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Release Mitigation Spray Safety Systems for Chemical Demilitarization Applications

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Abstract

Sandia National Laboratories has conducted proof-of-concept experiments demonstrating effective knockdown and neutralization of aerosolized CBW simulants using charged DF-200 decontaminant sprays. DF-200 is an aqueous decontaminant, developed by Sandia National Laboratories, and procured and fielded by the US Military. Of significance is the potential application of this fundamental technology to numerous applications including mitigation and neutralization of releases arising during chemical demilitarization operations. A release mitigation spray safety system will remove airborne contaminants from an accidental release during operations, to protect personnel and limit contamination.

Sandia National Laboratories recently (November, 2008) secured funding from the US Army's Program Manager for Non-Stockpile Chemical Materials Agency (PMNSCMA) to investigate use of mitigation spray systems for chemical demilitarization applications. For non-stockpile processes, mitigation spray systems co-located with the current Explosive Destruction System (EDS) will provide security both as an operational protective measure and in the event of an accidental release. Additionally, "tented" mitigation spray systems for native or foreign remediation and recovery operations will contain accidental releases arising from removal of underground, unstable CBW munitions. A mitigation spray system for highly controlled stockpile operations will provide defense from accidental spills or leaks during routine procedures.

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NOMENCLATURE

DOE	Department of Energy
ECBC	Edgewood Chemical and Biological Command
EDS	Explosive Destruction System
GD	Soman, a nerve agent
GPOS	General Purpose Operations Shelter
GTR	German Tracktor Rocket
HD	Mustard Agent
PBA	Pine Bluff Arsenal
PBEDS	Pine Bluff Explosive Destruction System
PMNSCMA	Program Manager, Non-Stockpile Chemical Materials Agency
SNL	Sandia National Laboratories
VX	A nerve agent; IUPAC name *O*-ethyl *S*-[2-(diisopropylamino)ethyl] ethylphosphonothioate

1. INTRODUCTION

The Release Mitigation Spray Safety System for Chemical Demilitarization Applications project was initiated in 2008 with funding provided from the Program Management for Non-Stockpile, Chemical Materials Agency (PMNSCMA). The objective of the project was to design a prototype mitigation spray safety system that ultimately would be installed and tested or demonstrated in a selected NSCMA operational environment. In the non-stockpile processes, mitigation spray systems co-located with the current Explosive Destruction System (EDS) will provide security both as an operational protective measure and in the event of an accidental release.

There were four main tasks and deliverables associated with the original funding approved and received to perform this project:

- Task 1 – Test decon candidate chemistries for compatibility with current chemical detection systems.
- Task 2 – Modeling, analysis and testing to design a spray safety system to mitigate a release in an EDS operational geometry and environment.
- Task 3 – Build and install a prototype spray mitigation safety system at a selected operational test location.
- Task 4 – Test and characterize performance of installed prototype system.

The project was initially proposed at a funding rate of $500K per year, for each of two years, FY09 and FY10. The prototype design was to be specified based on customer specified site requirements and results of experimental aerosol chamber tests conducted at Sandia National Laboratories.

The NSCMA site initially chosen for design and installation of a release mitigation spray safety system was at the Pine Bluff Arsenal (PBA) facility, located at Pine Bluff, Arkansas. The PBA application would provide protection and limit contamination in the event of a leak during the demilitarization processing of German Tracktor Rockets (GTRs). PMNSCMA agreed that a deployed release mitigation safety spray system co-located with EDS would provide an additional safety measure that may be required to obtain complete site licensure and permitting. Therefore, in November, 2008, the project was accelerated to deliver an appropriate design, test demonstration and installation of a release mitigation spray safety system at PBA to meet the anticipated start of the GTR campaign in the fall, 2009.

However, because PBA was able to obtain the required licensure in a very timely manner and without the benefit of a deployed and functional release mitigation spray safety system, work on the project was suspended in the fall of 2009. Sandia technical staff continues to seek alternative funding sources within CMA, as well as other potential customers for the spray knockdown and mitigation technology.

This SAND report is a summary of activities and experimental test results that occurred from November 2008 through September, 2009. The report provides documentation for Tasks 1-3, outlined above; Task 4 has not yet materialized as explained previously. Monthly progress reports outlining the experimental status and results, schedule, financial and any issues were provided to PMNSCMA throughout the funding period.

2. IDENTIFYING AND DEFINING THE CHEM DEMIL-SPECIFIC APPLICATION

2.1. Pine Bluff Arsenal

As outlined in the Introduction, the Pine Bluff Arsenal (PBA) was chosen by PMNSCMA for design and installation of a release mitigation spray safety system to provide protection and limit contamination in the event of a leak during the demilitarization processing of German Tracktor Rockets (GTRs). PMNSCMA agreed that a deployed release mitigation safety spray system co-located with the Explosive Destruction System (EDS) would provide an additional safety measure that may be required to obtain complete site licensure and permitting. Therefore, in November, 2008, the project was accelerated to deliver an appropriate design, test demonstration and installation of a release mitigation spray safety system at PBA to meet the anticipated start of the GTF campaign in the fall, 2009. Release mitigation spray team members toured the PBEDS facility on December 9, 2008. The trip was productive in acquiring an understanding of the customer's facility and provided initial understanding into real-time customer operational needs for an applied prototype system.

Frequent communications with the NSCMA customer were highly useful in defining requirements for monitoring, demonstration, and the release mitigation spray system. In addition to practical requirements for a deployed system, discussion of potential for live-agent testing through the customer's existing contract with the Edgewood Chemical Biological Center (ECBC) commenced. Refer to Section 2.2.

However, because PBA was able to obtain required permits to allow continued operations without implementation of additional safety features, the Pine Bluff EDS was no longer an application for the prototypic system. The team devised a project plan for the remainder of fiscal year funding. This plan would provide an operational demonstration of the spray mitigation system in the full scale geometry of interest, but at a facility located at Sandia. The system could then be deployed to a site selected by the CMA at a later date. The plan was presented to, and accepted by our NSCMA customers in early March, 2009. The details of the revised test plan are described in Section 2.2.

2.2. Revised Test Plan

The revised plan would provide an operational demonstration of the spray mitigation system in the full scale geometry of interest, at a new test facility located at Sandia. A prototype spray system could then be deployed to a site selected by the CMA at a later date.

- Two threat scenarios for the Spray Knockdown System (see Sections 3.1 and 3.2) were produced based on a theoretical accident at a typical EDS deployment and operation site such as Spring Valley, MD. In the proposed scenarios, mustard (HD) or phosgene were the agent threats. The agent mass, explosive mass, and other airborne source considerations are identified within the threat scenarios described in Section 3. Based on this example, Sandia committed to design a prototype spray system to mitigate the airborne source threat outlined in the identified scenarios.
- Sandia committed to complete a series of tests in Sandia's aerosol chamber facility, using the parameters referred to in the above scenarios as the airborne source threat definition. The spray system in the aerosol chamber was first characterized, then the neutralization efficacy of

DF200 sprays for neutralization of G-agent simulant were evaluated, adjusting the spray system operational parameters in an effort to improve the spray system efficacy.

- Sandia committed to procure and install a 30'x60' structure to support development and testing of the system. Both a General Purpose Operations Shelter (GPOS) and sprung steel structure were initially examined as options. Based on Sandia's initial assessment, a plan was implemented to acquire and install a sprung steel structure to support development and testing and to conduct an operational demonstration of an optimized prototypic spray knockdown system before the end of the fiscal year. The sprung steel structure would provide a permanent, durable facility that would increase Sandia's long-term testing capabilities and better enable Sandia to develop a flexible system to meet a broader range of applications. The loaded cost to provide this prototypic spray knockdown system in the sprung steel structure was ~$135K, while the cost of the tented GPOS structure was $117K. The Sandia team reasoned that the small cost difference between the tented GPOS structure and the proposed sprung steel structure was justified to alleviate potential ES&H concerns and to provide greater flexibility in developing a spray knockdown system capability that could also be used to meet other operational needs. Key points provided to NSCMA for this justification included:

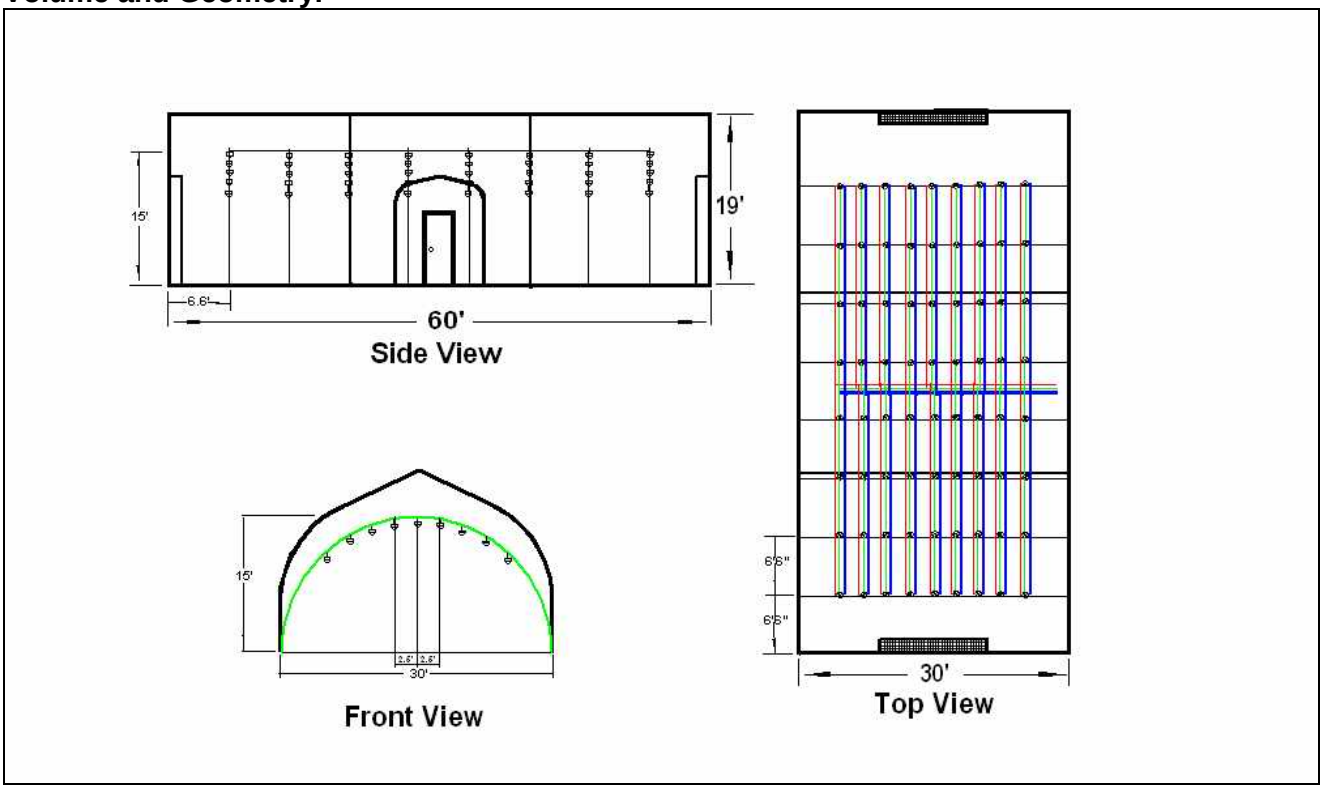
    - The temporary exterior material and flooring of the tented GPOS did not meet Sandia's ES&H requirements for the types of testing planned, as environmental releases could occur during spray system optimization and testing.
    - The GPOS geometry was duplicated within the sprung steel structure to recreate the exact geometry of interest for the current application, while providing room for diagnostic and control systems to be installed within the confinement as required for safe testing. The spray system manifold was designed to be modular for easy installation, and to the same geometry of the tented GPOS structure, then installed within the sprung steel structure. An outline view of the GPOS geometry within the sprung steel structure is represented by the green line on the Front View of Figure 1. The spray system manifold was fitted with a tarp cover to provide a barrier matching the GPOS tent geometry. Therefore, Sandia would be able to provide an operational demonstration of the release mitigation spray system within the customer's current operational geometry, while having the flexibility to explore other geometries and applications in the future.
    - The permanency and ES&H qualities of the sprung steel structure will pay off in the ability to support demonstration of, and/or design for various CMA operational scenarios in years to come. As a result, Sandia will be better able to continue developing useful protective systems for many applications within the CMA program.

- Following completion of efficacy testing in Sandia's aerosol chamber facility, we continued to optimize the current DF-200 decontaminant formulation for use in neutralization of mustard agent. While this was successfully performed through a small series of bench-scale solution efficacy tests, schedule and resource constraints precluded evaluation of the modified formulations in the aerosol test chamber under operational spray system conditions.
- In December, 2009 Sandia submitted two modified DF-200 formulations optimized for mustard efficacy, to use for efficacy testing with live mustard agent. The formulations were submitted for testing using a modified live agent test protocol developed for used in an EDS environment rather than the historical stirred reactor protocol as performed previously[1].These

[1] ECBC TR-474, "Stirred Reactor Decontamination Studies of DF-200 Formulations with VX, HD, and GD", performed from 2002-2004

modified formulations are scheduled to be evaluated for efficacy in decontamination of a series of 17 different agents (nerve, vesicants, arsenicals, industrials, irritants and smokes), using a modified micro-scale, stirred reactor procedure. The modified stirred reactor procedure carries out reactions with and without the addition of granular iron and copper, to mimic the atmosphere within an EDS. Final results of these modified stirred reactor tests for five different live agents are shown in Appendix E.

Figure 1 – Front, Side and Top Views of a Spray Knockdown System Within an Operational Volume and Geometry.



3. MODELING THE THREAT

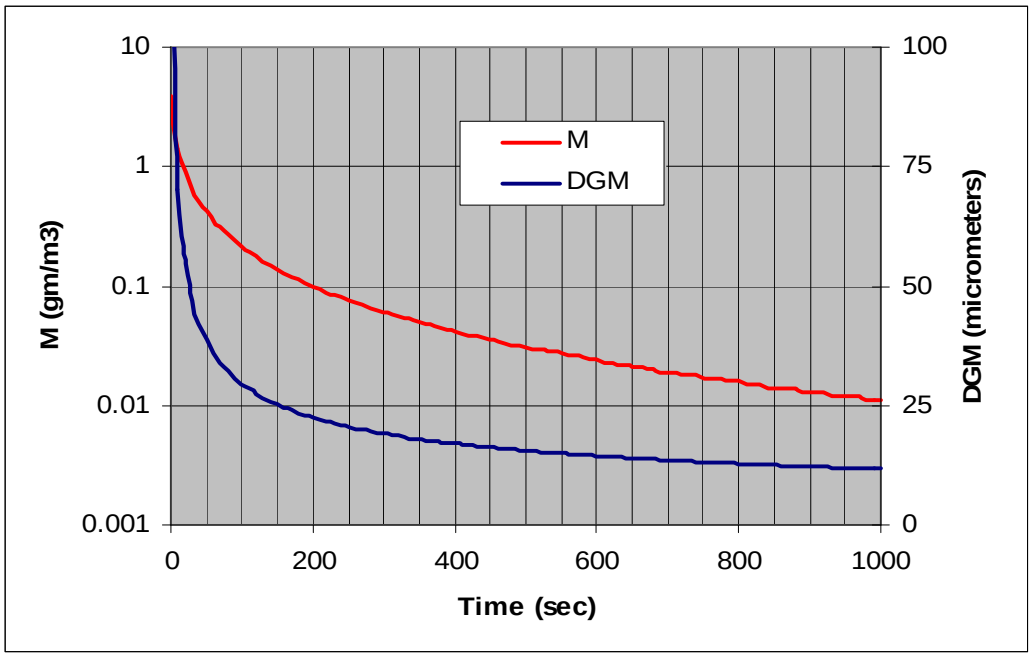
In the absence of a customer assigned, site-specific application, a set of threat scenarios for the Spray Knockdown System were produced, based on a theoretical accident at a typical EDS deployment and operation site such as Spring Valley, MD. In the proposed scenarios, HD or phosgene were the agent threats. The agent mass, explosive mass, and other airborne source considerations were identified as described on the following pages. Based on these considerations, the team proceeded with an experimental design to mitigate the airborne source threat outlined in the alternative scenarios.

