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ECBC-TR-435

EVAPORATION RATES OF DECONTAMINATION SOLUTIONS FROM OPERATIONALLY RELEVANT SUBSTRATES

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The findings in this report are not to be construed as an official Department of the Army position unless so designated by other authorizing documents.

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The evaporation rates of two decontamination solutions, DeconGreen™ and DF200™, were measured from surfaces with multiple and independent methods. Evaporation was measured by mass of residual decontaminant and video image processing. A wind tunnel was used to simulate an arid climate, with a low relative humidity and high temperature. The decontamination solution, DF200™, evaporated faster from bare and CARC-coated aluminum by a factor of approximately 2.8 compared to DeconGreen™.

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PREFACE

The work described in this report was authorized under Contract No. DAAD13-03-D-0017, Task Order Nos. NC-23 and Geo-NC-33. This work was started and completed in July 2005.

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EVAPORATION RATES OF DECONTAMINATION SOLUTIONS FROM OPERATIONALLY RELEVANT SUBSTRATES

1. INTRODUCTION

When choosing a decontamination technology, several factors under consideration include the type of contaminant, surfaces to be treated, and effectiveness of the decontaminant for the given situation. Another important quality is the climate. U.S. Army Regulation AR 70-38 outlines the different climate conditions expected throughout the world.[1] Environmental conditions may preclude the use of a decontaminant due to the physical properties of the decontaminant at certain environmental conditions. A liquid decontaminant may become too viscous to apply when exposed to low temperatures; humidity may impact the sorption capacity of solid-state decontaminant technologies; and volatile active ingredients may evaporate at higher temperatures and reduce effectiveness of the treatment. Therefore, environmental and climactic conditions may effect the "working time" for using decontamination technologies.

1.1 Background.

The purpose of this work is to establish a baseline of activity for a decontamination solution as determined by physical state. These decontamination solutions contain mixtures of volatile and non-volatile components. An assumption is made that effective decontamination requires the decontaminant to remain on the surface and in a liquid state. Upon evaporation of the volatile compounds and the drying of the solution, the decontaminant is considered exhausted.

The goal of these experiments was to determine the evaporation rate for both DeconGreen™ and DF200™ from operationally relevant surfaces with a given set of environmental conditions. The decontamination efficacies of DeconGreen™ and DF200™ are outside the scope of this study. Efficacy studies are documented elsewhere.[2,3,*,**] The scope of this work is to establish the ability of the decontaminant to remain in the liquid state over time within given environmental conditions.

The evaporation process was measured with several independent techniques. Samples were contained within the controlled environment of the wind tunnel. The samples were monitored with video analysis to document changes to

*Brickhouse, M.D.; Hall, M.; Henderson, V.; Procell, L.; O'Connor, R.; Reiff, L.; Sumpter, K.; Winemiller, M. *Stirred Reactor Decontamination Studies of DF200 Formulations with VX, HD, and GD;* U.S. Army Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD; unpublished data January 2006; UNCLASSIFIED Report.

**Wagner, G.; Procell, L.; Sorrick, D.; Hess, Z.; Gehring, D.; Henderson, V.; Brickhouse, M.; Rastogi, V.; Turetsky,A. *Development of New DECON GREEN™: A How-To Guide for the Rapid Decontamination of CARC Paint*; U.S. Army Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD; unpublished data January 2006; UNCLASSIFIED Report.

decontamination solution and periodically weighed to determine mass loss. The environmental conditions chosen represented an arid or desert climate, with negligible relative humidity, air temperatures ranging from 32- to 49-°C and a maximum surface temperature of 63 °C.[1]

1.2 Overview of Decontamination Solutions.

The DF200™ is a fielded decontamination solution technology. According to a 2003 memorandum assessing the military utility of the decontamination solution, "DF200™ is a three part aqueous solution containing quarternary ammonium compounds and hydrogen peroxide with a final pH of approximately 10."[2] Water and volatile compounds comprise approximately 75% of the total volume of DF200™.

