle of impact for the same sub-sample  $n_{ij}$ , or re-located the tripod in the AS to position it at the point in space corresponding to the next point air velocity of interest. Once the investigators verified the position of the blank swatch, the test protocol was repeated starting with the test assembly purging.

#### **Controls**

The 13 boxes with Pb-loaded ACU swatches used in the main study were transported in a vehicle from the loading site to the study site located 350 miles away. At the arrival to the study site, the investigators collected one randomly selected swatch from each box for analysis to examine the effect of transportation on Pb load.

The investigators collected 9 handling control swatches to examine the effect of swatch handling during the test protocol on Pb load. The investigators exposed one randomly selected Pb-loaded swatch to the full protocol under test conditions of each sub-sample  $n_{ij}$ , with exception of the AS's cleaning action.

The investigators collected 4 filtered air supply controls throughout the study to confirm the assumption that the filtered air supply system equipped with a HEPA filter would produce an air supply containing amounts of Pb negligible to this study. The air

samples were collected in the same manner as the airborne breakthrough Pb particles. The investigators attached a new 37mm cassette to the chamber, turned on both the vacuum and breakthrough collection pumps, and sampled the air supplied to the chamber at a flow rate of 3.8 L/min for 15-30 minutes. The flow rate was verified with the flow meter DryCal<sup>®</sup> Defender 510-M before and after the sampling.

Three backpressure controls were conducted throughout the study. The purpose of the control was to ensure the developed test assembly allowed the collection of the breakthrough without causing a backpressure on a swatch. In an experimental setting inside the AS, the investigators conducted an airborne breakthrough Pb collection from 10 Pb-loaded swatches to one 37mm cassette without activating the AS. Each of the 10 swatches remained attached to the chamber for a breakthrough collection duration of 30 seconds (5s before AS activation + 15s AS cleaning cycle + 10s waiting period).

To assess the effectiveness of chamber cleaning, the investigators conducted a control wipe test following each cleaning before the chamber was put together for the next test.

#### **Analytical Methods**

The investigators delivered all samples to a certified lab for analysis. ACU swatches were analyzed for Pb mass using a modified EPA 3052 method of digestion and the standard EPA 200.8 (ICP-MS) method of analysis (LOQ =  $1.0 \mu g$ /sample). The 37mm cassettes and single MCE filters were prepared and analyzed for Pb mass using modified NIOSH method 7300 (ICP-MS) and included the wiping of the cassettes' interior surfaces (LOQ =  $0.5 \mu g$ /sample). The wipes were prepared using a modified ASTM 1644 method of digestion and analyzed with modified EPA 200.8 (ICP-MS)

method of analysis (LOQ = 0.5  $\mu$ g/sample). For swatches, wipes, and air filter samples, results from the instrument were reported in  $\mu$ g/L and then converted to  $\mu$ g/sample as follows:  $\mu$ g/sample =  $\mu$ g/L (sample) x final vol (L) x dilution factor. A detailed description of all modifications of analytical methods can be found in Appendix A.

#### **Statistical Analysis**

To randomly select a swatch for a sub-sample or control test, a set of two random numbers were used to first identify the box and then the position of a swatch in the box. Random numbers were generated using the RANDBETWEEN function in MS Excel<sup>®</sup>.

The investigators used MS Excel to calculate the percent reduction of Pb for each swatch in each sub-sample  $n_{ij}$  using the formula:

$$D_{Pb} = \frac{X_B - X_A}{X_B} \times 100\%$$

where  $D_{Pb}$  - Pb reduction, %;

 $X_B$  - Pb mass on the swatch before exposure in AS, which is equal to Pb load;  $X_A$  - Pb mass on the swatch after exposure in AS.

The Pb breakthrough was expressed in percent of Pb breakthrough amount to the initial Pb load on swatches. To calculate the percent Pb breakthrough collected from each sub-sample  $n_{ij}$ , the investigators used the formula:

$$B_{Pb} = \frac{B_f + B_w}{\sum X_B} \times 100\%$$

where  $B_{Pb}$  - Pb breakthrough, %;

 $B_f$  - breakthrough Pb mass collected with the 37mm filter cassette;

 $B_w$  - breakthrough Pb mass collected with the wipe;