As presented in Sections 3.1 and 3.2, modeling of threat agent release conditions produced a comprehensive understanding of airborne threat vapor/particle distribution and concentrations, and particle fall-out over time. Potential airborne exposure levels could be compared to target safe exposure levels to determine orders of magnitude reduction in initial threat agent exposure level required to attain safe exposure levels, i.e., required neutralization efficacy. As shown in Section 7 (Experimental Aerosol Test Chamber Results) and Appendix A (Aerosol Chamber Test Matrix and Results Summary), experimental testing demonstrated rapid, effective knockdown and neutralization of aerosolized CW agent simulants from initial high-threat levels to >5 orders of magnitude reduction, corresponding to inhalation exposure levels below the median lethal dose (LD_{50}).

3.1. Alternative Scenario # 1, HD Threat Definition

We assumed a worst-case scenario, that a 4.2 inch mortar shell containing 6.5 pounds of mustard (HD) exploded and released mustard to the interior of a 30' by 60' by 12' to 19' high structure. The source term for a center burster configuration with 0.73 pounds of explosive is liquid drop dispersion with an estimated geometric mass mean droplet diameter of 150 micrometers and a geometric standard deviation of about 2.5. This gives an initial airborne dispersion mixed throughout the building volume of about 4.8 gm/m^3. The larger droplets settle out most quickly.

Figure 2 – A Modeled HD Threat Particle Distribution and Fall Out over Time



As seen from Figure 2, the airborne geometric mass mean drop size and mass concentration decrease from 150 to 30 micrometers and 3.9 to 0.22 gm/m^3 in 100 seconds. This predicts that there would be about 0.9 gm/m^3 additional vapor for a total airborne concentration of 4.8 to 1.12 gm/m^3, to establish the design to mitigate for this airborne source. As verified by experimental results, the mitigation release safety spray system effectively reduced these initial airborne simulant concentrations.

3.2. Alternative Scenario # 2, Phosgene Threat Definition

Another agent material used in the 4.2 inch mortar shell is 6.25 pounds of phosgene. Explosive dissemination of this amount of material in the 600 m^3 volume of the structure would yield an average concentration of 4.72 gm/m^3. It is reasonable to assume that all of the phosgene would be vaporized as it would be explosively dispersed into drops and any drops impinging on surfaces would vaporize, given that the volume of air has sufficient heat capacity to vaporize all 6.25 pounds of phosgene with less than a degree K drop in temperature.

Another type of munition with a phosgene fill is the Livens projectile that contains 28 pounds of phosgene. The burster in this munition consists of 2.11 oz (60 gm) TNT and would threaten the integrity of the structure. Release of 28 pounds of phosgene in the 600 m^3 volume of the building would produce 21 gm/m^3 average concentration and would be vaporized with about a 4 degree K drop in air temperature.

3.3. Potential for Destruction of Sprung Steel Containment Structure

There is concern that the 0.73 pounds of explosive utilized in the above threat scenarios would destroy the containment building. Consider a 30’ by 60’ by 12’ to 19’ high structure. Using a correlation from Lees in which the mass of explosive (M, in pounds) and the overpressure (P, in psi) is related to the distance (X, in feet) from the explosive.

$$X = M^{1/3} \exp[3.5031 - 0.7241\ln(P) + 0.0398 (\ln(P))^2]$$

where X = Distance in feet to a given overpressure P
M = TNT equivalent mass, lbs
P = overpressure, psi (psi = pounds per square inch)

With the 0.73 pounds of HE in the center of a 30 by 60 by 19 building, there would be 1 psi overpressure at the far wall (30 ft), 2 psi overpressure at the ceiling (19 ft), and 3 psi overpressure at the near wall (15 ft). This would likely rupture a sprung building, as indicated in Table 1, Explosion Overpressure Damage Estimates.

Table 1 – Explosion Overpressure Damage Estimates

Overpressure, psi	Expected Damage
0.04	Very loud noise (143 dB); sonic boom glass failures
0.1	Breakage of small windows under strain
0.15	Typical pressure of glass failure
0.30	10% of windows broken
0.5	Windows shattered, limited minor damage to house structures
0.7	Upper limit for reversible effects on humans
1.0	Partial demolition of houses; corrugated metal panels fail and buckle; skin lacerations from flying glass
2.0	Partial collapse of walls and roofs of houses
2.4	Eardrum rupture of exposed populations
2.5	Threshold for significant human lethality
3.0	Steel frame building distorted and pulled away from foundation
5.0	Wooden utility poles snapped
10	Probable total building collapse. Lungs hemorrhage
20	Total destruction. 99% fatality due to direct blast effects

4. FORMULATION CHEMISTRY OPTIMIZATION AND COMPATIBILITY WITH CHEMICAL DETECTION SYSTEMS

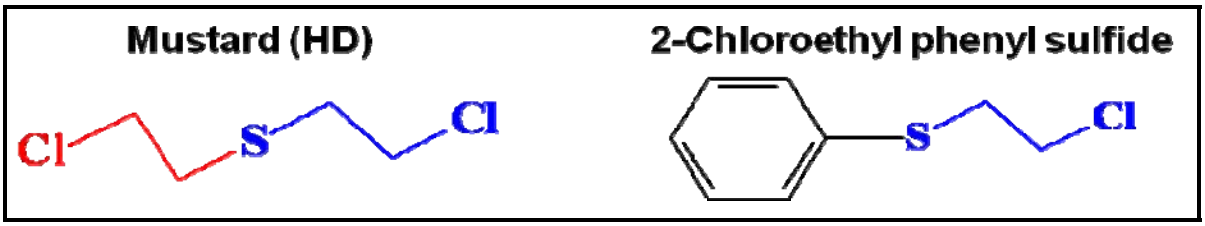
4.1. Optimization of DF-200 chemistry

The standard DF-200 chemistry was optimized to better mitigate for HD releases. Increasing the solubility of agent into the decontamination formulation is key to achieving an optimized neutralization. Two modified formulations were developed by altering the primary surfactant and co-solvents of the standard DF-200 formulation. The chart below shows the efficacy (average of the two modified formulations) test results in neutralization of the HD simulant, 2-chloroethyl phenyl sulfide, illustrated in Figure 3. The efficacy tests were performed in solution at a simulant:decontaminant challenge ratio of approximately 1:125.

Table 2 – Comparison of Neutralization Efficacy of DF-200 and Modified DF-200 Formulations (Average Results)

Decontaminant	HD Simulant (solution tests)	
	5 Min.	60 Min.
DF-200	70.0	99.8
DF-200, modified	99.3	ND

Figure 3 – Physical Structures for HD and a Preferred HD Simulant, 2-Chloroethyl Phenyl Sulfide



4.1. MINICAMS Assessment

To test candidate decontaminant chemistries for compatibility with the current chemical detection system (MINICAMS), an assessment of potential DF200 interference components was planned as Task 1 of the initial project schedule. However, problems in obtaining the required software to operate the MINICAMS caused Sandia to attempt to operate the MINICAMS using a DOS rather than the preferred MINILINK or CHROMLINK software package. The software was not purchased due to cost and PBA was advised by the software manufacturer not to lend Sandia a copy of the current software since the manufacturer does not sell standalone software versions. Unfortunately, Sandia could not successfully obtain data via the DOS-run MINICAMS system, thus this task was not completed.

5. NOZZLE CHARACTERIZATION

5.1. ElectroStatic Spray Nozzle Characterization

Sandia began ElectroStatic Spray (ESS) nozzle characterization the third week of March 2009, with the spray system manifold thoroughly examined and any worn or damaged parts were replaced.

Nozzle characterization testing was conducted in the 8' by 8' by 8' aerosol test chamber. This testing was to assess and understand the average spray particle size and size distribution as a function of nozzle air and liquid flow feed pressures. Fundamental nozzle performance data was then referenced accordingly during the subsequent spray system performance testing and optimization efforts. Refer to Figures 4 and 5 for the ESS nozzle characterization set-up.

A test matrix was performed to determine average particle size and size distribution as a function of air pressure and liquid (deionized water) flow rates. Initial nozzle characterization testing was performed using deionized water. Once nozzle performance was assessed over a wide range of test conditions, additional characterization was performed using DF-200 and the modified DF-200 liquid solutions. As averaged initial data indicated (see Figure 6, lower graph), an encouraging trend was observed in that over the range of liquid flow rates, as the dispersion air reaches close to 100psi the (averaged) droplet diameter becomes less than 30µm.

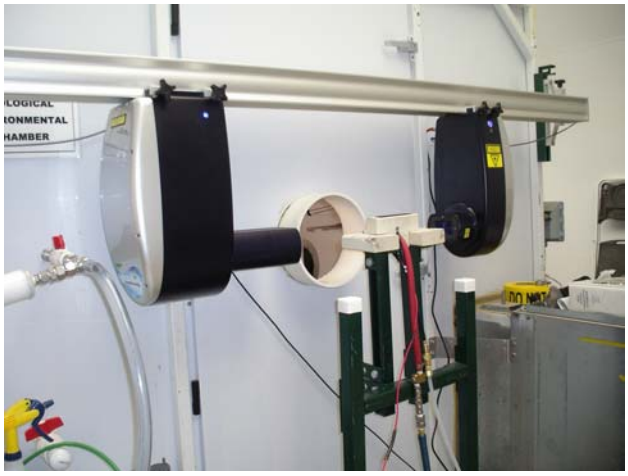


Figure 4 – ESS Nozzle Characterization
An ESS nozzle is mounted on the white block in the center of the photograph, facing into the opening of the chamber door. The red/blue line is for fluid flow and the white/black line is for air flow into the nozzle. The Malvern Spraytec is mounted to the chamber and positioned to allow the laser to pass through the spray nozzle output.

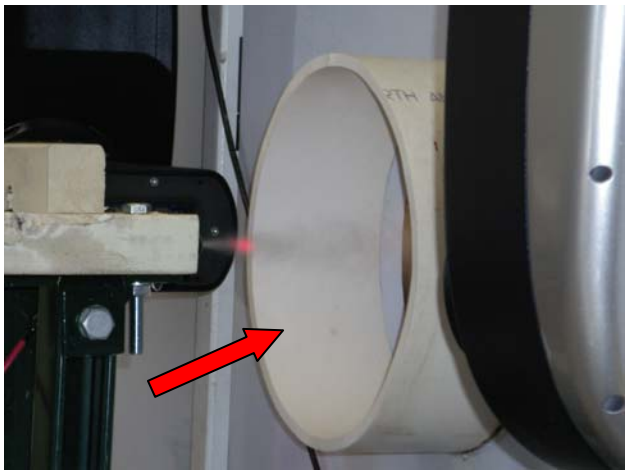
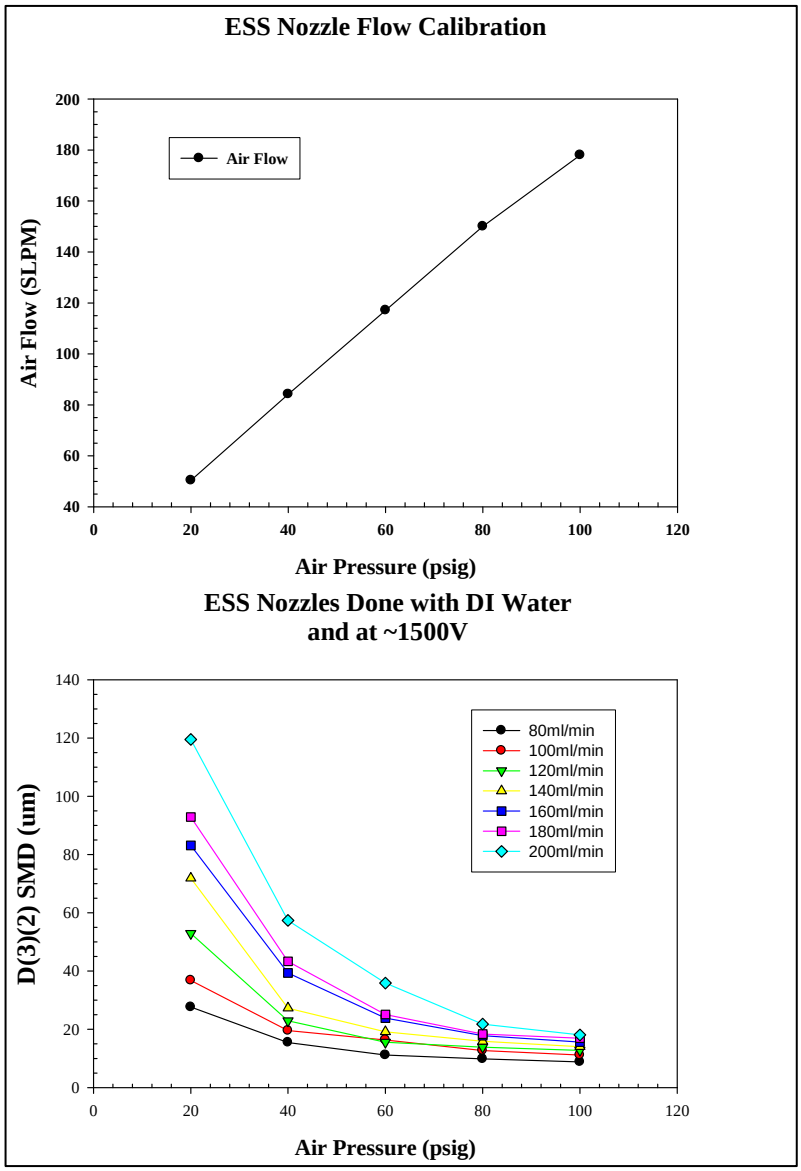


Figure 5 – ESS Nozzle Characterization (side-view)
A close-up photo of the spray emitted from the ESS nozzle tip (red arrow). Note the red glow from the Malvern Spraytec laser as the laser passes through the spray output.