DeconGreen™ is an emerging technology. Similar to DF200™, DeconGreen™ is also a three part aqueous solution and a base-activated peroxide technology. However, DeconGreen™ is a different blend of surfactants and activator compounds. Water and volatile compounds comprise approximately 50% of the total volume of DeconGreen™.

2. INSTRUMENTATION AND MATERIALS

2.1 Wind Tunnel.

The U.S. Army Edgewood Chemical Biological Center (ECBC) Wind Tunnel is a stainless steel chamber designed to measure and monitor evaporation processes within a controlled environment. A photograph of the ECBC Wind Tunnel is shown in Figure 1. The ECBC Wind Tunnel was designed by the Aerodynamics Group of the Smoke and Target Defeat Team for use in the Agent Fate Program. This technology is leveraged for the experiments in this study, transitioning it from Agent Fate Program to decontamination studies.

Samples of interest are inserted via a Teflon piston into test section of the tunnel. A series of feedback circuits and sensors automatically control and record the wind velocity, relative humidity (RH), and pressure within the test section. Eight independent thermocouple and heater sets monitor and control portions of the wind tunnel to ensure the entire apparatus maintains the set temperature. The temperature of the air and the substrate are also monitored and controlled. The instrument was set for a low humidity value. However, the system is not calibrated to measure humidity < 5% RH. Therefore, the system is assumed to have an RH between 0 and 5%.

The ECBC Wind Tunnel is controlled by a custom computer code. This application program enables complete control over the environmental parameters and conditions inside the tunnel and securely logs the data for analysis. Sensors in the tunnel provide feedback, whereas algorithms facilitate adjustments to automatically match the target settings. A schematic represents the regions of the tunnel and shows

the target and actual parameters. The graphical interface is shown in Figure 2. Complete documentation is being compiled in a separate reference publication.

The ECBC Wind Tunnel also incorporates video equipment for monitoring and measuring the evaporation process visually. The glass window in the side of the tunnel enables the capture of liquid film profile video footage.

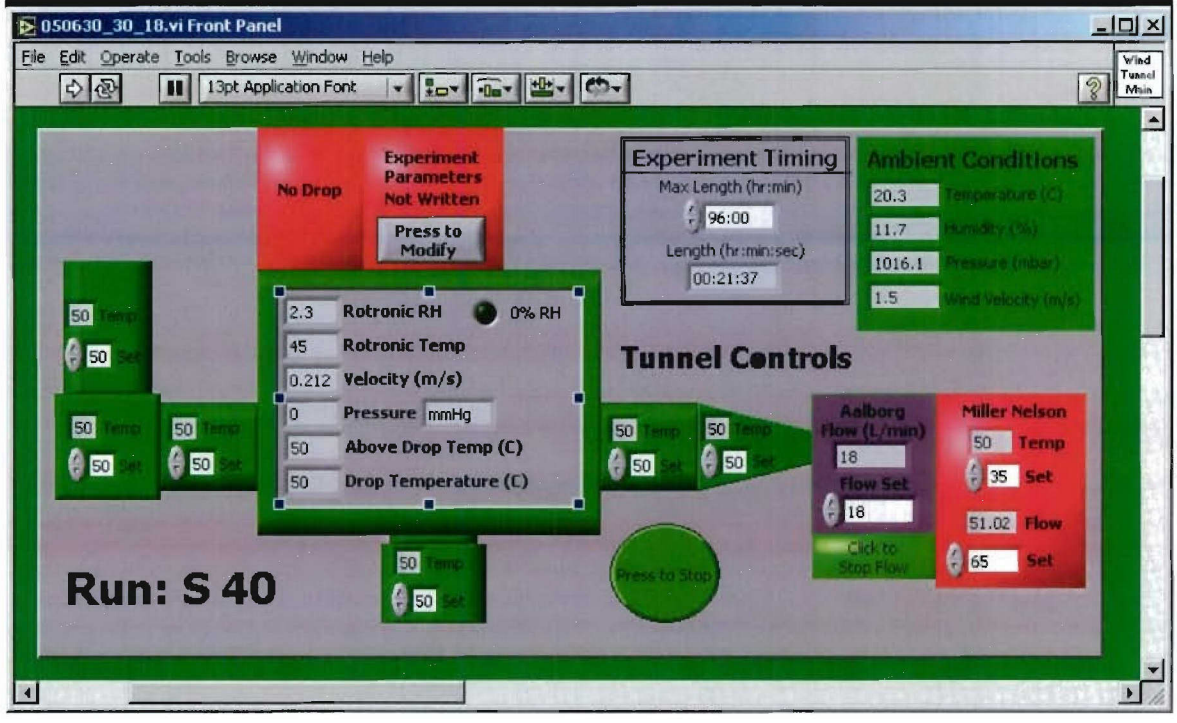
2.2 Video and Photographic Equipment.