 $X_B$  - Pb load on each swatch in the sub-sample

The investigators analyzed the Pb reduction data using SPSS® Statistics version

22 software. The Kolmogorov-Smirnov test and visual examination of frequencies

histograms were used to assess the normality of data distribution. Descriptive statistics and 95% confidence intervals for a mean Pb reduction were obtained for each point air velocity and each angle of impact. The investigators conducted a two-way analysis of variance (ANOVA) of Pb reduction data by point air velocity and blocking for angle of impact. The underlying assumption of equal variances was verified using the Levene's test and visual examination of the error bar charts. The Tukey multiple comparison and Pearson's correlation was performed for significant effects from the overall F-test. To examine the effects of transportation and handling on Pb load, the investigators conducted two-tailed ( $\alpha = 0.05$ ) t-tests for samples of calculated differences between the Pb load and Pb mass measured on transportation and handling control swatches. Since there was only one Pb breakthrough value for each sub-sample, the investigators could not conduct ANOVA but intended to determine the Pearson's correlation coefficient for point air velocities and angles of impact.

#### RESULTS

#### **The Pilot**

The mean Pb reduction for sub-samples  $n_{11}$  and  $n_{33}$  was 48.9% and 56.1% respectfully. Swatches transportation and handling were not found to have a statistically significant effect on Pb load (p-values of 0.220 for both t-tests). With exception of one sample, all breakthrough Pb amounts collected with three techniques tested during the pilot were below LOQ of 0.5 µg/sample. The 37mm cassette that collected the breakthrough Pb from 5 swatches in sub-sample  $n_{33}$  contained 0.75 µg of Pb. Backpressure and filtered air supply controls were below LOQ. Two qualitative Pb tests using the LeadCheck<sup>®</sup> swabs (threshold = 1 µg) on internal surfaces of the chamber

performed after the exposure of 9 swatches in each sub-samples  $n_{11}$  and  $n_{33}$  were positive. Complete results of the pilot can be found in Chapter 3.

#### The Main Study

The visual examination of frequency distributions of Pb reduction and tests of normality suggested a normal distribution of data (Figures 34 and 35). Descriptive statistics for the mean Pb reduction for each point air velocity and each angle of impact have been summarized in Table 7 and depicted on Figure 36. The Levene's test and visual examination of the error bar charts verified the assumption of equal variances (Figure 37). The ANOVA tests of between-subjects effects for point air velocities blocked by angles of impact showed that there was no statistically significant deference between effects of three angles of impact on Pb reduction (p-value = 0.881) while the effects of the three point air velocities were not the same (p-value < 0.000) as shown on Table 8. The Tukey multiple comparison of effects of the three point air velocities on Pb reduction determined that the effect of 6,900fpm velocity was different from the 4,200fpm and 1,800fpm velocities (Tables 9 and 10). The Pearson's coefficient of correlation between the point air velocities and the Pb reduction was 0.416 (p-value < 0.000) (Table 11). The estimated marginal means and 95% confidence intervals for Pb reduction shown in Table 12.



\*. This is a lower bound of the true significance.

a. Lilliefors Significance Correction

Figure 34. Frequency distribution histogram and tests of normality for the Pb reduction data in the total sample n.



Figure 35. Frequency distribution histograms of the Pb reduction data in sub-samples  $n_{ij}$ .

Dependent Variable: Reduction							
Velocity	Angle	Mean	Std. Deviation	Ν			
1,800	0°	13.410	7.1891	10			
	45°	8.230	11.5492	10			
	90°	10.260	6.3033	10			
	Total	10.633	8.6292	30			
4,100	0°	10.860	6.8362	10			
	45°	13.080	10.3763	10			
	90°	12.920	3.8078	10			
	Total	12.287	7.3127	30			
6,900	0°	17.960	4.6150	10			
	45°	18.850	3.8888	10			
	90°	19.780	8.6202	10			
	Total	18.863	5.9106	30			
Total	0°	14.077	6.7878	30			
	45°	13.387	9.9497	30			
	90°	14.320	7.5187	30			
	Total	13.928	8.1147	90			

Table 7. Descriptive statistics of Pb reduction by point air velocities and angles of impact.



Figure 36. Boxplots for Pb reduction data by point air velocity and angle of impact.



Figure 37. The Levene's test and error bar charts for point air velocities and angles of impact.