Figure 6 – ESS Nozzle Flow Characterization
The top graph represents a near-linear response with respect to air flow (SLPM) and air pressure (psig). The bottom graph is a display of volume mean particle size (µm, y-axis) as a function of air pressure (20-100 psig) at various liquid flow rates (80-200 ml/minute).



5.1.1. Fine-tuning Droplet Dispersion – Deionized Water

As averaged initial data indicated (Figure 6, lower graph), an encouraging trend was observed, as the dispersion air approached 100psi, the (average) droplet diameter became less than 30µm over the range of liquid flow rates. However, initial data also indicated some droplet dispersion at the larger end of the particle diameter spectrum (approximately >300µm) across all flow rates (see Figures 7 and 8). This observation was confirmed by investigating the spread and magnitude of larger particles at

various locations of the spray output to measure the particle size distribution across both horizontal and vertical displacement of the produced spray. Because larger particles will fall out much quicker than smaller droplets, an investigation of the spray dispersion and fallout patterns along both of these axes would allow a determination of the magnitude of the observed larger particle distribution. This process could potentially rule that the initial observations were an artifact of the spray measurement system. The magnitude of the larger particle size distribution related to the total spray output could then be quantified with greater accuracy.

Figure 7 – Average Particle Size Distribution from ESS Nozzle, Collected Using the Malvern Spraytec (Flows = 140ml/min of Deionized Water and 100 psi of Air)

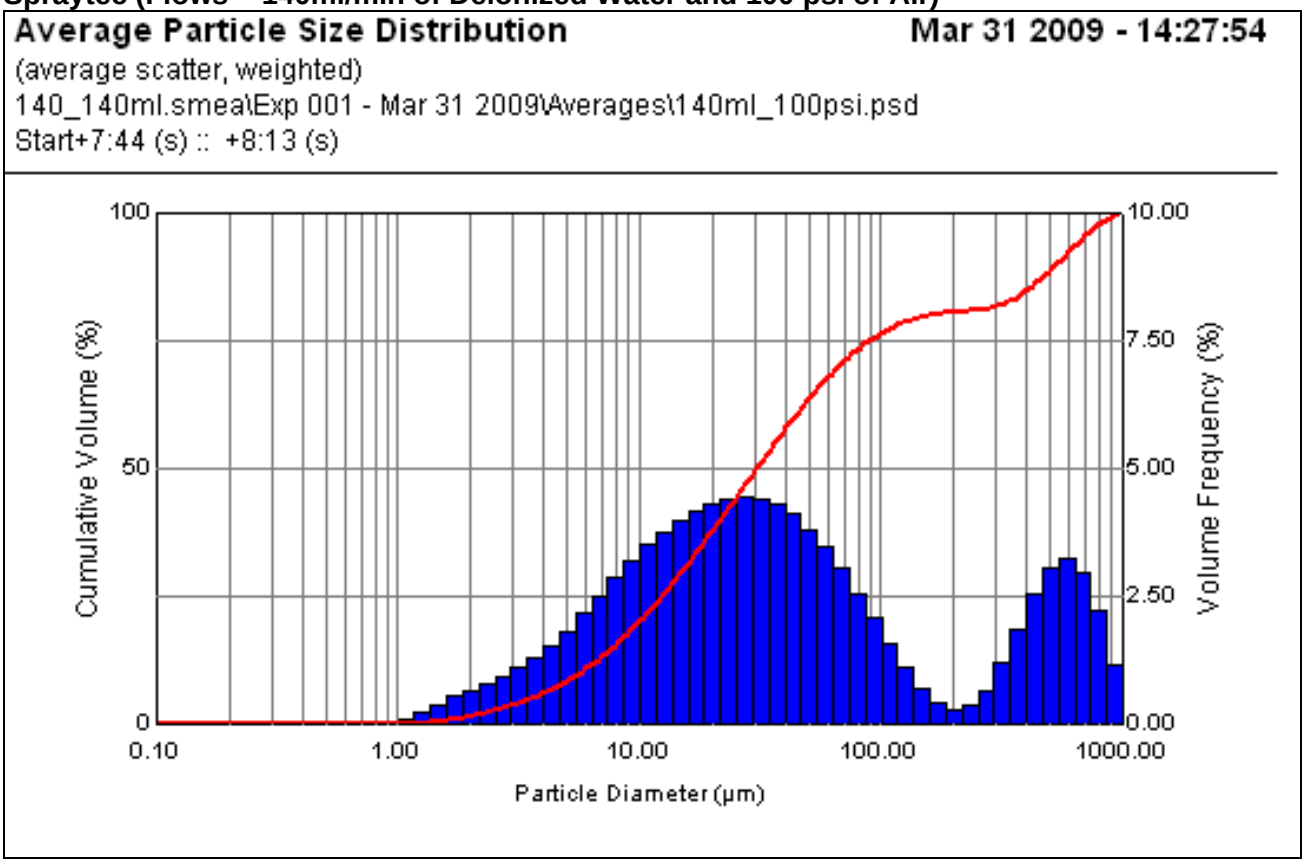
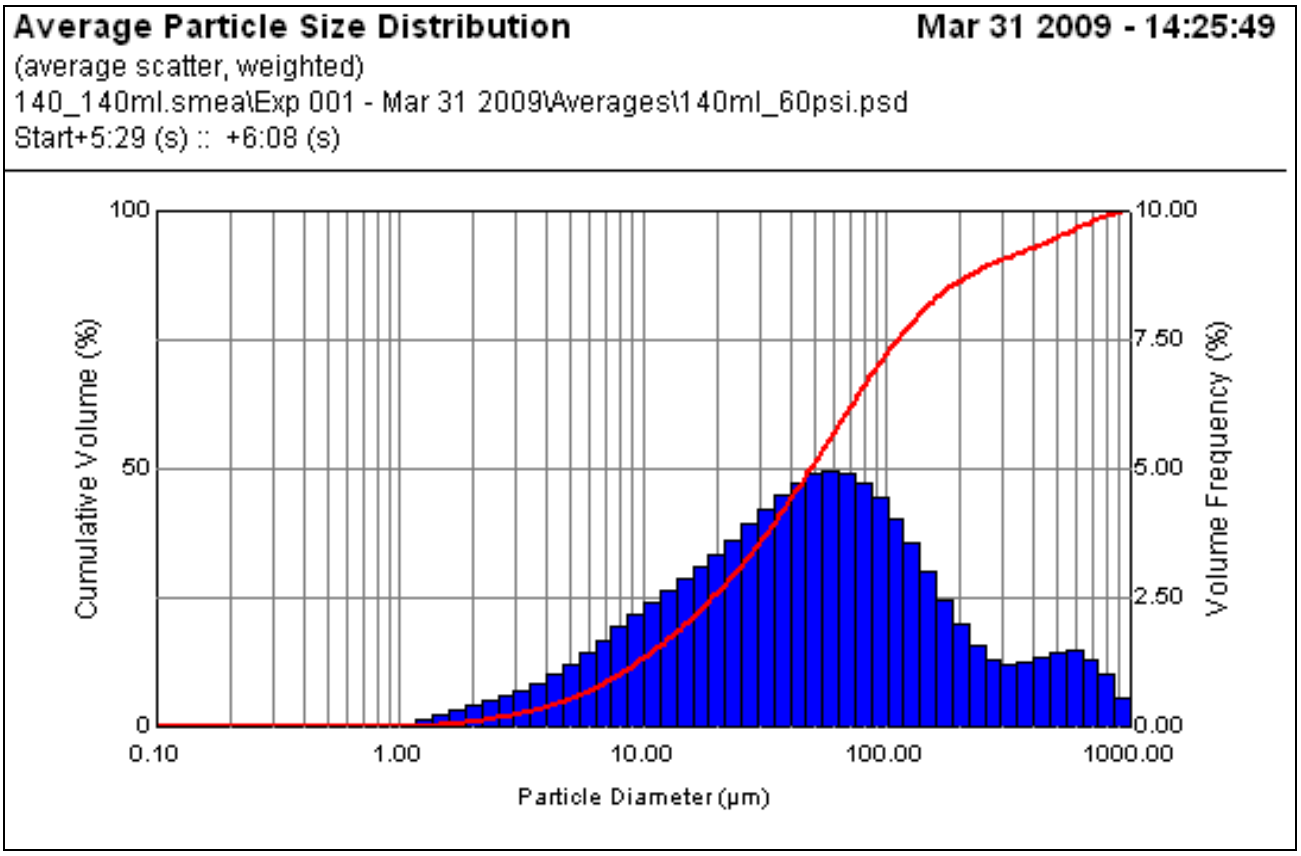


Figure 8 – Average Particle Size Distribution from ESS nozzle, Collected Using the Malvern Spraytec (Flows = 140ml/min of Deionized Water and 60 psi of Air)



The conclusion from characterization of the larger particle distribution was that the initial observation was an artifact of the system, possibly caused by either a discrepant algorithm or light scattering. The larger particle distribution was highly consistent among various test conditions (horizontal and vertical axes spray distributions). All particle size data consistently show nearly the same distribution in the primary and center modes, as well as the larger particle mode.

5.1.2. Fine-tuning Droplet Dispersion – DF-200

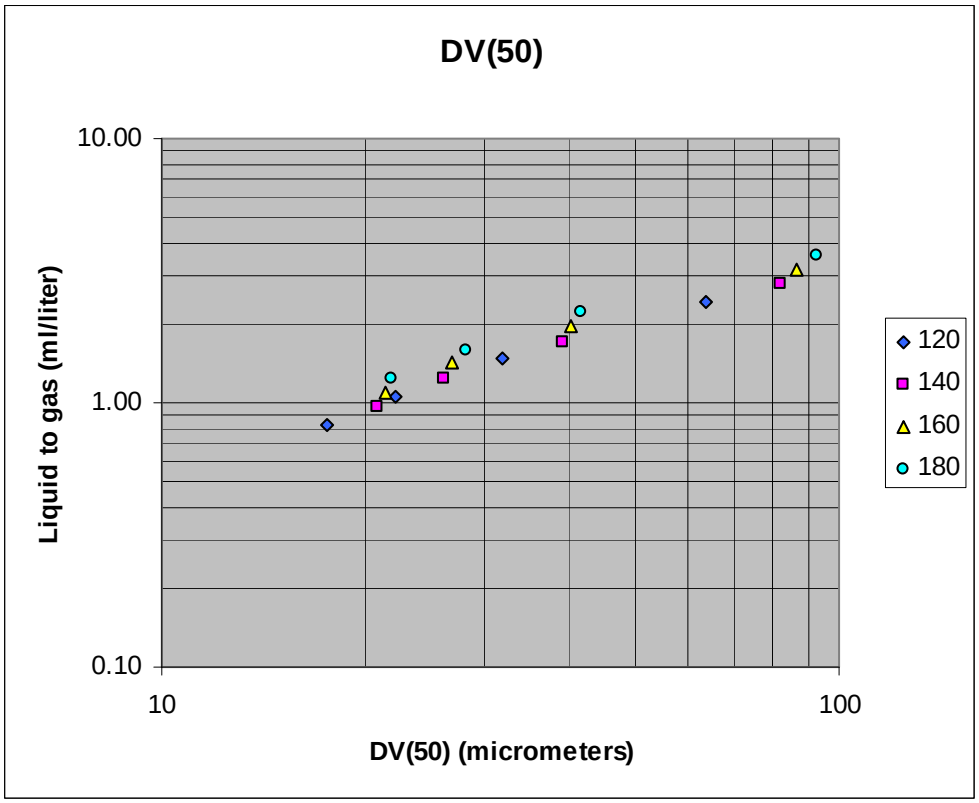
Characterization of the ESS nozzle using DF200 sprays was performed the week of April 27. Review of the data revealed inconsistent trends for DF200 particle size as a function of liquid and air flows, producing doubt regarding the data produced at liquid flows at 160, 180 and 200 ml/min. One potential hypothesis was that gas generation (from active formulation in pressurized decon tank) created sufficient back pressure at these higher liquid flows and that this pressure impeded and/or altered capability to determine and verify accurate liquid flow measurements. Another potential contributing factor was the 7 µm filter that was placed in line for BIT nozzle evaluation (performed during the 2006-2007 timeframe) that remained in the test system. This filter was not installed during the prior Cloud Knockdown LDRD testing performed in 2004-2005. Historical flow measurements collected during the Cloud Knockdown project could be correct, based on current trends. There was

discussion of increasing the filter pore size, as opposed to removing the filter completely, as it is thought that a spray system requires liquid filtering. An assessment of pressure drops across different filter pore sizes should be performed prior to start of future aerosol decontamination test projects.

To answer the question of inconsistent trends for DF200 spray particle size as a function of liquid and air flows, the following tests/evaluations were completed prior to simulant testing in the aerosol chamber.

- Assess pressure buildup in decon storage tank by monitoring pressure increase over time.
- Assess pressure buildup/release as decon formulation is released from tank. This will require measuring pressure upstream of the decon tank.
- Add a pressure transducer immediately or as close to point before nozzle, to assess pressure entering the nozzle.
- Re-measure liquid flows (while monitoring pressures) over liquid flows of 160, 180 and 200 mls/min (decon).
- Repeat evaluation of DF200 spray particles over feed air pressures of 20-100 psig and liquid flow rates of 80, 100, 120, 140, 160, 180 and 200 ml/min.

Figure 9 – Plot of DF200 Spray Particle Size (Mass Mean, micrometers) vs. ESS Nozzle Liquid Flow to Air Flow Ratio. The data series collected from 120 to 180 ml/min liquid flow rates.



5.1.3. In-situ Charge Measurement

Although not accomplished, the test team intended to measure the charge of aerosolized decontaminant spray in-situ from the ESS nozzles, as well as to investigate the decontamination

efficacy across a range of applied voltages. In-situ charge measurement and APS measurements collected of the total spray, top to bottom, taking care to keep both measurements at the same line, would enable a collection of charge to mass ratio data. A Faraday cage device was constructed the week of April 12, ant the electrometer required to measure the in-situ electrical charge failed during the week of April 20. The electrometer was shipped to the manufacturer for repair the same week. Upon return from the manufacturer, in-situ charge measurement was never performed due to personnel and scheduling constraints.

6. EXPERIMENTAL AEROSOL CHAMBER DESCRIPTION, TEST CONDITIONS AND AEROSOL CHAMBER TESTS

The aerosol chamber spray system test parameters were predicated on results of nozzle characterization, expected efficacy performance and the simulant threat concentration.

6.1. Experimental Aerosol Chamber Description

The aerosol test chamber (Figure 10) consisted of **two** compartments and was served by two systems—a spray system and an air purge system. The overall dimensions of the chamber were 8 ft wide by 8 ft high by 16 ft long. The total interior volume was 512 ft^3 ($14.5m^3$). The chamber was constructed of polypropylene sheets, welded at the seams to make the chamber watertight. A center wall divided the chamber into two equal volume compartments. Access doors were located at each end of the chamber with a valved floor drain in each compartment. A spray nozzle manifold (Figure 11) was attached to the ceiling of the west compartment. Four windows constructed from 3/8" clear static free PVC were located on the north and south walls. Glove ports and feed-through boxes were located on the south wall of the compartments. The chamber contained a HEPA filtration (with Carbon filters) exhaust system capable of purging the chamber interior volume in one minute. The two floor drains were plumbed to an electrical sump pump, enabling delivery of chamber drainage to a 1000-gallon wastewater holding tank.