Digital photographs were collected with a Canon Digital Rebel camera in fully automatic mode and fitted with an 18-55 mm general purpose lens. Video footage was collected using a Sony XC-ST50 camera fitted with a Fugi HF50HA-1 lens and 30-mm tube extender. Analogue video signal was digitized with a Flashbus MV video capture card and processed using Image Pro Plus software (Media Cybernetics, Silver Spring, MD).

Figure 1. ECBC Wind Tunnel

Note: The ECBC Wind Tunnel was designed by the Aerodynamics Group of the Smoke and Target Defeat Team. This instrument enables the measurement of evaporation processes within a controlled environment. Incorporated features include precision wind speed, temperature and relative humidity controls with multiple video and vapor monitoring capabilities.

Figure 2. ECBC Wind Tunnel Graphical User Interface



Note: Software control module for the ECBC Wind Tunnels. The green schematic represents the regions of the tunnel and shows the target and actual parameters. Temperature, wind velocity, and relative humidity are monitored and controlled.

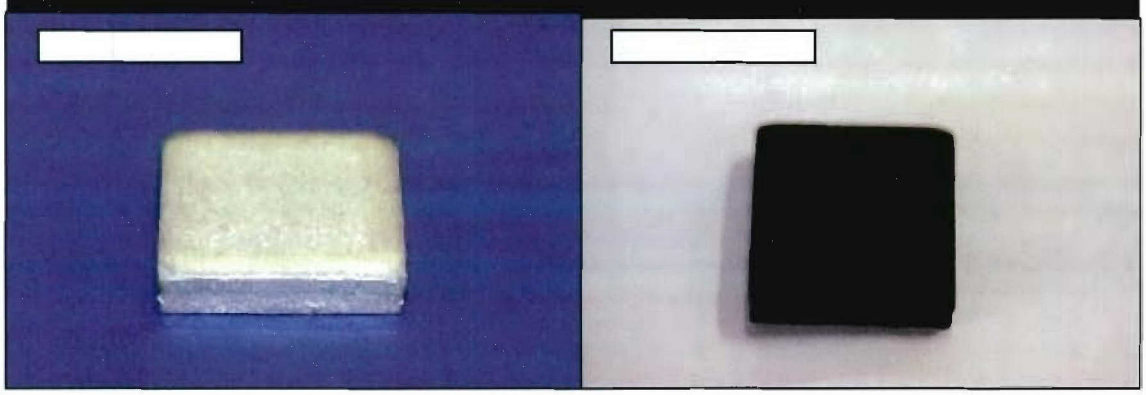
2.3 Coupon Materials.

The test materials selected are a subset of operationally relevent materials used for military vehicles and equipment. Sheets of aluminum 5052 were acquired and cut into 1.3 cm^2 coupons for testing. CARC-painted aluminum coupons were prepared by painting the aluminum squares in accordance with (IAW) with MIL-C-53039A, #383 Green.[4] The coupon preparation was performed by the ECBC Experimental Fabrication Shop. Figure 3 displays photographs of representative coupons.