# Table 8. ANOVA test result of effects of point air velocities on Pb reduction blocked by angles of impact.

Source	Type III Sum of Squares	df	Mean Square	F	Sig.	Noncent. Parameter	Observed Power <sup>b</sup>
Corrected Model	1151.254 <sup>a</sup>	4	287.813	5.195	.001	20.779	.961
Intercept	17458.469	1	17458.469	315.116	.000	315.116	1.000
Velocity	1137.190	2	568.595	10.263	.000	20.526	.984
Angle	14.064	2	7.032	.127	.881	.254	.069
Error	4709.287	85	55.403				
Total	23319.010	90					
Corrected Total	5860.541	89					

Dependent Variable: Reduction

a. R Squared = .196 (Adjusted R Squared = .159)

b. Computed using alpha = .05

# Table 9. Multiple comparison between effects of the three point air velocities on Pb reduction.

		Mean			95% Confidence Interval	
(I) Velocity	(J) Velocity	Difference (I-J)	Std. Error	Sig.	Lower Bound	Upper Bound
1,800	4,100	-1.653	1.9219	.667	-6.238	2.931
	6,900	-8.230	1.9219	.000	-12.815	-3.645
4,100	1,800	1.653	1.9219	.667	-2.931	6.238
	6,900	-6.577	1.9219	.003	-11.161	-1.992
6,900	1,800	8.230	1.9219	.000	3.645	12.815
	4,100	6.577	1.9219	.003	1.992	11.161

Dependent Variable: Reduction Tukey HSD

Based on observed means.

The error term is Mean Square(Error) = 55.403.

\*. The mean difference is significant at the .05 level.

## Table 10. Grouping of the effects of point air velocities.

Tukey HSD<sup>a</sup>

		Subset for alpha = .05				
Velocity	N	1	2			
1,800	30	10.633				
4,100	30	12.287				
6,900	30		18.863			
Sig.		.667	1.000			

Means for groups in homogeneous subsets are displayed. Based on observed means.

The error term is Mean Square(Error) = 55.403.

a. Uses Harmonic Mean Sample Size = 30.000.

#### Table 11. Correlation between point air velocities and Pb reduction.

		Reduction	Velocity
Reduction	Pearson Correlation	1	.416
	Sig. (2-tailed)		.000
	Ν	90	90
Velocity	Pearson Correlation	.416**	1
	Sig. (2-tailed)	.000	
	Ν	90	90

\*\*. Correlation is significant at the 0.01 level (2-tailed).

Table 12. Estimated marginal means and 95% confidence intervals for Pb reduction.

			95% Confidence Interval				
Velocity	Mean	Std. Error	Lower Bound	Upper Bound			
1,800	10.633	1.359	7.931	13.335			
4,100	12.287	1.359	9.585	14.989			
6,900	18.863	1.359	16.161	21.565			

Dependent Variable: Reduction

Dependent Variable: Reduction

			95% Confidence Interval				
Angle	Mean	Std. Error	Lower Bound	Upper Bound			
0°	14.077	1.359	11.375	16.779			
45°	13.387	1.359	10.685	16.089			
90°	14.320	1.359	11.618	17.022			

Dependent Variable: Reduction

				95% Confidence Interval			
Velocity	Angle	Mean	Std. Error	Lower Bound	Upper Bound		
1,800	0°	13.410	2.367	8.700	18.120		
	45°	8.230	2.367	3.520	12.940		
	90°	10.260	2.367	5.550	14.970		
4,100	0°	10.860	2.367	6.150	15.570		
	45°	13.080	2.367	8.370	17.790		
	90°	12.920	2.367	8.210	17.630		
6,900	0°	17.960	2.367	13.250	22.670		
	45°	18.850	2.367	14.140	23.560		
	90°	19.780	2.367	15.070	24.490		

The estimated marginal means of Pb reduction for point air velocities of 1,800, 4,200, and 6,900 fpm were 10.6, 12.3, and 18.9% respectfully (Figure 38). The estimated marginal means of Pb reduction for 0-, 45-, and 90-degree angles of impact were 14.1, 13.4, and 14.3% respectfully (Figure 39). The lowest estimated mean Pb reduction among all sub-sample was 8.2% and the highest was 19.8%. Swatches transportation and handling during the main study did not have a significant effect on Pb load (p-values of 0.208 and 0.768 respectfully).



Figure 38. Mean Pb reduction and 95%CI for each point air velocity.





Relatively significant difference between the Pb reduction in the pilot and main study warranted additional statistical examination of results. The investigators determined the Pearson's correlation coefficient between Pb load and Pb reduction was 0.885 (Table 13) and conducted a one-way ANOVA with post hoc Tukey comparison between the residual amounts of swatches following the application of AS (Table 14).

		Pb_Load	Pb_Reductio n
Pb_Load	Pearson Correlation	1	.885**
	Sig. (2-tailed)		.000
	Ν	116	116
Pb_Reduction	Pearson Correlation	.885**	1
	Sig. (2-tailed)	.000	
	N	116	116

Table 13. Correlation between Pb load and Pb reduction.