The primary components of the Knockdown Spray System included decontaminant/water purge tanks (2-2 gallon capacities), spray head manifolds, and nozzle heads with seven micro spray nozzles/head. Nine electrostatic nozzle heads (Maxcharge™) were also installed, allowing the formation of optimally sized spray droplets of 30 to 40 microns in diameter, with a required air pressure of 20 to 90 psi. The ESS nozzle air consumption was typically 2.9-10 CFM per nozzle, and the liquid flow rate was 50-200 ml/min for each nozzle.

Figure 10 – An Aerosol Test Chamber, 14.5 m^3 total volume

Figure 11 – The Spray Nozzle Manifold Mounted on the Ceiling of the Aerosol Test Chamber

6.1.1. Aerosol Chamber Primary Instrumentation

- **BioSamplers** aerosol samplers (impingers), SKC Model No. 225-9595, with 20 ml collection vessel capacity. The BioSamplers were operated at approximately 10 liters per minute.
- **Collison Nebulizer,** BGI Incorporated Model No. CN-60, used to aerosolize the chemical simulants. The Collison Nebulizer contains 24 jets and was typically operated at 40psig at an airflow rate of approximately 80 liters per minute. A 6-jet nebulizer operated at 80psig was also available.
- **Aerodynamic Particle Sizer (APS),** TSI Inc., Model 3321 was used to characterize the particle diameter of the **simulants** in the chamber and distinguish between vapor and particulate. The APS typically operates at a total flow rate of 5 liters per minute with particle size range detection between 0.5 to 20.0um aerodynamic sizing.
- **Miran Portable Ambient Air Analyzer,** Thermo Environmental Instruments Model 205A, used to measure air concentrations of the chemical simulants. The Miran operates at approximately 14 liters per minute with storage capacity of up to 10 multigas measurements.
- **Malvern Spraytec Real-time Liquid Droplet Sizing system**, Malvern Inc., Model RS500, used to measure the liquid droplet size distribution from the spray nozzles. The Spraytec uses laser diffraction as the technique for characterizing particle size distribution.
- **Vacuum and Atmosphere Control System,** Vaccuum Atmosphere Company Model HS-PC-1, controls the internal atmosphere of the aerosol test chamber.
- Thermocouples and pressure transducers were placed in the chamber for measurement and monitoring of critical test parameters such as temperature, pressure, humidity, liquid flow rate (nozzle) and air flow rate (nozzle).

A data acquisition system monitored and electronically recorded all electronic output. The knockdown spray system could be operated by either manual or computer control. Computer control enabled the system to be run with a minimum amount of laboratory personnel and generated more precise, consistent and overall higher quality data sets. A data acquisition and control system was constructed using off the shelf components acquired from National Instruments (NI) Inc and an Industrial Type Ruggedized computer. A program was written using Lab View as the control and data acquisition software to control the NI hardware. Below is a description of the hardware used to measure temperature, pressure, liquid and air flow rates.

- Fine Tip TJ Probe Type T Thermocouple Model # TJFT72-T-SS-116G-6-SMPW-M were used to monitor air and liquid solution temperature measurements. Two thickness, 1/16”and 1/8” diameter were used; the 1/16” thermocouples were installed directly into Swagelok fittings for faster response for air and liquid measurements. The larger, 1/8” diameter thermocouples were installed through the wall of the chamber and were more rugged to hold up to the spray solutions dispersed inside of the chamber.
- Several types of pressure transducers were used to measure pressure. : Differential pressure transducers in the range from 0 to 30 inH_2O were used to measure room differential pressure and HVAC duct pressure. Absolute pressure transducers in the range of 0 to 200 psia measured the absolute pressure in the air systems used to supply air to the nozzles. Gage type pressure transducers in the range of 0-150 were used to measure the pressure in the solution tanks, which feed the nozzles.
- The flow rate of liquid solution delivered to the nozzle bank was measured with a low-flow impeller type meter, Omega model number FRP301 with a linear zed analog output to allow for direct connection to the data acquisition system. The flow range for this model was 0 to 18 liters per minute. The accuracy for this unit was 1% of full scale reading and the maximum pressure was 150 psi.
- The air flow delivered to the nozzle bank was measured with two types of mass and volumetric gas flow meters from two different manufactures in order to cover the wide range of air flow conditions. The Omega model FVL-1621A has a flow range of 0-53 CFM (~1500LPM). The second type of flow meter was a CAM5200 which has a range of 2 to 200 CFM (~5717LPM). Both units were ordered with linear zed analog output to allow for direct connection to the data acquisition system.

6.2. Experimental Aerosol Chamber Test Conditions

Aerosol test chamber simulant testing began the first week of June, 2009. The initial tests parameters mimicked those used during the 2004-2005 LDRD cloud knockdown project as a means to baseline the existing spray system and compare it to historical test parameters and data. The G-agent simulant diphenylchlorophosphate (CAS# 2524-64-3) was the initial test simulant, dispersed at levels comparable to agent concentrations according to scenarios developed for the NSCMA program. Following G-agent simulant baseline tests, aerosol test chamber testing was scheduled to resume using 2-chloroethyl phenyl sulfide, the preferred sulfur mustard simulant, as the threat simulant. A total of 12 aerosol chamber tests were completed, with results provided in Appendix A, Aerosol Chamber Test Matrix and Results Summary.

The commercial DF200 formulation was the initial decontaminant spray, and modified DF200 formulations were also scheduled to be evaluated for efficacy. Aerosol test chamber testing continued through May, June and July. These tests were critical to determine spray system neutralization capability of agent simulants and to then optimize the spray system parameters and/or decontaminant chemistry to increase agent simulant neutralization.

Several parameters were considered in the design and execution of aerosol chamber tests. Parameters included choice of simulant, desired simulant concentration (based on modeled threat scenarios), flow to collison required to achieve desired simulant concentration; desired decontaminant spray density (based on liquid decontaminant flow and air flow into nozzle), magnitude of charge applied to nozzles and the aerosol sample collection method and efficiency.

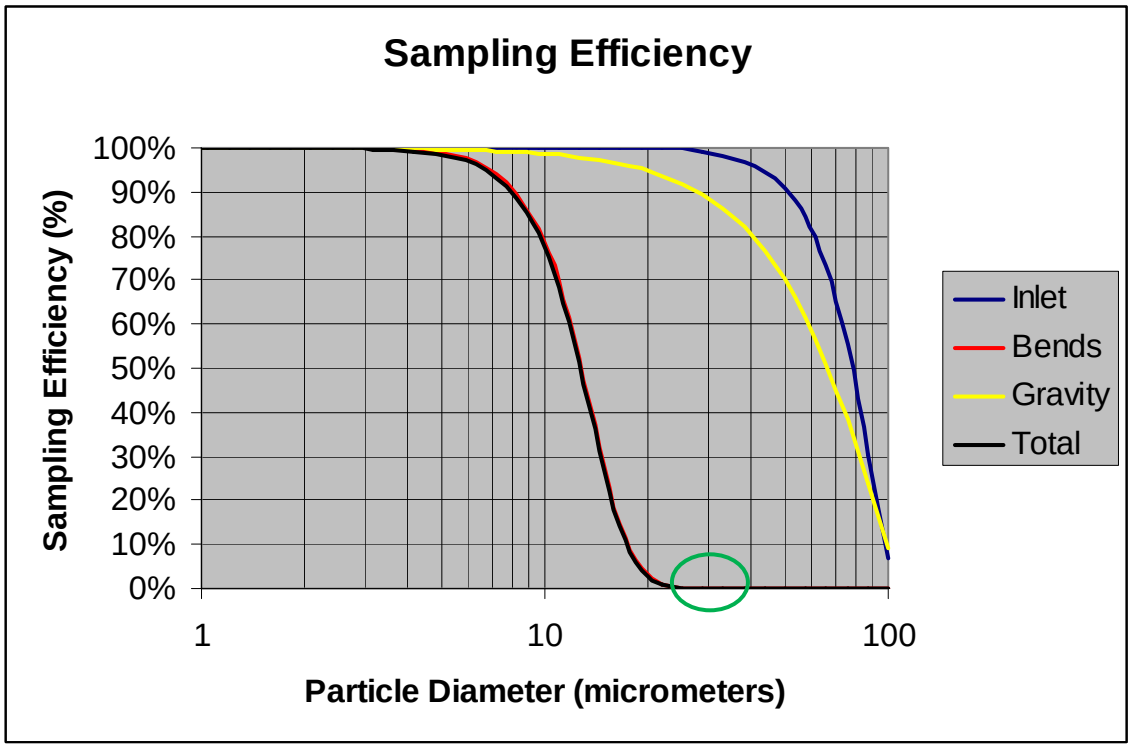
6.2.1. Aerosol Sampling Efficiency

An important factor in aerosol sampling is the collection efficiency of the biosamplers used to pull aerosol samples from the chamber environment. An assessment of our original system sampling efficiency was performed to determine if improvements or changes should be made to increase the aerosol sampling efficiency. Results of this assessment are outlined in the following paragraphs.

Sampling efficiency through the biosamplers was calculated for the following conditions:

- The sample line is 0.5 inch OD with 0.060 inch walls for an ID of 0.380 inch.
- The sample line length is 1 meter horizontal run
- There are two 90 degree bends in the sample line
- The sample inlet faces downward and draws the sample up.
- The sample flow is nominally 10 liters per minute

Figure 12 – Sampling Efficiency of the Sampling Lines (as measured in the 14.5 m^3 test chamber at the start of the project).

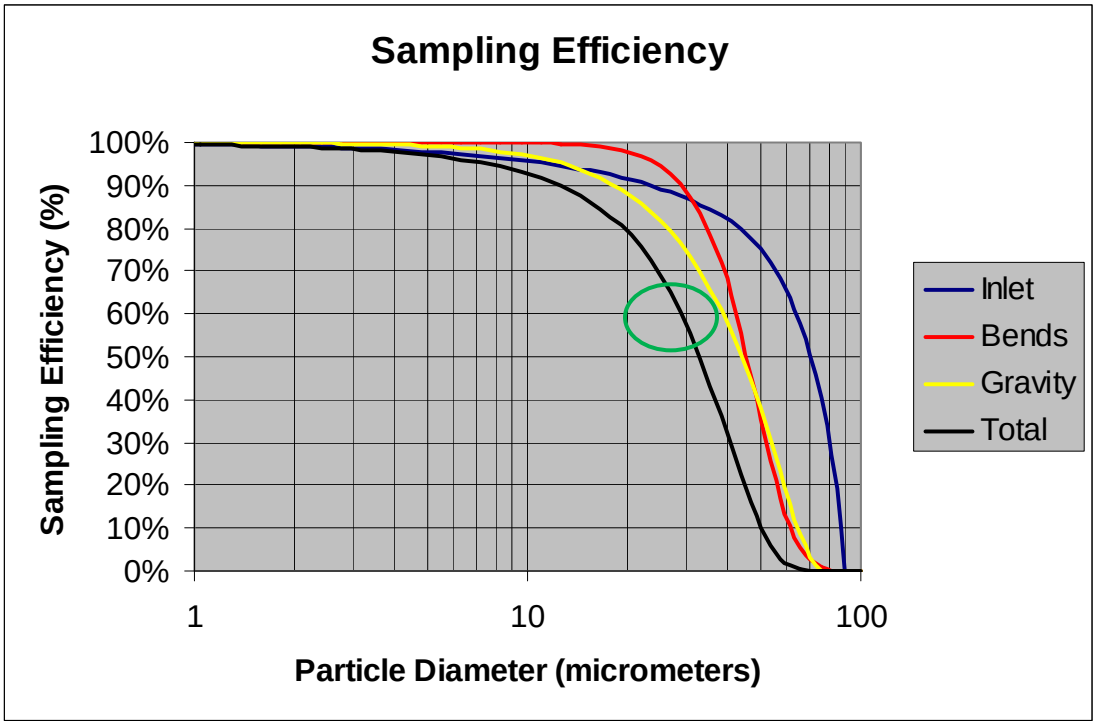


Consider the example of measuring 30 micrometer particles. Results indicate that we will not collect any, and that this is due to bend losses. If we change the sample line to 1 inch OD with 0.060 inch walls we get better sampling efficiency, as described below.

The sample line is 1.0 inch OD with 0.060 inch walls for an ID of 0.380 inch.

- The sample line length is 1 meter horizontal run
- There are two 90 degree bends in the sample line
- The sample inlet faces downward and draws the sample up.
- The sample flow is nominally 10 liters per minute

Figure 13 – An Example of Increased Sampling Efficiency in the 14.5 m^3 Test Chamber (obtained by modification to sampling line diameter, increased diameter).



By changing the sample tube diameter, we were able to sample more than 50% of the 30 micrometer particles (as noted by the green circle).

For sampling efficiency and collection of vapors using the SKC aerosol samplers, one assumption was that these devices collect at a high rate. In their work, Hogan et al shows size dependent collection efficiencies for particles down to about 15 nanometers. Collection efficiency for the SKC sampler drops to a minimum of about 5 % for 50 to 30 nanometers and then increases to about 10% at 20 nanometers and up to 35% at about 15 nanometers indicating the increased collection efficiency from diffusion. Gas has a much higher diffusivity and as long as the agent gas is soluble in the liquid we would expect high collection efficiency of gases and vapors.

6.3. Experimental Aerosol Chamber Test Description

6.3.1. Aerosol Chamber Test Description

The typical aerosol chamber test was performed as follows: The 14.5 m^3 aerosol test chamber was charged with CW agent simulant for a duration adequate to achieve airborne aerosol concentrations at customer-defined threat densities. Aerosol samples were collected throughout the simulant charging process using the SKC aerosol BioSamplers with iso-octane as the collection medium. A charged DF200 spray was deployed for a predetermined duration, typically 1 or 2 minutes, using specified spray system parameters such as nozzle air pressure, liquid pressure, etc. Test spray parameters were based on results of nozzle spray characterization profiles. Typical charged spray droplet sizes were an average of 30 µm, dispersed at a spray density of ~ 120 - 140 g/m^3. Aerosol concentrations were again measured immediately after the end of the charged spray deployment, and at selected times following the charged spray deployment. Sampled aerosolized CW simulant are separated from the aqueous DF200 and solubilize in the organic iso-octane collection medium phase, neutralizing the decontamination reaction to indicate aerosolized simulant concentration representative of the timeframe from which the sample was collected. Chemical agent simulant (vapor and particle) collected in the iso-octane was analyzed by gas chromatography (GC). Results were reported as aerosol concentration (gm/m^3) and plotted versus time (minutes). All spray system parameters were monitored and tracked electronically by a Data Acquisition System.