2.4 Decontamination Solutions.

The decontamination solutions were obtained as commercial products. The lot numbers for the DeconGreen™ components were Part A, 1DG6-E4 Barcode (01)00724995028203 1; Part B, 1DG7-B5 Barcode (01)00724995082048; Part C, 1DG8-B5 Barcode (01)00724995084509. The lot number for the DF200™ components were 5313 6850-01-501-1044. Fresh batches of decontamination solution were mixed immediately prior to deposition onto the surface. The pH of each solution component was documented with pH paper ColorpHast 0-14 strips (E.Merck, Gibbstown, NJ) to verify the active ingredients.

Figure 3. Representative Coupon Samples



Note: Photographs of representative coupons for bare aluminum (left) and CARC coated aluminum (right). The white rectangles represent one centimeter.

2.5 Laboratory Equipment.

Sample mass was measured with a microscale balance (Sartorius – Model M 5E) and last calibrated on 4 March 2005. All other glassware, gloves, and supplies were standard laboratory grade.

3. TESTING AND ANALYSIS

3.1 Wind Tunnel Preparation and Equipment.

Two ECBC Wind Tunnels were set to the appropriate parameters and allowed to equilibrate overnight. One tunnel was used for the testing, and the second tunnel was used to condition the coupons samples prior to testing. Sample coupons were washed in soapy water, rinsed with distilled water, and allowed to air dry. The coupons were conditioned in another wind tunnel for several hours at the test conditions.

Distinction between major and minor changes of the sample was determined with image processing techniques. Image subtraction methods were used to determine the fraction of pixels displaying change.

3.2 Test Parameters and Procedures.

The ECBC Wind Tunnel parameters were set to 46 °C, 0% RH and a wind velocity of 0.22 m/s. This wind speed is equivalent of a 2 mph wind as measured 2 m from the surface. The cross sectional velocity profile had been characterized previously to correlate standard wind speed (at 2 m) and the boundary layer wind speed that affects the sample at ground level. Wind profiles were chosen and characterized IAW

standardized methods for small scale wind tunnels as outlined in Testing Requirements for Predictive Model Development using Hooded Evaporation Devices (HEDs).† These conditions represent an arid or desert environment as described in Army Regulation 70-38, Research, Development, Test and Evaluation of Materiel for Extreme Climatic Conditions.[1] According to Field Manual FM3-5, the application ratio of decontaminant to H class agents is 55:1, yielding an application volume approximately 1.0 mL /20 cm^2.[5] These values are based on the information for the DS2 decontaminant. However, smaller volumes were chosen for the deposition to ensure the decontaminant did not flow over the edges of the test coupon. Therefore, 20 µL of solution was applied to the bare aluminum coupon samples, while 6 µL of solution was applied to the CARC-coated aluminum coupons. Fresh batches of decontaminant were mixed within 10 min of each test. Each decontaminant was applied to the surface as a liquid. Although the name Decontamination Foam 200 suggests the technology to be a foam, the technology is only approved for deposition as a liquid.

The following procedures were followed for each test.

1) Mix a fresh batch of decontamination solution.

2) Retrieve a coupon from the conditioning wind tunnel.

3) Record the mass of the coupon blank.

4) Deposit an aliquot of decontamination solution on the coupon.

5) Obtain the mass of the sample.

6) Photograph the sample.

7) Contain the sample to protect from the atmosphere and transfer to the wind tunnel.

8) Insert the sample into the wind tunnel.

9) Observe the sample with video footage.

10) Remove the sample when the sample stops changing visually.

11) Contain the sample and transfer to the balance.

12) Obtain the mass of the sample.

†Kilpatrick, W; Ling, E; Hin, A; Brevett, C; Fagan, M; Murdock Jr., P ,"Testing Requirements for Predictive Model Development using Hooded Evaporation Devices (HEDs)," AFRL-HE-WP-TR-2004-DRAFT, Air Force Research Laboratory, unpublished data 2004.