\*\*. Correlation is significant at the 0.01 level (2-tailed).

# Table 14. Post hoc Tukey comparison between the residual amounts of swatches following the application of AS.

Dependent Variable: Pb\_Residual

Tukey HSD

					95% Confidence Interval		
(I) Pb_Load	(J) Pb_Load	J)	Std. Error	Sig.	Lower Bound	Upper Bound	
77.9	82.7	-5.9178	1.3137	.000	-9.038	-2.798	
	109.2	14.2998	1.5351	.000	10.654	17.946	
82.7	77.9	5.9178	1.3137	.000	2.798	9.038	
	109.2	20.2176	1.5351	.000	16.572	23.863	
109.2	77.9	-14.2998	1.5351	.000	-17.946	-10.654	
	82.7	-20.2176	1.5351	.000	-23.863	-16.572	

\*. The mean difference is significant at the 0.05 level.

The results of the breakthrough Pb collection have been summarized in Table 15.

Out of nine 37mm filter cassettes only one sample displayed an amount of Pb above the LOQ: sub-sample n11 (6,900 fpm at 90°) collected 0.79  $\mu$ g of Pb and was unaffected by any of the controls. Two of the wipe test results were above the LOQ but were invalidated by positive backpressure and cleaning controls.

Point	Point Angle Breakthrough Pb on 37mm filter cassettes, µg					/sample	Breakthrough Pb on wipes, µg/sample				
Air	of			Controls					Controls		
fpm	Impact °	Filter	Backpressure (n=3)	Filtered air (n=4)	Field (n=4)	Media (n=3)	Wipe	Cleaning	Backpressure (n=3)	Field (n=3)	Media (n=3)
									1.9		
6,900	90°	0.79					0.74	1.4			
	45°	< 0.5					0.72	0.84			
	$0^{\circ}$	< 0.5					< 0.5	< 0.5			
									0.55		
4,100	90°	< 0.5	a <b>1</b> 1	o]]	11 م	o]]	< 0.5	< 0.5		all	all
	45°	< 0.5	<0.5	<0.5	<0.5	<0.5	< 0.5	< 0.5		< 0.5	<0.5
	$0^{\circ}$	< 0.5					< 0.5	< 0.5			
									<0.5		
1,800	90°	< 0.5					< 0.5	< 0.5			
	45°	< 0.5					< 0.5	< 0.5			
	$0^{\circ}$	< 0.5					< 0.5	< 0.5			

Table 15. Results of the breakthrough Pb collection.

#### DISCUSSION

This study intended to be the first in a series of studies aimed at evaluating the overall effectiveness of ASs as a Pb exposure reduction measure on IFRs. The terms "efficiency" and "effectiveness" have been used in previous AS studies interchangeably although with a different connotation. When using the term "efficiency", most authors referred to how well an AS performed its primary task, which is removing particulate matter of a garment. The use of term "effectiveness" appeared to have a broader meaning encompassing several aspects of AS employment. The effectiveness was viewed by authors as a measure of an AS's ability to achieve the goal for which it was employed (e.g. did the use of the AS decrease the amount of particulate matter introduced by an employee into a clean room). The effectiveness also encompassed other aspects of AS employment such as practicality and cost-effectiveness. Since there is no AS standard or reference defining these terms, the authors of this study applied them as they were most commonly used in prior AS studies.

An assessment of ASs' effectiveness on IFRs could be problematic considering a large number of variables involved in the processes taking place on a firing range, the lack of standardization on AS design and performance, and the absence of regulatory limits of clothing Pb contamination. Such an assessment could likewise be difficult without a full assessment of AS efficiency specific to a particular contaminant and at relevant levels of contamination. Examining AS effectiveness should also assess potential side effects of AS application such as noise exposure, inhalation of re-suspended Pb particles, and spread of Pb on to otherwise clean parts of a body due to Pb breakthrough.

The study reported here intended to obtain some basic knowledge about AS efficiency and examine one of the potential side effects.