6.3.1.1. Aerosol Chamber Test Protocol

Aerosol chamber tests were performed using the protocol attached in Appendix B. The protocol describes at a high level, the supply preparation, aerosol sampling, aerosol sample processing, isooctane extraction, extracted simulant dilution and preparation for GC analyses, and general clean-up procedures.

In preparation for aerosol test chamber simulant testing, the GC analytical methods were evaluated to ensure compatibility with predicted simulant concentration ranges and other chemistry issues such as appropriate collection solvent and solubility. The GC methods are attached in Appendix C.

6.3.2. Surface Sampling and Assessment of Aerosols Near Chamber Floor

To answer concerns of more spray knockdown to the bottom of the chamber vs. spray knockdown and neutralization of airborne simulant, an assessment of liquid collected near the chamber floor was performed. A funnel and small beaker were set up to catch and collect spray fallout. Liquid sample was collected at 5 and 30 minutes post decon spray from floor level and immediately quenched in solvent (iso-octane). Results from GC analyses of pooled liquid samples collected near the chamber floor were all non-detectable.

Assuming adequate detection sensitivity, collection of aerosol samples immediately above the chamber floor could provide a means of monitoring off-gassing from the floor surfaces.

In future efforts, surface sampling may be enabled via chamber modifications. Additional ports and glove-handling can be added to accommodate surface sampling, particularly near the lower end of chamber. Sampling lower areas of the chamber atmosphere will provide assurance of decontamination efficacy despite any evidence that contaminants are forced (either through natural particle fallout or by more aggressive means) to the floor.

6.3.3. Aerosol Sampling of Total Airborne Matter

Glass fiber filter samples were also collected during the final few aerosol chamber tests. The purpose of the filter samples was to assess the total airborne concentration and to provide confirmation of pre-spray simulant concentrations and the rate of particle fallout following decontaminant spray. **Filter sample results also provided a comparison between the rate of post-spray particle fallout and the neutralization rate of simulant captured in vapor and aerosol droplets (as collected by BioSamplers and measured by GC)**. Results of glass fiber filter sampling are displayed in Figures 14 and 15. In addition to GC analyses of aerosol samples and APS measurements (of simulant only), the glass fiber filters served as a third method to confirm and correlate vapor and aerosol concentrations within the chamber.

6.3.4. Modifications to the Aerosol Test Chamber

Hardware upgrades and modifications to the aerosol chamber included calibration and installation of decon liquid and air flow meters (& incorporation into the Data Acquisition Center), as well as APS installation and operation. Calibrated nozzle air flow meter indicates a higher than expected total maximum air flow of 130 CFM, for a nominal flow of about 14 CFM/nozzle.

A primary setback was failure of the APS unit in July. A working unit was acquired and installed in the aerosol test chamber to resume testing in early August.

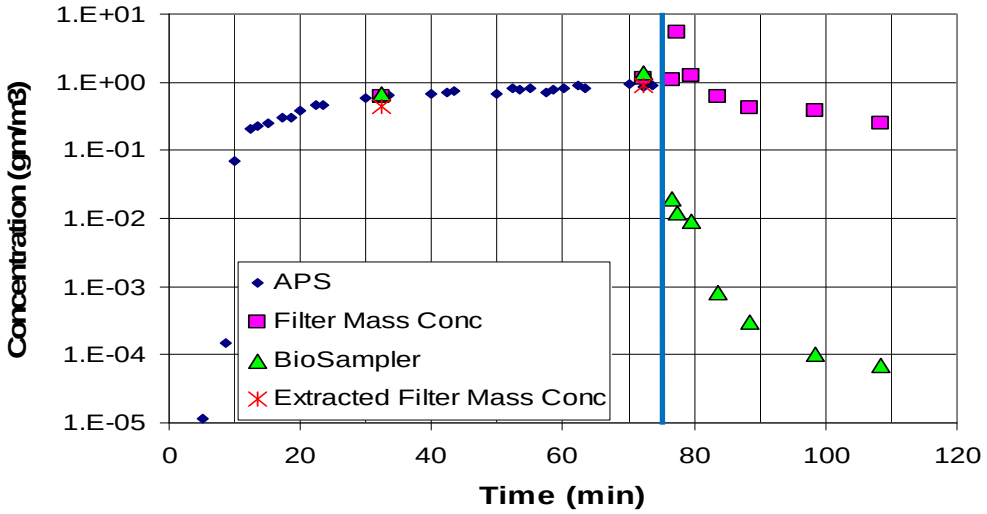
7. EXPERIMENTAL AEROSOL TEST CHAMBER RESULTS

Initial aerosol chamber test results indicated an average of greater than 2.5 orders of magnitude immediate decrease in G-agent simulant aerosol concentrations, an average of greater than 4 orders of magnitude decrease in G-agent simulant aerosol concentration 15 minutes after the DF-200 neutralization spray, and non-detectable levels of G-agent simulant 30 minutes following the DF-200 neutralization spray. Spray system parameters such as decon liquid flow rate, nozzle air pressure and collison pressure were varied, impacting the decon spray density, decon particle size and simulant concentration, respectively. Data plots are attached to this report in Section 7.1. Refer to Appendix A for a complete test matrix summary and data chart.

7.1. Representative Aerosol Chamber Test Results

Figure 14 – Plot of G-agent Simulant Neutralization

An expected 1-minute charged DF200 spray (decon spray time = 75-76 seconds, noted by the blue vertical line) in an **un-mixed** (a non-optimized condition) aerosol chamber environment. Note the post-decon spray rate of decrease in simulant vapor and particle neutralization (BioSampler data, noted by green triangles) is significantly greater than rate of total (simulant plus decon spray) particle fallout (glass fiber filter mass concentration, noted by magenta squares). This demonstrates that any airborne simulant collected as vapor and/or particles is quickly and effectively neutralized. Data plot represents results of test performed on August 10, 2009.



Two final test matrices were developed based on staged-spray strategies. The strategies were to 1), decrease air flow into the ESS nozzles during the second half of a 2-minute spray to produce larger charged droplets and increase capture of smaller droplets (via interception and impaction) as the smaller drops settle out (results in Figure 16), and 2) produce neutralized droplets during the second minute of a 2-minute spray (results in Figure 17). Although the rate of neutralization remained high, the neutralization rate using the charged/neutral approach was not as fast as the approach using charged sprays only. By comparison, charged sprays demonstrated an additional order of magnitude reduction in airborne simulant concentration within 5 minutes post-spray, and the airborne simulant concentration was not detected 25 and 35 minutes post-decon spray.

Figure 15 – Rapid Knockdown and Neutralization of Aerosolized G-Agent Simulant
~2-minute charged, staged DF200 Sprays (Reduced air flow during final 52 seconds). Data plot represents results of test performed on August 12, 2009.

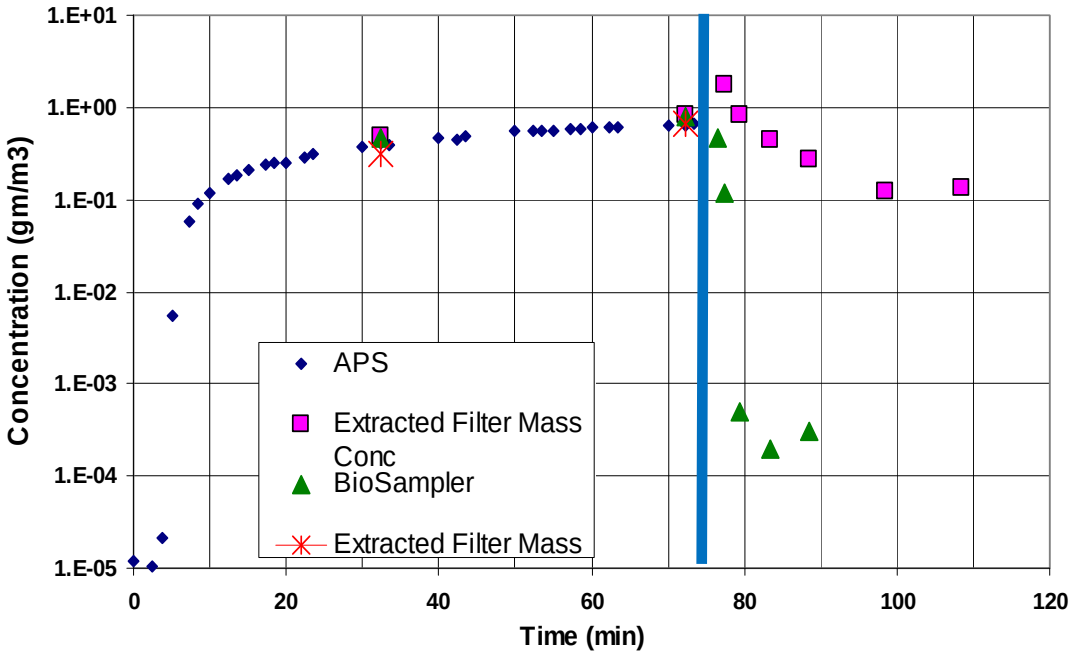


Figure 16 – Rapid Knockdown and Neutralization of Aerosolized G-agent Simulant
1-minute charged, followed by a 1-minute neutral DF200 spray; reduced air flow during final minute. Data plot represents results of test performed on August 17, 2009.

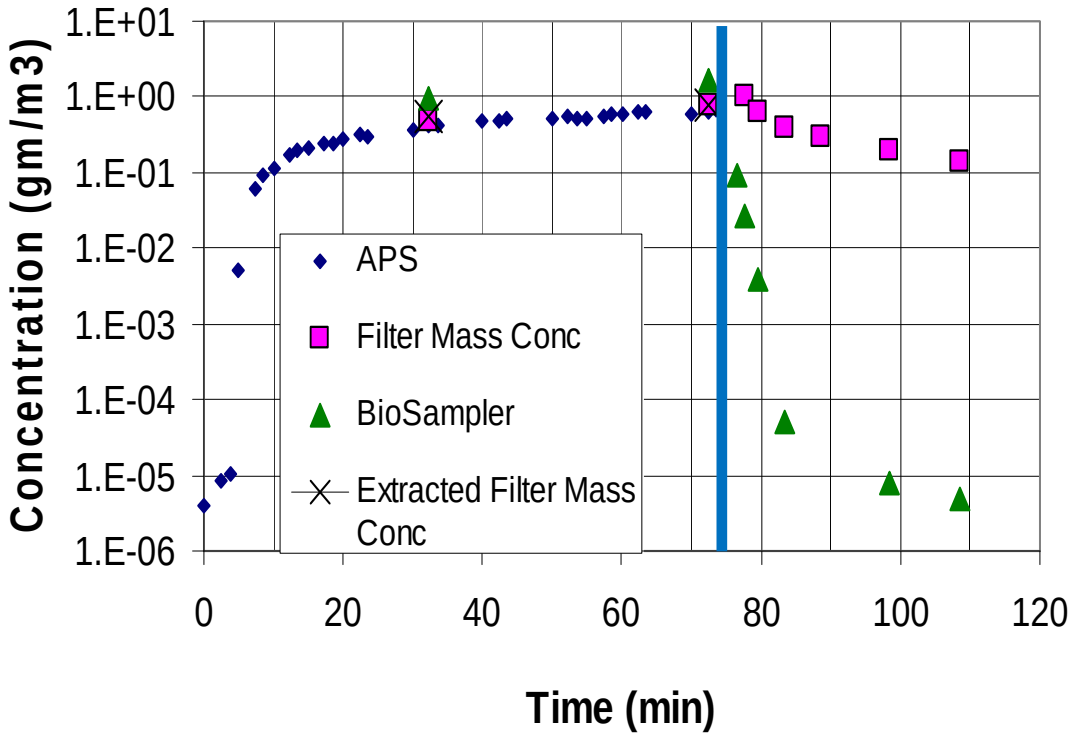
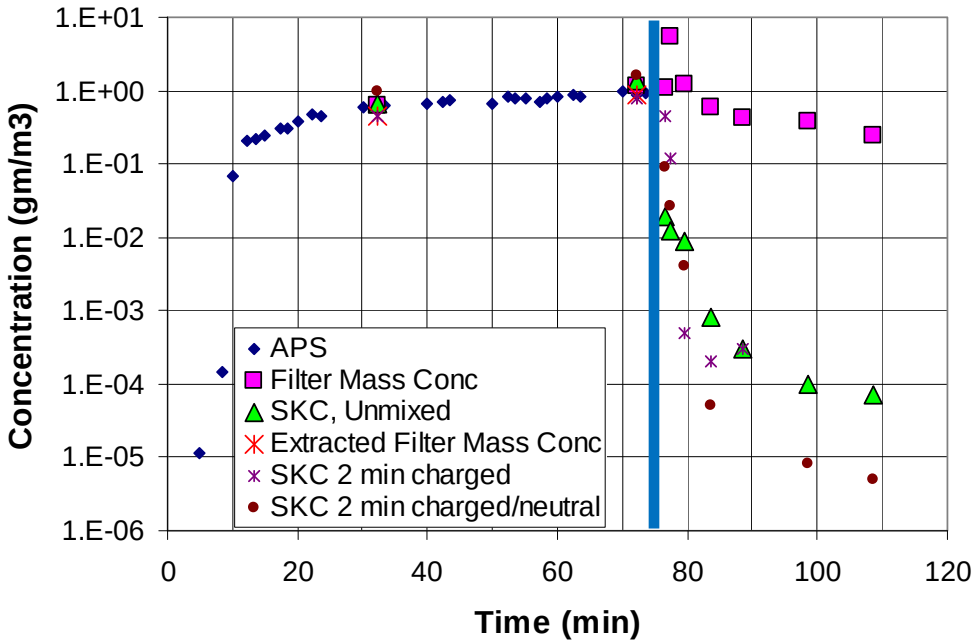


Figure 17 – A Composite Data Plot

Increased reduction in airborne simulant concentration is impacted by increased spray duration, staged spray droplet size change, and electrostatic charge. These impacts are displayed in this cumulative display of data collected from three different tests.



8. FULL SCALE OPERATIONAL GEOMETRY STRUCTURE

8.1. Explosive Destruction System Full Scale Operational Geometry

The current Explosive Destruction System (EDS) I and II are housed in Compact Al-Weather Mobile Shelter Systems (CAMSS30) which contains an internal rigid aluminum frame-support, soft wall canvas sides, temporary rubber flooring and doors. The CAMSS30 is a true semi-circle in cross-section, measuring 58.5 ft. L x 29.5 ft. W x 15 ft. H. Below are photos of the CAMSS30 shelters setup, located at the remote testing facility at Sandia Laboratories.

Figure 18 – Front View of CAMSS30 at Sandia Location

Figure 19 – Front View of CAMSS30 with EDS System Installed

8.2. Full Scale Operational Geometry – A New Test Capability

A Sprung Steel structure nominally 60 ft. L x 30 ft. W x 19 ft. H (at the highest center line) was designed, purchased and erected at the Aerosol Characterization Facility at Sandia National Laboratories. The total volume of the Sprung Steel structure is approximately 33,000 ft^3. The Sprung Steel Structure is a membrane covered aluminum frame structure, installed on a permanent concrete pad. There are no permanent utilities in this structure, but temporary electrical power and compressed air were installed. There are two personal access doors on each side of the structure and two 12' x 12' rolling service doors on either end. Installation required extensive planning for ES&H permits and procedures, as well as SNL site permits, as listed in Appendix D. A portion of the site preparation and installation costs were offset with contributing SNL Center 1500 support funds.