13) Re-contain and re-insert the sample into the wind tunnel for a set time period.

14) Remove the sample, contain, and transfer to the balance.

15) Obtain the mass of the sample.

16) Repeat steps 13 -15 until the change in mass is less than 0.5 mg.

All time increments were measured using the computer collecting the video and logging the wind tunnel conditions. Using this common time source correlates the wind tunnel log, video timestamp, and timeline notations in the notebook.

4. RESULTS

The results are separated into three major sections as follows: individual evaporation charts, comparative plots, and tabulated mass accounts. Each section is detailed in the following paragraphs.

The individual evaporation results each follow the same general format. Each figure contains three sections consisting of a general timeline (yellow), a plot of the drop mass (pink), and video still frames (blue). All three sections correspond to the same time axis. The yellow section shows the overall timeline of the experiment, distinguishing the time periods for preparation, evaporation within the wind tunnel, and for obtaining the mass of the remaining drop fraction. The pink middle section displays a graph of the drop mass versus time. A color digital photograph was obtained during each weighing and was included with the corresponding mass data point. The blue bottom section displays periodic still images from the video obtained during the evaporation process.

Comparative evaporation figures display the sample mass and time lines of both decontamination solutions for a single substrate. Each set of comparative evaporation results follows the same format. The timeline for DeconGreen™ and DF200™ are shaded green and red, respectively. A direct comparison of sample mass is recorded along the same time axis. The data points for DeconGreen™ are noted with green circles, and the data points for DF200™ are noted with red triangles.

The tables document the mass of the drop, the percentage of loss, and the change from the previous mass. The time is listed as elapsed minutes from the first insertion into the wind tunnel.

The results for DeconGreen™ on bare aluminum are displayed in Figure 4 and documented in Table 1. The results for DF200™ on bare aluminum are displayed in Figure 5 and documented in Table 2. An evaporation graph comparing

DeconGreen™ and DF200™ on bare aluminum is displayed in Figure 6. The results for DeconGreen™ on CARC-coated aluminum are displayed in Figure 7 and documented in Table 3. The results for DF200™ on CARC-coated aluminum are displayed in Figure 8 and documented in Table 4. Image-enhanced video results for DF200™ on CARC-coated aluminum are displayed in Figure 9. An evaporation graph comparing DeconGreen™ and DF200™ on CARC-coated aluminum is displayed in Figure 10.

The results for DeconGreen™ on bare aluminum are documented in Figure 4. After insertion of the coupon into the tunnel, major changes were noted visually for the first 20 min. Major changes noted include the drop bubbling and the emergence of a large foam skeleton. Minor changes were noted visually for the next 30 min, including a slow collapse of the foam structure. The coupon was removed after 48 min and weighed. Upon exposure to the atmosphere, the foam structure rapidly deteriorated. The process of wind tunnel and sample weighing was repeated. No visual changes were noted during subsequent time periods within the wind tunnel. The sample was removed and weighed again. Upon completion of the experiment, the sample was placed in the open atmosphere. Several hours later, the foam structure noted at the end of the experiment was gone, but small drops of liquid were noted on the surface (data not shown).

Table 1 documents the mass of the drop, the percentage of loss, and the change from the previous mass. The time is listed as elapsed minutes from the first insertion into the wind tunnel. Thus, the initial sample mass was recorded 3 min prior to placement in the wind tunnel.