The results of the main study showed that Pb reduction is positively correlated with air velocities, which agrees with finding of other studies. At the same time, this study observed a statistically significant (p = 0.003,  $\alpha = 0.05$ ) difference between Pb reductions at air velocities of 4,100 fpm and 6,900 fpm. The 53% increase in reduction (from 12.3% Pb reduction at 4,100 fpm to 18.9% Pb reduction at 6,900 fpm) contradicts with a finding by some authors that increasing air velocity above 4,000 fpm would not produce an additional benefit in reduction (26). Such statement may stand true in cases where application of 4,000 fpm ensures a very high contaminant reduction and therefore increasing the velocity further would not be relatively significant. This implies that the effectiveness of an AS should be examined for each particular application and using a contaminant of concern in relevant amounts as became evident in significantly different Pb reduction results of the pilot and main study.

In the main study, the Pb reduction from swatches loaded with 77.9 and 82.7  $\mu$ g of Pb ranged between 8.2% and 19.7%. The Pb load on swatches used in the pilot was ca. 40% greater (109.2  $\mu$ g) and the resulting Pb reduction in the two examined sub-samples was 48.9% and 56.1%. The significant correlation between the three loads and resulting Pb reductions (r = 0.885, p < 0.000 at 2-tailed  $\alpha$  = 0.01) suggests that greater Pb reductions could be observed with greater loads. In addition, the residual amounts of Pb on swatches after AS application were significantly different between swatches of different loads (all three p < 0.000). This indicates that the difference in Pb reduction was not just due to greater Pb load but also because swatches in different loads retained Pb

differently. Since all ACU swatches were prepared from the same lot, this could potentially be explained with different environmental conditions (air temperature and relative humidity) present during each loading event, which potentially could had an unknown effect on the particle size distribution or adhesion of Pb particles to fabric as was mentioned in Chapter 2.

The means Pb reductions for 0-, 45-, and 90-degree angles of impact were 14.1, 13.4, and 14.3% respectfully and were not significantly different (p = 0.881). Prior studies observed varying effects of angles on a contaminant reduction. Hirasawa et al. (26) have examined the effects of an angle of impact on removal efficiency of dust particles of various size at air velocity of 4,000 fpm and found that angle of impact had effect on removal of particles of aerodynamic size  $\geq 20 \ \mu\text{m}$  and no effect on particles  $\leq 20 \ \mu\text{m}$ . Considering the results of forensic and range studies showing that vast majority of GSR particles are <4  $\mu$ m in size (8; 10; 30; 38), the effects of angles of impact observed in this study are rather expected. Assessing the efficiency of this particular AS model, angles of impact would have a low relevance since the proper cleaning protocol calls for an individual to turn around inside the AS during the cleaning cycle and therefore each part of a uniform would potentially be exposed to air velocities at all three angles examined in this study.

The examination of the Pb breakthrough, on the other hand, was less conclusive. The results of the breakthrough Pb collection showed that the chamber cleaning technique was inefficient and positive cleaning controls have invalidated the positive results of the breakthrough Pb collection with the wipes. The positive results of qualitative Pb tests with LeadCheck<sup>®</sup> swabs cannot be viewed as valid as well due to

absence of cleaning controls during the pilot. Only two samples of the breakthrough collection with 37mm cassettes were above the LOQ and were supported with negative controls. Collecting the breakthrough from multiple swatches (5 swatches in the pilot and 10 swatches in the main study) exposed to point air velocity of 6,900 fpm at 90-degree angle of impact in both the pilot and main study resulted in Pb breakthrough amounts of 0.74 and 0.79  $\mu$ g respectfully. Although the Pb breakthrough results do not allow a meaningful quantitative and statistical description of the Pb breakthrough, the investigators concluded that the Pb breakthrough did take place in some extent during the study.

This was the first study to use a Pb-containing GSR as a reference material and in amounts believed to be relevant to levels of clothing contamination taking place at IFRs. The goal of this research was to evaluate the efficiency of AS in removing Pb contamination from ACU, however, a caution should be exercised when using the results of this study in drawing conclusions about AS efficiency or effectiveness due to many study limitations.

Foremost, the study used a reductionistic approach by examining individual, separate swatches of ACU fabric and not an entire piece of a uniform. Such approach did not account for the effects of fabric flapping on Pb reduction during the cleaning process. Also unaccounted were the effects of fabric folds, creases, seams, and parts of a uniform with double layers of fabric, which have been shown to affect a contaminant retention in prior studies (40).

Second, the study used a particular model of AS. Although the point air velocities and angles of impact examined in this study could be present in other AS models, a

different number and location of nozzles as well as different cleaning cycle duration in other models may affect the resulting Pb reduction. The study also did not account for the AS purging function usually expressed in total airflow through the booth, which has been shown to affect a contaminant reduction in prior studies (37).