Since this structure is not a true semi-circle, a PVC frame was fabricated and installed inside this structure, and a plastic inner liner fitted over the PVC frame. This served two purposes: (1) to duplicate the inner geometry of the CAMSS30, and (2) to provide secondary containment for sprayed material. The structure emulates the actual shelter used in the field. It is located on the east side of a similar structure near Building 6540 in technical Area III at Sandia National Laboratories.

Figure 20 and Figure 21 – Sprung Structure Aluminum Inner Frame Installation and Completion

Figure 22 – Sprung Structure Membrane Liner Installation

Figure 23 – Sprung Structure Installation Complete

8.3. Full Scale Operational Geometry – Full Scale Spray Knockdown System

A full scale spray knockdown system was designed and fabricated to install directly into the existing CAMSS30 shelters. The system was modular in design and constructed from very light aluminum booms. The booms or manifolds contained the liquid, air and high voltage electrical feeds, could be assembled with quick connections and installed directly onto the aluminum inner structure of the CAMSS30 shelters. The spray booms contained the flow controllers for both the air and liquid feeds to the electrostatic spray nozzles. ESS personnel provided technical evaluation and consultation during the system design and installation

The system was designed to deliver approximately the same spray density as was being used in the spray characterization chamber. The spray system was very carefully characterized in terms of spray density, droplet particle size, and the effects of varying the air and solution feed rates on these parameters. There were two spray nozzles attached to each end of a 6’ 6” long spray boom, with 36 booms attached to the inner side of the PVC structure. The spray nozzles were spaced along the inner radius of the PVC sub-structure at 2’ 6” intervals. There were 18 spray booms at the front half of the system and 18 in the rear half, with a common manifold to feed the liquid and air for both halves of the spray system. There are a total of 72 spray nozzles in this system.

The system contained two pressurized solution tanks with capacities of 20 gallons each that could be filled and operated in various configurations. They could run independently, with pre-mixed DF200 in one tank and the other tank used to rinse the system with water; or, they could be used simultaneously with two separate solutions that were mixed in the mixing tube prior to feeding the liquid manifold for the spray nozzles. The first example allowed for the solutions to be prepared and the tanks to be filled well in advance of the actual testing, subject to the recommended maximum hold time for mixed reactive DF200 of 8 hours.

Figure 24 – Schematic of a Spray System Layout

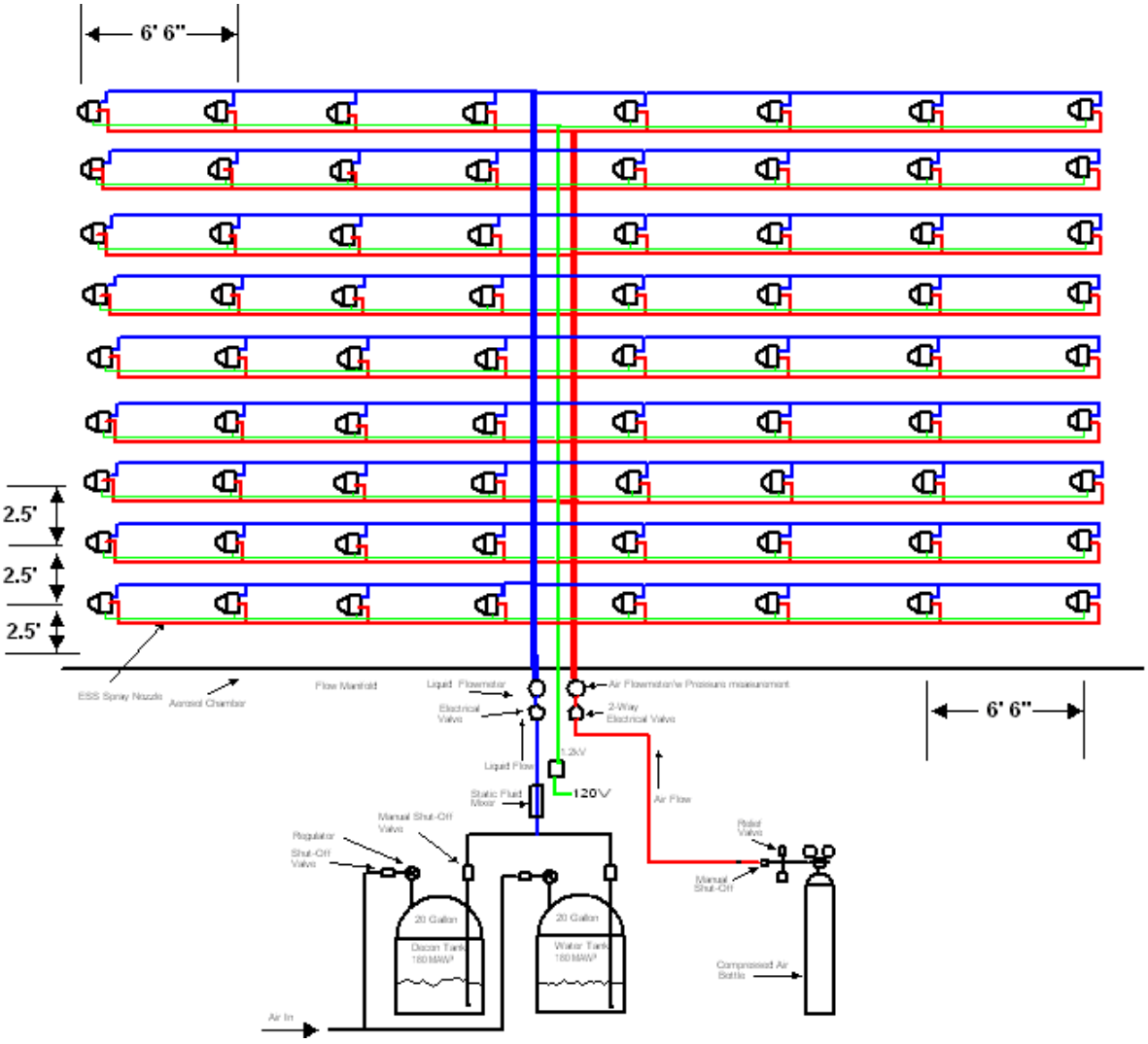


Figure 25 – Photo of Spray Nozzle Assembly Installed Within Tented Operational Geometry

Figure 26 ─ Photo of Tented Geometry inside Interior Sprung Structure

Figure 27 – Photo of a Single Spray Nozzle

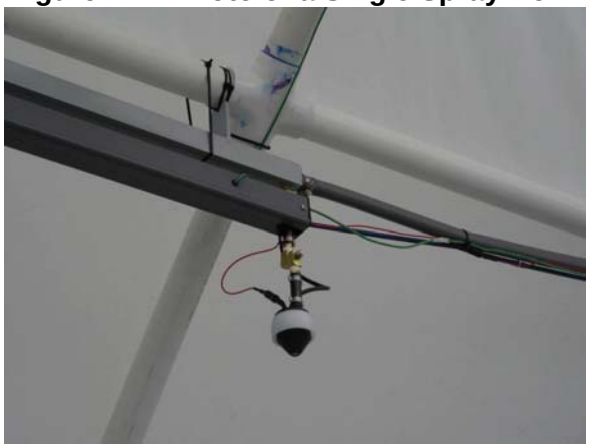


Figure 28 – Three Modular Spray Booms

9. R&D ROADMAP

9.1. R&D Roadmap Development

An extensive R&D Roadmap was presented to NSCMA during an internal program review (IPR) in September, 2009. The R&D Roadmap began with a summary of the project status (as of the IPR date), i.e., an operational geometry was installed at Sandia; the experimental data collected during the year strengthened the proof of concept, relative to the 14.5 m^3 test chamber; and the project team had begun to explore optimization strategies.

The follow-on R&D Roadmap was structured in phases, each with primary objectives. Approximate funding requirements and the proposed schedule and duration of each Phase are noted. Phases are described in greater detail in sections 9.1.1 through 9.1.3.

- **Phase I - Test and characterize performance of current prototype system**
  - $710K, 6 months
- **Phase IIA – Fielded System, Operational Demonstration & CONOPS**
  - $720K, 6 months
- **Therefore, from project start to Fielded System**
  - $2,065K, 22 months
- **Phase IIB – R&D to bound the Performance Envelope**
  - $1,350K, 12 months

The objectives identified in the R&D Roadmap to NSCMA were to: 1) test and characterize a prototype system performance, providing a go or no-go for full field application acceptance and readiness; 2) field a qualified mitigation spray safety system; and 3), obtain valuable R&D to understand and better define the depth and optimization of the spray knockdown capability, thus providing a matrix of known scenario applications. The total estimated funding requirement for the proposed R&D Roadmap provided to NSCMA in September 2009 was $3,415K. Further details regarding the estimates are not provided in this report, as the estimates are time-sensitive.

9.1.1. R&D Roadmap Development, Phase I, Test and Characterize performance of current prototype system

The overall objective of Phase I would be to test and characterize the performance of the current prototype system. This would begin by performing the planned, yet uncompleted, experimental tests in the current aerosol chamber. A total of 6 tests would be required to determine an optimized decontaminant chemistry for G-agent and mustard simulants. Test parameters would focus on use of an increased mixing fan capability, and the performance of staged sprays using optimized neutralization chemistry. Additionally, the MINICAMS interference assessment would be completed, requiring purchase of the appropriate software, as described in Section 4.1

Phase I would also include a demonstration to assess performance of the spray system performance and proof of concept in the full-scale operational geometry and volume. The demonstration would be based on Threat Scenarios 1 & 2 (described in Sections 3.1 and 3.2). DMMP (Dimethyl methylphosphonate, CAS # 756-79-6) is recommended as a simulant for the demonstration. DMMP is

considered to be a better simulant for G-agent physical characteristics (vapor pressure) than diphenylchlorophosphate, even though DMMP does not mimic the decontamination mechanism.

During Phase I, aerosol sampling would be assessed using a reduced number of sampling points in the operational volume. This factor became a requirement when calculations revealed that the sampling density required to sample at the historical level would be a logistically challenging and limiting factor (limiting in terms of personnel, analytical capacity and cost resources). The proposed sampling scheme is based on a 9 x 9 x 3 grid, with 21 aerosol samplers in a cluster design (6 samplers/cluster), for a maximum total of 126 samples collected per sampling time. The samplers would be distributed at three vertical levels, high, mid- and low throughout the full-scale volume chamber. The project team estimated a cost of $6K per sampling node, with a 4 week installation. Additional Lab View programming would be required to monitor and track sampling data. A large pump for adequate flow would be required to enable the expanded aerosol sampling capability. Adding the increased mixing capability would require the purchase of DC valves and a DC power supply. The total estimated cost for hardware was $185K.

Another feature of Phase I presented to NSCMA was the incorporation of the **B**uilding **R**estoration **O**perations **O**ptimization **M**odel (BROOM) to increase the speed, ease and accuracy of sampling collection, track analyses, and provide sampling and simulant analyses contour maps.

Spray system functionality testing would be required to test manifolds, switch and high voltage controls, etc. It was estimated the functionality testing would require 4 weeks to complete, at a cost of ~$118K labor plus purchases.

Additional scheduling and cost estimates for Phase I:

- 1 pre-Demonstration readiness test – 1 week preparation, 1 week to assess, perform
- Deliver Full-Scale Demonstration – 1 week preparation, 1 week to assess, perform and preliminary report
  - Provides proof of concept in operational geometry and volume
- Analysis and Reporting
- Management and Development
- Travel to CWD 2010 for 4 staff personnel
- BROOM Integration
- Schedule:
  - Months 1-3: Purchases, installation
  - Months 4 and 5: Characterization of operational volume
  - Month 6: Pre-demonstration readiness
  - Month 7: Final Demonstration
  - May, June: CWD 2010

At the completion of Phase I, a go/no-go for full scale application would be apparent. The total estimated cost of Phase I was expected to be ~ $700-$720K.

9.1.2. R&D Roadmap Development, Phase IIA, A Fielded System (in 2010)

The overall objective of Phase IIA would be to field, install and test a prototype release mitigation spray safety system. To achieve this, the initial task was defined as “assembly and deployment of a prototype”, and would require the following subtasks, all involving significant hardware purchases,

installation and operation: (1) A spray system mounted to the existing structure, (2) additional sensors (automated or manual operation, to be determined), (3) hardware purchases such as tanks, plumbing, an air compressor, electrical modifications, upgrades and modifications to the existing data acquisition system, and sampling hardware, (4) detection pre-filters (to mitigate overload of the MINICAMS detection system upon spray system deployment), and (5) floor containment. Since the prototype would be installed at a site external to Sandia, hardware shipping and logistics need to be considered, as well as travel required for prototype spray system design. These added to an FTE estimate of 1 staff plus 1 technologist, ½ time for 6 months, for a total of 0.25 FTE.

The second task required to field, install and test a prototype system during Phase IIA is the development of CONOPS and Guidance for Use, at an estimated staffing level of 2 staff, ½ time for 6 months. An understanding of the current event response is imperative for satisfactory completion of this task.

Additional expenses were accounted under this task for travel and management support.

A final deliverable under this objective would be to perform an Operational Demonstration (Qualification Test) at a selected facility. This Qualification Test would require travel expenses for 5 project personnel, at four weeks per person. The objective of the Qualification Test would be to demonstrate acceptable performance of the prototype release mitigation spray safety system at the customer's selected site.

The schedule to accomplish Phase IIA would be 6 months from the identification of a location. The estimated cost to complete PhaseIIA was projected to be from $710-$725K.

Overall, the R&D Roadmap highlighted a plan to provide a fielded, demonstrated acceptable mitigation release safety spray system at the customer's location of choice. Phase IIA carried the project from the proof of concept stage, through to deployment-ready. Phase IIB, described in Section 9.1.3, describes additional R&D that would be required to understand and bound the potential performance of a mitigation release safety spray system.

9.1.3. R&D Roadmap Development, Phase IIB – Technology Development

To explore and bound the performance envelope of a mitigation safety spray system, an intensive modeling effort would first narrow the parameter space for experimental testing. A variety of different threat scenarios (agents, likely concentrations and particle/vapor distributions based on dispersion technique) would be tested and evaluated in the 14.5 m^3 aerosol test chamber in order to baseline system performance. Baseline tests would be followed by experiments in the full scale facility to pinpoint efficacy and mitigation safety spray system capability. An estimated minimum range of eight to twelve tests would be performed at the full-scale level over a four month schedule. Experimental testing in the full scale facility would require not only an increased aerosol sampling density, but also a confirmation of adequate sampling instrumentation density to confirm smooth concentration contours, thus providing greater instrumentation for enhanced spatial resolution. Conceptually, to increase the sampling capability, 21 sampling clusters, each housing 6 different aerosol sampling devices would be utilized throughout the full scale experimental space. Modeling and testing may dictate alterations in the conceptual sampling design or proposed sampling methodology.