Table 1 - DeconGreen™ on Bare Aluminum

Elapsed Time from Wind Tunnel Insertion	Mass of Drop	Mass Loss	Change from Last Mass
min	mg	%	%
-3	23.9	-	-
42	7.9	67%	-
58	6.9	71%	12%
90	6.6	73%	5%

The results for DF200™ on bare aluminum are documented in Figure 5. Rapid bubbling of the solution was noted upon deposition onto the substrate. After insertion of the coupon into the tunnel, major changes were noted visually for the first 5 min. The major changes noted include the rapid evaporation of the liquid phase and the emergence of crystalline material on the surface. Minor changes were noted

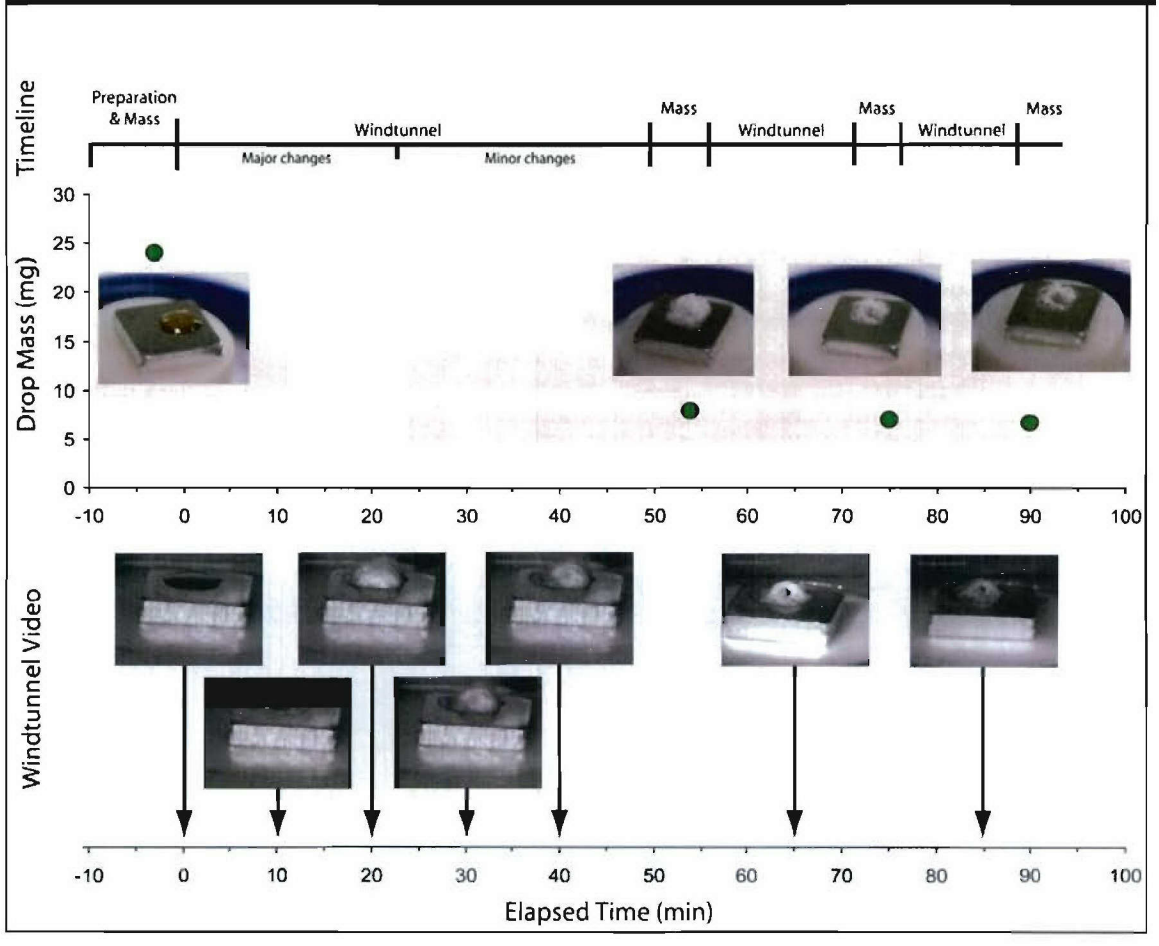
visually for the next 12 min and include a slower evaporation of liquid from the crystalline material. The sample was removed after 18 min and weighed. No changes were noted upon exposure to the atmosphere. The process of wind tunnel and sample weighing was repeated. No visual changes were noted during subsequent time periods within the wind tunnel. The sample was removed and weighed again.