Third, the results of this study are limited to the amount and unique characteristics of Pb load on swatches as was evident from different Pb reduction results of the pilot and main study. The size distribution and morphology of Pb particles in loads used in this study were not examined, but forensic studies have shown that various types and brands of ammunition often produce unique GSR characteristics (9). The method of gravitational settling from air used to load ACU swatches with Pb-containing GSR particles is also a limiting factor in this study. Particles' deposition to clothing due to other mechanisms such as direct contact or rubbing against a contaminated surface during kneeling and prone firing on IFRs could result in a different strength of particles' adhesion affecting the Pb retention by fabric and therefore Pb reduction during AS application. Thus, generalization of results of this study to other types of ammunition or mechanisms of clothing contamination should be avoided. Furthermore, since the estimation of the extent of Pb clothing contamination on IFRs used in the selection of target Pb loads was based on indirect and qualitative data, its relevance is also arguable.

Fourth, the study examined only one type of garment used by service members on IFRs. Besides the ACU made of 50/50 cotton/polyester fabric, other garments used on IFR consist of fire-retardant ACU (FRACU), GORETEX<sup>®</sup>, and other materials used in various body armor outfits. The roughness and air permeability of these materials could be different from the ACU potentially affecting the Pb retention, reduction, and

breakthrough. Thus, the findings of this study should not be generalized to other materials.

Lastly, the methods and materials developed and used in this study are not standard techniques and have not been fully validated. The investigators took every effort within the time and funding limitations of this study to ensure the integrity of used methods either experimentally or with appropriate controls. Nevertheless, the efficiency and appropriateness of some techniques remained questionable, which became evident during the examination of Pb breakthrough.

#### CONCLUSION

This is the first study to examine the efficiency of ASs in removing a GSRspecific Pb from ACU swatches loaded with amounts of Pb in the order of micrograms per square centimeter. The study found a positive and significant correlation between selected point air velocities and observed Pb reduction. The angles at which air velocities fell on ACU swatches did not have a significant effect on Pb reduction. The observed Pb reduction ranged from 8.2% to 56.1% and was dependent on the amount and characteristics of Pb load. The study confirmed the presence of Pb breakthrough during AS application but was unable to describe it quantitatively and statistically. The study results are limited to the AS model, ACU material, characteristics of Pb load, study approach, and methods.

#### RECOMMENDATIONS

Occupational hygiene practitioners, researches, and AS manufacturers could use the methods and results of this study to design future studies of ASs efficiency in a particular application. Prior to conducting further research, the authors recommend

quantitative examination of clothing contamination with Pb taking place on IFRs and proper validation of methods described in this study. The authors recommend further examination of AS efficiency using a wholistic approach with various types of garments. Further research is also needed to describe quantitatively the Pb breakthrough and other potential side effects of AS application such as noise exposure and inhalation of resuspended Pb particles.

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## **CHAPTER 5: Conclusion**

The goal of this research was to evaluate the efficiency of AS in removing Pb contamination from ACU. The study has succeeded by meeting its objectives and obtaining data allowing decision-making on study hypotheses. Based on the results and limitations of this study, the researches recommend several topics for future research.

#### **STUDY OBJECTIVES**

The objectives of this research were:

4) Develop a method of loading uniform samples with Pb-containing GSR.

This study was the first to load fabric swatches with a desired amount of a GSRspecific Pb in the order of micrograms per square centimeter. The developed loading procedure was accurate and reproducible but limited to the type of weapon, ammunition, and swatch material used in this study. The loading procedure developed in this study could be used to produce GSR-loaded fabric samples to examine the efficiency of clothing cleaning techniques such as air showering, vacuuming, and laundering.

5) Assess the effects of various air velocities and angles of impact on Pb removal from ACU.

This was the first study to examine the efficiency of ASs in removing a GSRspecific Pb from ACU swatches loaded with amounts of Pb in the order of micrograms per square centimeter. The study found a positive and significant correlation between selected point air velocities and observed Pb reduction. The angles at which air velocities fell on ACU swatches did not have a significant effect on Pb reduction. The observed Pb reduction ranged from 8.2% to 56.1% and was dependent on the amount and

characteristics of Pb load. The study results are limited to the AS model, ACU material, characteristics of Pb load, study approach, and methods. Occupational hygiene practitioners, researches, and AS manufacturers could use the methods and results of this study to design future studies of ASs efficiency in a particular application.

6) Examine the effects of various air velocities on the breakthrough of Pb through ACU during AS application.