Additionally, use of Sandia's BROOM tool will increase the speed, accuracy and ease of sample collection, as well as tracking sample analyses. BROOM can be used extensively to provide both

sampling and analyses contour maps. The data output from BROOM can be used in a real-life event to more accurately and quickly provide this important data to decision makers.

9.1.3.1. Agent/Simulant Selection

An R&D focus of Phase IIB would be to improve and optimize a prototype system that would effectively mitigate and neutralize a variety of agent releases. Decontamination efficacy would be evaluated over a range of simulant choices, based on the following physical properties, thus covering a wide range of potential agent:decontaminant scenarios for which to design and provide protection against.

- Vapor vs. particle
- Vapor insoluble vs. water soluble vapor
- Water insoluble particle droplets
- Water soluble particle droplets

9.1.3.2. Droplet Size, Charge, Chemistry

The four primary spray system parameters found to be significant in providing effective neutralization are droplet size; charge (for enhanced particle collection); chemistry optimization; and mixing. Droplet size may be altered by the use of staged sprays, or deploying multiple spray sizes to enhance overall efficacy performance. Charging may be altered by considering a wider magnitude of applied voltage and a change in polarity. How charging impacts aerosolized peroxide concentrations, as well as concentrations of other aerosolized decontaminant constituents would be another factor for consideration. Chemistry may be optimized by use of alternative surfactants or addition of different solvents that impart a greater solubility of the agent with the decontaminant.

9.1.3.3. Mixing, Macro- and Micro-scale

Mixing is the parameter that may be more heavily focused in the Phase IIB effort. There is potential for large gains in efficacy when there are an increased number of collisions and contact time between the aerosolized agent particle/vapor and aerosolized decontaminant. An initial mixing characterization would be performed in the test chambers, using propylene glycol smoke to assess flow and movement in the chamber. Three confirmation measurement methods that could be employed include 10 DustTracs (log data), glass fiber filters and GC/FID/FPD analyses.

Mixing in both the macro- and micro-scales would be assessed. On the Macro-scale, parameters to consider include the number and placement of fans, nozzle number, nozzle position and orientation, and the use of a multitude of nozzles or nozzle types to produce multiple droplet sizes. On the Micro-scale, factors such as air entrainment and droplet/chemistry interactions can increase mixing and contact time. An estimated 2 month schedule would be required to complete a thorough mixing characterization test plan.

Thus, in Phase IIB, performance optimization strategies would be primarily based on staged sprays and improved mixing. The total schedule for completion of Phase IIB was estimated to require 12 months; costs for travel to support increased program development activities, project management for staff plus one Post Doc employee were added to produce a total estimate of ~ $1,350K for determination of efficacy in an optimized full-scale system.

9.2. Ongoing Program Development

Project status briefings were presented at the bi-yearly U.S. Army CMA Environmental and Monitoring Roundtable meetings (November, 2008; March and November, 2009).

An abstract was submitted and accepted for presentation at the Chemical Weapons Demilitarisation Conference (CWD 2009), May 2009 in Warwickshire, UK.

Project staff members continue to brief other agencies who could potentially benefit from incorporation of the spray knockdown technology into their operations. More recently, presentations have been provided to the US Army Corps of Engineers, responsible for Recovery operations, prior to dismantlement and decontamination in the EDS at Spring Valley.

As this report is being written, a toxicological study assessing the potential respiratory effects of inhalation exposure to aerosolized DF200 sprays is in progress. Preliminary results were not available at the time of printing this report.

10. CONCLUSIONS

The spray knockdown technology continues to improve with respect to understanding the impact of changes to spray system parameters and system optimization. Initial data was collected that demonstrated an improved efficacy gained by the use of staged spray strategies. Additional gains in efficacy can be made by continuing to investigate the use of staged sprays, as well as improved mixing, both on the macro- and micro-scales. Obtaining a clearer understanding of the chemistry of aerosolized decontaminant sprays, i.e., aerosolized peroxide and other component concentrations may drive further formulation development.

A prototype mitigation spray safety system has been constructed within a full-size operational geometry at Sandia National Laboratories. Further projects will most likely focus on meeting specific, customer-supplied requirements for applications of interest, as well as improvements in performance by addressing the outstanding R&D objectives.

11. REFERENCES

1. F. Lees, *Loss Prevention in the Process Industries*, Butterworth Press 1980
2. Hogan, et. al., "*Sampling methodologies and dosage assessment techniques for submicrometer and ultrafine virus aerosol particles*," Journal of Applied Microbiology, 2005, 99, 1422–1434

APPENDIX A: AEROSOL CHAMBER TEST MATRIX AND RESULTS SUMMARY

G-Simulant Aerosol Test Results

Test #	Date	Simulant	Objective	Dispersal Time	ESS liq flow rate & Press	ESS Air Press (psia)	ESS Flowrate	Charge	Collision Pressure& flowrate	Decon	Spray Duration (min)	Results
1	June 8, 2009	G-Agent Diphenylchlorophosphate	Baseline	50 min (2.6g/m3)		~80	~9 cfm per nozzle	1500VDC	47.5 psia lpm	Easy Decon	1.0 min	< 2 log immediate decrease; nearly 4 log decrease at 15 min; non-detect at 30 min
2	June 15, 2009	G-Agent	Baseline	50 min (2.6g/m3)	1.28 lpm 59psia	83	~9 cfm per nozzle	1500VDC	38 psia lpm	Easy Decon	1.0 min	2 log immediate decrease; 4 log decrease at 15 min; 5 log at 30 min
3	June 17, 2009	G-Agent	Baseline	50 min (2.5g/m3)	1.45 lpm 64psia	83	~9 cfm per nozzle	**No Charge**	38 psia lpm	Easy Decon	1.0 min	2 log immediate decrease; 4 log decrease at 15 min; nd at 30 min
4	June 22, 2009	G-Agent Diphenylchlorophosphate	Baseline	50 min (2.4g/m3)	2.5 lpm 84psia	82	~9 cfm per nozzle	1500VDC	37 psia lpm	Easy Decon	1.0 min	2.5 log immediate decrease; 4 log decrease at 15 min; nd at 30 min
5	June 24, 2009	G-Agent	Baseline	50 min (2.3g/m3)	2.46 lpm 82psia	57	~9 cfm per nozzle	1500VDC	38 psia lpm	Easy Decon	1.0 min	< 2 log immediate decrease; 4 log decrease at 15 min; >4 log at 30 min
6	June 29, 2009	G-Agent	Baseline	50 min (2.1g/m3)	2.02 lpm 61 psia	72	~9 cfm per nozzle	1500VDC	38 psia lpm	Easy Decon	1.0 min	2.5 log immediate decrease; >4 log at 15 min; nd at 30 min
7	July 1, 2009	G-Agent Diphenylchlorophosphate	Baseline	50 min (2.3g/m3)	1.5 lpm 53 psia	75	~9 cfm per nozzle	1500VDC	37 psia lpm	Easy Decon	1.0 min	<2 log immediate decrease; >4 log at 15 min; nd at 30
8	July 9, 2009	G-Agent	Baseline	70 min (3.3g/m3)	1.6 lpm 59 psia	74	~9 cfm per nozzle	1500VDC	36 psia lpm	Easy Decon	1.0 min	2.5 log immediate decrease; 4 .5 log at 15 min; nd at 30 min

9	Aug 5, 2009	G-Agent	Lower air flow	70 min (3.6g/m3)	1.7 lpm 57 psia	54	~9 cfm per nozzle	1500VDC	36 psia	Easy Decon	1.0 min	2 orders of mag immediate; 4 orders of mag at 15 min; 5 orders of mag at 30 min
10	Aug 10, 2009	G-Agent	Lower air flow	70 min (2.9g/m3)	1.7 lpm 55 psia	54	~9 cfm per nozzle	1500VDC	36 psia	Easy Decon	76 seconds	UN-MIXED 2 log immediate decrease; 3.5 log at 15 min; >4 orders of mag at 30 min
11	Aug 12, 2009	G-Agent	Staged 2-minSpray	70 min (3.8g/m3)	1.7 lpm 55 psia	31	cfm per nozzle	1500VDC 2 minutes	36 psia	Easy Decon	99 seconds total spray; 52 seconds lower flow	Greater than 3 log immediate decrease; >3.5 log decrease at 15 min; ND at 30 min
12	Aug 17, 2009	G-Agent	Staged 2-minSpray	70 min (3.5 g/m3)	Data not available	Data not available	~9 cfm per nozzle	1500VDC 1 minute only	Data not available	Easy Decon	2.0 min	2.5 log decrease immediate; >4 log decrease at 10 min.; 5 log decrease at 30 min

APPENDIX B: AEROSOL CHAMBER TEST PREPARATION AND PROTOCOL

Tests were performed using the following protocol. The protocol describes at a high level, the supply preparation, aerosol sampling, aerosol sample processing, isooctane extraction, extracted simulant dilution and preparation for GC analyses, and general clean-up procedures.

OPERATING PROCEDURE FOR
Demil Simulant Testing

Building 823 Labs /1085/1089/1097/
Building 6542 and Sprung Structure
Revised: July 24, 2009
Department 06375

Prepared by
Mollye Wilson

OBJECTIVE:

To evaluate the neutralization of chemical simulant by SNL developed decontamination system.

MATERIALS:

Chemical Agent Simulant
SKC BioSamplers (SKC Model No. 225-9595, operated at ~10 liters per minute)
Data Acquisition System (LabView)
Aerodynamic Particle Sizer (TSI Inc., Model 3321)
SNL Aerosol Testing chamber (512 ft^3, 14.5 m^3)
Solvent-Isooctane
Vortex (VWR, Model: VM-3000, Catalog # 58816-121)
Centrifuge (Thermo Electron Corporation, Model: Precision Durafuge 100)
GC-FPD (Agilent Hewlett-Packard 6890, ChemStation software)
50mL Conical
Pipette
Balance (Mettler Toledo, Model: AX205)

EXPERIMENTAL OUTLINE:
Aerosol Chamber Testing - Chemical & Biological Systems, Dept. 06375

PRE-1085

1. Label (with numbers 1-20 or 1-40) and tare weigh 15mL conicals with caps. Leave form with weights in 823/1085.
2. Label GC vials with sample time letter (A, B, C…) and dilution factor.
3. Prepare GC to run pre-standards and rinses. (See GC protocols)
4. Package supplies for transport to 6542. Use the two yellow and black transportation containers.

   a. Place clean biosampler veins in bubble envelopes. Place in small clear plastic boxes /w blue lids with fragile stickers.
   b. Place clean biosampler cups with lids in plastic boxes.
   c. Cooler with ice packs (to keep return samples cool).
   d. Pre-weighted 15 mL conicals.
   e. 50 mL conicals (are kept in 6542 but we need to make sure that we maintain our supply.)
   f. 10 mL pipette tips or disposable transfer pipettes (for removing solvent layer of the sample)
5. Print Spreadsheet for test data (data template)

PRE-6542

1. Label biosampler cups with chamber port number (1, 5, 7) and sample time letter (A, B, C…).
2. Label 50 mL conicals (chamber port number (1, 5, 7) and sample time letter (A, B, C…) to match biosampler cups).
3. Add 20mL of solvent (ISOOCTANE) to biosampler. Use the Dispensette in chemical hood for consistent volume. (BrandTech Dispensette Organic 2.5-25mL)
4. Collect tare weight for labeled collison jar(s) (jar and cap) Use balance in chemical lab (Mettler Toledo Model AX205)
5. Add simulant to collison jars, collect weight. Generally approximately 30-50g /collison. Test parameters will determine.
6. Prepare decon for deployment (deployment is programmed in LabView)

TEST

1. Attach appropriate collison to appropriate location on the chamber (#1 on left side and #2 on the right side).
2. Attach first set of biosamplers to chamber (background on port #3 and first samples on ports #1, #5, & #7). Ensure proper biosamplers are in place and correct vacuum line (labeled with port number) is attached for each sampling interval and removed after sampling.
3. Collect background sample (programmed in LabView)
4. Remove biosampler unit (veins and cup). Cap off sampling port. Remove veins from cup and cap biosampler. (If veins need to be re-used, use them for a pre decon sample.)
5. Initiate simulant loading of chamber (programmed in LabView)
6. Ensure proper biosamplers are in place and correct vacuum line (labeled with port number) is attached for each sampling interval and removed after sampling. Biosamplers are generally loaded on opposite port locations (1, 5, 7 or 2, 6, 8) so that two sets can be loaded at a time leaving plenty of time to remove and reload before the next sample time. This is very important for the post decon sample as there is only 1 min in-between samples.

POST-6542 (can be started while chamber test is still running)

1. Transfer sample to 50mL conical (with matching label).
2. Pour DF-200 into biosampler bottom and let sit while sample is in the centrifuge.
3. Vortex sample (in 50 mL conical) (VWR model # VM-3000) Maximum speed (3200rpm) for 20 seconds
4. Centrifuge sample in 50 mL conical bottom tube (Thermo Electron Corp Precision Durafuge 100) at Speed= 5000 rpm for Time= 15 minutes) Max 6 samples at a time. Ensure centrifuge is balanced before starting.

5. Pre-decon samples –
    a. ensure there is not a bi-layer
    b. Transfer into labeled (1-20)/tare-weighted 15 mL conical. Add chamber port number (1, 5, 7) and sample time letter (A, B,C…) to the label – should match the biosampler and 50mL conical label.
    c. Refrigerate samples in cooler with ice packs in yellow and black transportation container.
6. Post-decon samples - Transfer solvent layer to labeled/tare-weighted 15 mL conical (use 10 mL pipette or transfer pipette). Solvent layer should be the top layer. Chill samples in cooler.
7. Empty remaining aqueous layer into liquid hazardous waste container and place empty 50 mL conicals in hazardous waste bag.
8. Empty DF-200 from first biosampler cup into following biosampler cups until all cups are decontaminated.
9. After last post decon sample is taken remove collison jars and cap with appropriate lid.
10. Rinse collison housing and stem with Methanol (MeOH). Collect rinseate for disposal in hazardous waste container.
11. Remove stem and place in MeOH and take into 6542 lab.
    a. Rinse collison stems multiple times with MeOH disposing of rinse in hazardous waste container
    b. Store collison stems in MeOH until next test.
12. Collect post weight of collison jar (with lid) and simulant.
13. Dispose of remaining simulant into hazardous waste container.
14. Empty remaining DF-200 from biosampler cups into the collison jars.
15. Empty DF-200 rinse into hazardous waste container.
16. Fill collison jars with remaining (not used) DF-200 and seal lids.
17. Place collisons with DF-200 into zip lock bag and place in clear plastic box for transport.
18. Place biosampler cups and dirty biosampler veins in clear plastic boxes. Cushion with paper towels.
19. Package samples for transport – cooler with ice packs in yellow and black transportation container.
20. Place plastic boxes with biosampler cups, veins, and collisons in transportation container.
21. Ensure all hazardous waste containers are closed and stored properly.
22. Transport to 823

<u>POST-823</u>

1. Process samples in hood.
2. Wipe off exterior of sample conical (to remove condensation).
3. Collect post weight of the 15 mL conicals with samples.
    a. Use the same balance used to collect tare weights (hood in 1085).
4. Prepare GC sample vials
    a. Vortex samples Maximum speed for 20 seconds
    b. Transfer aliquot into GC vial
    c. May need to dilute (pre-decon samples likely 150uL sample into 1650 uL of solvent or greater depending on test.) Always add the solvent first so the math on the spreadsheet works out correctly.
        i. Example: Test time A – Add 1650µL isooctane to GC vial, take weight, add 50µL from 1A, take weight, add 50µL from 5A, take weight, add 50µL from

7A, take weight, vortex and place on GC in location indicated on the sequence table.

d. May run undiluted (later post-decon samples likely can be run undiluted [600 µL of each 1A, 5A, 7A]. If not try at 900µl [300µL of each 1, 5, 7 or 2, 6, 8] to 1mL solvent) Always add the solvent first so the math on the spreadsheet works out correctly.

5. Run on GC following standard GC protocols.
   a. Run standards (2,6,9) pre and post-sequence run
   b. Run appropriate solvent rinses.
   c. Bake out
6. Complete spreadsheet with appropriate data and GC results. Spreadsheet is located in the Decon share file in the Chemistry folder in the DeMIL folder.
7. Report data.

Aerosol Chamber Testing – Fire & Aerosol Sciences, Dept. 01532

PRE

1. Leak check chamber
2. Program sampling sequence (LabView)
3. Take background biosampler and APS

TEST

1. Initialize simulant "loading"
2. Take Pre-decon biosampler and APS readings
3. Prepare decon system – ensure valve settings are correct
4. Deploy decon system
5. Initialize post-decon sampling sequence

POST

1. Deploy remaining decon (or extra if needed)
2. Wait appropriate time for decon to occur (30 min)
3. Deploy DI "rinse" water through the decon system
4. De-energize nozzle heads
5. Evacuate Chamber through carbon filter bank (TIME = 10 min)
6. Rinse off walls, ceiling, floors, and nozzle heads.
7. Remove nozzle head covers, rinse off contacts with DI water if needed
8. Remove liquid from the chamber to approved effluent storage tank
9. Leave chamber open to dry
10. Analyze and collate the LabView and APS data
11. Report LabView and APS data

APPENDIX C: GC ANALYTICAL METHODS

C.*1.* *"HI" GC method, for G-agent simulant at high concentrations.*

In preparation for aerosol test chamber simulant testing, the GC and GC/MS analytical methods were evaluated to ensure compatibility with predicted simulant concentration ranges and other chemistry issues such as appropriate collection solvent and solubility.

method: C:\CHEM32\1\METHODS\G_HI_0809ISO.M on: 8/14/2009 at 1:59:02 PM
Modified

Method Information
Method: Modified: C:\CHEM32\1\METHODS\G HI 0809ISO.M
8/14/2009 at 1:59:02 PM
Method for analysis phosphorous-based simulant for G-agent at high concentration.

Injection Source and Location
Injection Source: GC Injector
Injection Location: Dual
6890 G C METHOD
OVEN Initial temp: 100 'C (On) Initial time: 0.00 min Maximum temp: 280 'C Equilibration time: 1.00 min
Ramps: # Rate Final temp Final time 1 25.00 250 2.00 2
O.O(Off) Post temp: 100 'C Post time: 0.00 min Run time:
8.00 min

FRONT INLET (SPLIT/SPLITLESS) Mode: Pulsed Split Initial temp: 230 'C (On) Pressure: 15.68 psi (On) Split ratio: 65:1 Pulse pressure: 11.0 psi Pulse time: 1.50 min Split flow: 194.2 mL/min Total flow: 199.9 mL/min Gas saver: On Saver flow: 15.0 mL/min Saver time: 1.50 min Gas type: Helium

BACK INLET (SPLIT/SPLITLESS) Mode: Pulsed Split Initial temp: 230 'C (On) Pressure: 15.57 psi (On) Split ratio: 85:1 Pulse pressure: 9.0 psi Pulse time: 1.50 min Split flow: 251.6 mL/min Total flow: 257.3 mL/min Gas saver: On Saver flow: 15.0 mL/min Saver time: 1.50 min Gas type: Helium

COLUMN 1 Capillary Column Nominal length: 30.0 m Nominal diameter: 320.00 urn Nominal film thickness: 0.25 urn Mode: constant flow Initial flow: 3.0 mL/min Nominal init pressure: 15.69 psi Average velocity: 49 em/sec Inlet: Front Inlet

COLUMN 2 Capillary Column Nominal length: 30.0 m Nominal diameter: 320.00 urn Nominal film thickness: 0.25 Mode: constant flow Initial flow: 3.0 mL/min Nominal init pressure: 15.58 urn psi Average velocity: 48 em/sec Inlet: Back Inlet

Modified on: 8/14/2009 at 1:59:02 PM

Outlet: Front Detector Outlet: Back Detector Outlet pressure: ambient Outlet pressure: ambient

FRONT DETECTOR (FPD) BACK DETECTOR (FPD) Temperature: 250 'c (On) Temperature: 250 'c (On) Hydrogen flow: 150.0 mL/min (On) Hydrogen flow: 75.0 mL/min (On) Oxidizer flow: 130.0 mL/min (On) Oxidizer flow: 95.0 mL/min (On) Oxidizer Gas Type: Air Oxidizer Gas Type: Air Mode: Constant column+makeup flow Mode: Constant column+makeup flow Combined flow: 60.0 mL/min Combined flow: 40.0 mL/min Makeup flow: On Makeup flow: On

Makeup Gas Type: Helium Makeup Gas Type: Helium Flame: On Flame: On Lit offset: 2.00 Lit offset: 2.00 Photo mUltiplier: On Photo multiplier: On

SIGNAL 1 SIGNAL 2 Data rate: 20 Hz Data rate: 20 Hz Type: front detector Type: back detector Save Data: On Save Data: On Zero: 130.8 (Off) Zero: 0.0 (Off) Range: 0 Range: 0 Fast Peaks: Off Fast Peaks: Off Attenuation: 0 Attenuation: 0

COLUMN COMP 1 COLUMN COMP 2 Derive from front detector Derive from back detector

THERMAL AUX 1 THERMAL AUX 2 Unknown Thermal Aux Type Unknown Thermal Aux Type

VALVES POST RUN Valve 1 Off Post Time: 0.00 min
Description:

TIME TABLE Time Specifier Parameter & Setpoint

GC Injector

Front Injector: Sample Washes 3 Sample Pumps 6 Injection Volume 1.00 microliters Syringe Size 10.0 microliters PreInj Solvent A Washes 3 PreInj Solvent B Washes 3 PostInj Solvent A Washes 3 PostInj Solvent B Washes 3 Viscosity Delay O seconds Plunger Speed Variable Injection Speed 2500.00 microliters/minutes Draw Speed 100.00 microliters/minutes Dispense Speed 4000.00 microliters/minutes PreInjection Dwell 0.00 minutes

Instrument 1 11/5/2009 1:32:13 PM ANA Page 2 of 3

Modified on: 8/14/2009 at 1:59:02

PostInjection Dwell

Back Injector: Sample Washes Sample Pumps Injection Volume Syringe Size PreInj Solvent A Washes PreInj Solvent B Washes PostInj Solvent A Washes PostInj Solvent B Washes Viscosity Delay Plunger Speed Injection Speed Draw Speed Dispense Speed PreInjection Dwell PostInjection Dwell

PM

0.00 minutes

3 6
1.00 microliters
10.0 microliters 3 3
3
3
O seconds Variable 2500.00 microliters/minutes
100.00 microliters/minutes 4000.00 microliters/minutes

0.00 minutes
0.00 minutes

Instrument 1 11/5/2009 1:32:13 PM ANA Page 3 of 3

C.2. *"LO" GC method, more sensitive for lower G-Simulant concentrations*

method: C:\CHEM32\1\METHODS\G_LO_0809ISO.M Modified on: 8/14/2009 at 1:59:40 PM

Method Information
Method: Modified: C:\CHEM32\1\METHODS\G LO 0809ISO.M 8/14/2009 at 1:59:40 PM
Method for analysis of phosphorous-based simulants at low concentration.

Injection Source and Location
Injection Source: GC Injector
Injection Location: Dual
6890 G METHOD
C
OVEN Initial temp: 100 'C (On) Initial time: 0.00 min Maximum temp: 280 'C Equilibration time: 1.00 min
Ramps: # Rate Final temp Final time 1 25.00 250 2.00 2 O.O(Off) Post temp: 100 'C Post time: 0.00 min Run time: 8.00 min

FRONT INLET (SPLIT/SPLITLESS) Mode: Pulsed Split Initial temp: 230 'C (On) Pressure: 15.71 psi (On) Split ratio: 3:1 Pulse pressure: 30.0 psi Pulse time: 1.50 min Split flow: 9.0 mL/min Total flow: 14.6 mL/min Gas saver: On Saver flow: 15.0 mL/min Saver time: 1.50 min Gas type: Helium

BACK INLET (SPLIT/SPLITLESS) Mode: Pulsed Split Initial temp: 230 'C (On) Pressure: 15.71 psi (On) Split ratio: 1.3:1 Pulse pressure: 42.0 psi Pulse time: 1.50 min Split flow: 3.9 mL/min Total flow: 9.6 mL/min Gas saver: On Saver flow: 15 mL/min Saver time: 1.50 min Gas type: Helium

COLUMN 1 Capillary Column Nominal length: 30.0 m Nominal diameter: 320.00 um Nominal film thickness: 0.25 um Mode: constant flow Initial flow: 3.0 mL/min Nominal init pressure: 15.72 psi Average velocity: 49 em/sec Inlet: Front Inlet

COLUMN 2 Capillary Column Nominal length: 30.0 m Nominal diameter: 320.00 um Nominal film thickness: 0.25 Mode: constant flow Initial flow: 3.0 mL/min Nominal init pressure: 15.72 um psi Average velocity: 49 em/sec Inlet: Back Inlet

1
Instrument 11/5/2009 1:30:21 PM ANA Page 1 of 3

method: C:\CHEM32\1\METHODS\G LO_0809ISO.M Modified on: 8/14/2009 at 1:59:40 PM Front Detector
Outlet: Outlet pressure: ambient

FRONT DETECTOR (FPD)
Temperature: 250 'C (On)
Hydrogen flow: 150.0 mL/min
Oxidizer flow; 130.0 mL/min
Oxidizer Gas Type: Air
Mode; Constant column+makeup
Combined flow: 60.0
Makeup flow: On
Makeup Gas Type:

Flame: On Flame: On
Lit offset: 0.50
Photo multiplier: On
SIGNAL 1 SIGNAL 2
Data rate: 20 Hz
Type: front detector
Save Data: On
Zero: 130.8 (Off) Zero: *0.0*
Range: 0 Range: 0
Fast Peaks: o Fast Peaks: Off
Off
Attenuation: Attenuation: 0

method: C:\CHEM32\1\METHODS\G LO Modified on: 8/14/2009 at 1:59:40

Post Injection Dwell

Back Injector: Sample Washes Sample Pumps Injection Volume Syringe Size PreInj Solvent A Washes PreInj Solvent B Washes PostInj Solvent A Washes PostInj Solvent B Washes Viscosity Delay Plunger Speed Injection Speed Draw Speed Dispense Speed PreInjection Dwell Postlnjection Dwell 0809ISO.M PM

0.00 minutes

3 6
1.00 microliters
10.0 microliters 3 3 3 3
O seconds Variable 2500.00 microliters/minutes
100.00 microliters/minutes 4000.00 microliters/minutes

0.00 minutes
0.00 minutes Instrument 1 11/5/2009 1:30:21 PM ANA Page 3 of 3

APPENDIX D: RESOURCES REQUIRED TO PLAN, PURCHASE AND INSTALL NEW SPRUNG STRUCTURE

- ***All of the ES&H permits and procedures were written and approved; they included:***
  - *NEPA - Written and through first review process, - To purchase and install Sprung Structure.*
  - *Security Service Request - Written and Approved - To remove security fence or install a man gate.*
  - *PHS -Written and Approved - To install, Sprung Structure, Spray System and perform demo.*
  - *OP - Written and in Review - for construction activities, erect Sprung Structure.*
  - *Discharge Permits - Written and Approved - For discharge of DF200*
  - *Biological Survey Permit - Written and in Review - To determine that there are no wildlife issues*
  - *Dig Permits - Written and in Review - For Grading, leveling and surface preparation.*
  - *Utility Spotting Permit - Written and in Review - For spotting Utilities before surface preparation.*
- ***The costs for erecting Sprung Structure:***
  - *30' x 80' Sprung Structure with 2 man doors, and 2 roll up garage doors with Sky Lights - $73K*
  - *Site Preparation - $30K - Includes Clearing Site, Grading, Gravel work, Creating a Man Gate in Fence, Rental Equipment and Setup and installation of Sprung Structure.*
  - *Site Preparation with optional concrete footer - $38K - This is the preferred option, it would allow us to pour a permanent concrete floor later in the future, if we get additional funding and or customers.*
  - *Temporary Leak Proof Floor - $5K to $30K there are a lot of options here, we could go as simple as lay down a leak proof tarp or build a wood floor and seal with roofing material, or the most expensive option would be asphalt or concrete.*
  - *Spray System - $10 to $30K the details have not been worked out on this yet and are only a rough estimate.*

Estimated Total - $125K to $150K*

***** As provided in email dated 1/26/2009 from Dan Lucero.***

Appendix E: Modified Stirred Reactor Kinetics Efficacy Test Results

Efficacy of Sandia Formulations against CW Agents
ECBC Modified Stirred Reactor, 2010
(funded by NSCMA)

% Destruction Efficiency

Decon Reagent	CN		HD		HD in HM		GD		VX	
	No Metals	Metals	No Metals	Metals	No Metals	Metals	No Metals	Metals	No Metals	Metals
DF-200	99.5	94.5	99.9	99.9	99.9	99.9	99.9	99.9	100	100
SNL Modified # 1	98.9	99.1	99.9	99.9	99.9	99.9	99.9	99.9	100	100
SNL Modified # 2	99.8	99.2	100	100	100	100	99.9	99.9	100	100

Test Conditions:

- 50±2 °C for 6 hours, duplicate
- Two treatment variables - with and w/out added 320 mg Fe & 1.5 mg Cu
- Volumetric loading of 1:100, (agent:reagent), stirred
- Post-reaction extraction, analyses by GC/MS

DISTRIBUTION

1	MS0734	Rita Betty	06375	
1	MS1135	Dan Lucero	01532	
1	MS1135	John Brockmann	01532	
1	MS0734	Mark Tucker	06375	
1	MS0614	Jonathan Leonard	02546	
1	MS0734	Mollye Wilson	06375	
1	MS0734	Matt Tezak	06375	
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