The study confirmed the presence of Pb breakthrough during AS application but was unable to describe it quantitatively and statistically.

#### **Hypotheses Decisions**

When making decisions on study hypotheses concerning the Pb reduction, the author used the data exclusively from the main study. The examination of Pb reduction in the pilot was limited only to sub-samples and did not allow a full quantitative and statistical assessment of effects produced by each point air velocity at all three angles of impact as it was outlined in corresponding specific aims. Since the results of the Pb breakthrough were quantitatively and statistically inconclusive, the author used a qualitative analysis when deciding on Pb breakthrough hypothesis and specific aims using the data from both the pilot and the main study.

<u>**Hypothesis #1**</u>. Exposure of ACU swatches loaded with Pb to high air velocities will result in  $\geq$  50% reduction in Pb.

**Specific Aim #1**. Determine the percent reduction by comparing Pb mass on ACU swatches before and after application of point air velocities of 4,100 and 6,900 fpm at 0, 45, and 90-degree angles of impact.

**Results**: - mean Pb reduction at 4,100 fpm was 12.3% with 95% CI of [9.6; 15.0] - mean Pb reduction at 6,900 fpm was 18.9% with 95% CI of [16.2;

21.6]

**Decision**: do not accept the hypothesis

**Hypothesis #2**. Exposure of ACU swatches loaded with Pb from GSR to high air velocities will result in the breakthrough of Pb through the fabric

**Specific Aim #2**. Measure Pb mass pushed through ACU swatches by point air velocities of 4,100 and 6,900 fpm at 0, 45, and 90-degree angles of impact.

**Results**: Pb breakthrough was registered at 6,900 fpm. **Decision**: accept the hypothesis.

<u>**Hypothesis #3**</u>. Exposure of ACU swatches loaded with Pb to low air velocity will result in  $\geq$  50% reduction in Pb without causing the breakthrough of Pb through the fabric.

**Specific Aim #3**. Determine the percent reduction by comparing Pb mass on ACU swatches before and after application of point air velocity of 1,800 fpm at 0, 45, and 90-degree angles of impact. Specific Aim #4. Measure Pb mass pushed through ACU swatches by
point air velocity of 1,800 fpm at 0, 45, and 90-degree angles of impact.
Results: - mean Pb reduction at 1,800 fpm was 10.6% with 95% CI of [7.9; 13.3]
- Pb breakthrough at 1,800 fpm was not registered

**Decision**: do not accept the hypothesis

#### FUTURE RESEARCH

Although many studies have examined Pb exposure at firing ranges, it appears that only a few had conducted an analysis of particle size-specific Pb mass distribution. Besides the study by Dams et al., the literature review for this study revealed one additional published study and one unpublished work on this subject. Further research of Pb mass distribution produced by various types of ammunition would enhance the accuracy of theoretical estimates for loading techniques such as reported in this study as well as for assessments of Pb exposures taking place on firing ranges.

The difference of Pb loads between some loading events was relatively (up to 39%) and statistically significant despite using the ammunition of the same lot and following the same loading procedure protocol. The only difference between these two and other events that the investigators had noticed was environmental conditions (air temperature and relative humidity) inside the chamber during the loading. The effects of air temperature and relative humidity on GSR particles' generation and behavior in the air following a firearm's discharge have not been found in the literature reviewed for this study and could be a subject of a future research.

A quantitative description of Pb contamination on clothing on IFRs and a role of Pb-contaminated clothing in "take-home" Pb exposure have not been found in the literature reviewed for the study. A number of studies measured Pb on various surfaces inside and outside IFRs and qualitatively examined Pb contamination of shooters' and range employees' clothing to assess the adequacy of hygiene and housekeeping practices. These results were used to estimate the degree of clothing contamination at IFRs when selecting a target for Pb loads for this study. The author recommends characterizing clothing Pb contamination at IFRs, which could be used in future studies of AS efficiency and effectiveness relevant to this application.

This study intended to be the first in a series of studies aimed at evaluating the overall effectiveness of ASs as a Pb exposure reduction measure on IFRs. The results of this study provide some basic and very limited information about the efficiency of ASs in this application and should not lead to any firm conclusions about the overall effectiveness of ASs. The effectiveness of ASs should be assessed by examining its ability to achieve the goal for which it is employed i.e. to reduce Pb exposure by minimizing Pb spread outside the range on contaminated clothing. To further assess the effectiveness, future studies could examine ASs' efficiency with a wholistic approach using full sets of garment and evaluate potential side effects of AS application such as noise exposure, inhalation of re-suspended Pb particles, and spread of Pb to otherwise clean parts of a body due to Pb breakthrough.

### **APPENDIX A: Analytical Methods**

### **ACU SWATCHES**

All fabric swatches were digested with a "MARS 6"<sup>™</sup> laboratory microwave oven (CEM Corp, Matthews, NC, USA). Digestion vessels were cleaned using a commercial, laboratory dishwasher, after cleaning in the dishwasher, each digestion vessel was charged with approximately 20 mL of 1:1 V/V (concentrated HNO<sub>3</sub>:H<sub>2</sub>O) acid solution and digested in the microwave using the same microwave sequence used for sample digestion.

Samples were removed from shipping containers using acid cleaned, plastic tweezers and placed in 75 mL "EASYPrep" <sup>TM</sup>(CEM Corp) microwave digestion vessels. Shipping containers were then rinsed 3 times with a total of 5 mL of concentrated HNO<sub>3</sub>. Microwave vessels were then sealed and placed in a digestion carousel. Samples were digested using the following sequence: twenty (20) minute ramp at 1200 Watts (W) to 165°C; ten (10) minute hold time at 165°C; fifteen (15) minute ramp at 1400 Watts (W) to 210°C; ten (10) minute hold at 210°C, vessels were then allowed to cool to ambient temperature. Vessels were opened and vented in a hood. Each vessel's solution was transferred to an acid rinsed, 100 mL, class A, glass volumetric flask. Each digestion vessel was then rinsed 3x with approximately 5 mL of deionized (DI) water, each rinse transferred to the volumetric flask. The flask was then diluted to mark using DI water and transferred to a 100 mL, commercially pre-cleaned HDPE vessel for analysis.

The swatch digestates were then analyzed by EPA 200.8 (SOP DLS 308). This procedure was used for the determination of lead in swatches by Inductively Coupled

Plasma – Mass Spectrometry (ICP-MS) using a Perkin Elmer ELAN 9000 ICP-MS instrument. No modifications to the standard EPA 200.8 method were used.

#### WIPES

Wipe samples were prepared using ASTM 1644 modified (SOP DLS 326). This method was used to digest the wipe samples by heating to  $85^{\circ}$ C -100°C with nitric acid and hydrogen peroxide. The standard ASTM 1644 method was modified by using a Hot Block rather than a Hot Plate for sample digestion, as described below.

Add 12.5 mL of 1:1 HNO3 to the wipe sample in the digestion vessel. Swirl, cover with a watch glass and heat on Hot Block at  $85^{\circ}$ C -100°C for 45 minutes. Remove from the Hot Block and cool to room temperature. Add 2.5 mL of DI H<sub>2</sub>O and 2.5 mL of 30% H<sub>2</sub>O<sub>2</sub> to the vessels. The digestates were then diluted to a final volume of 100mL and analyzed by EPA 200.8 (SOP DLS 308) using a Perkin Elmer ELAN 9000 ICP-MS instrument. Swirl, cover with a watch glass and heat on Hot Block at  $85^{\circ}$ C -100°C for 45 minutes. Remove from the Hot Block, cool to room temperature, and filter.

#### **37MM CASSETTES AND SEPARATE MCE FILTERS**

Air filter samples were prepared using NIOSH 7300 modified (SOP DLS 316). The standard NIOSH 7300 method was modified by digesting the samples with nitric acid only, rather than using both nitric and perchloric acids for digestion. Also, a Hot Block procedure was used for sample digestion, rather than Hot Plate, as described below.

Add 6.0 mL of 1:1 HNO<sub>3</sub> to sample filter in digestion vessel. Swirl, cover with a watch glass and heat on Hot Block at  $100^{\circ}C \pm 5^{\circ}C$  for 30 minutes. Remove from the Hot Block and cool to room temperature.

The digestates were then diluted to a final volume of 50mL and analyzed by NIOSH 7300 modified (SOP DLS 324) using a Perkin Elmer Nexion 350D ICP-MS instrument. The standard NIOSH 7300 method was modified by analyzing the samples with ICP-MS rather than ICP Atomic Emission Spectrometry (ICP-AES) in order to achieve lower detection limits by following EPA 200.8 methodology.

### **RESULTS REPORTING**

For swatches, wipes, and air filter samples, results from the instrument were reported in  $\mu$ g/L and converted to  $\mu$ g/sample as follows:

 $\mu$ g/sample =  $\mu$ g/L (sample) x final vol (L) x dilution factor

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