Table 2 documents the mass of the drop, the percentage of loss, and the change from the previous mass. As with the other tables, the time is listed as elapsed minutes from the first insertion into the wind tunnel.

Table 2 - DF200™ on Bare Aluminum

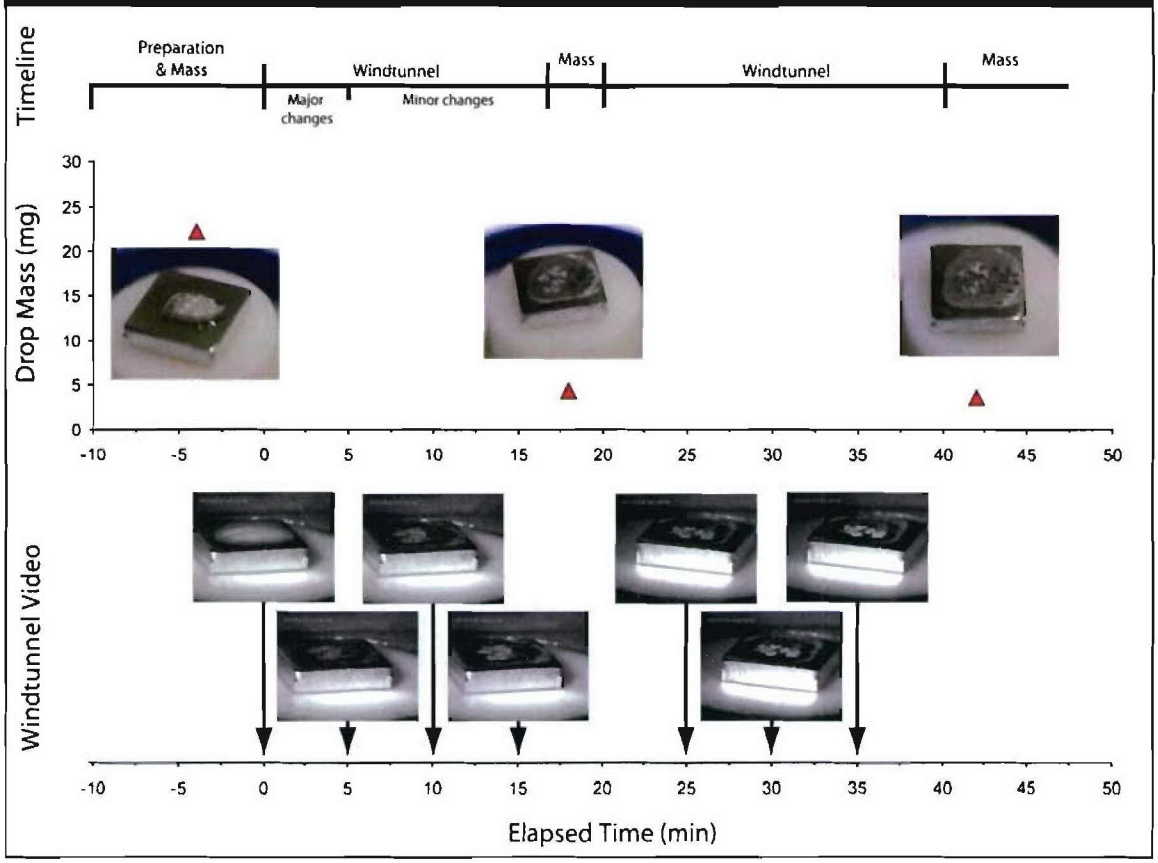
Elapsed Time from Wind Tunnel Insertion	Mass of Drop	Mass Loss	Change from Last Mass
min	mg	%	%
-4	22.2	-	-
18	4.3	82%	-
42	3.7	83%	14%

Figure 4. DeconGreen™ on Aluminum



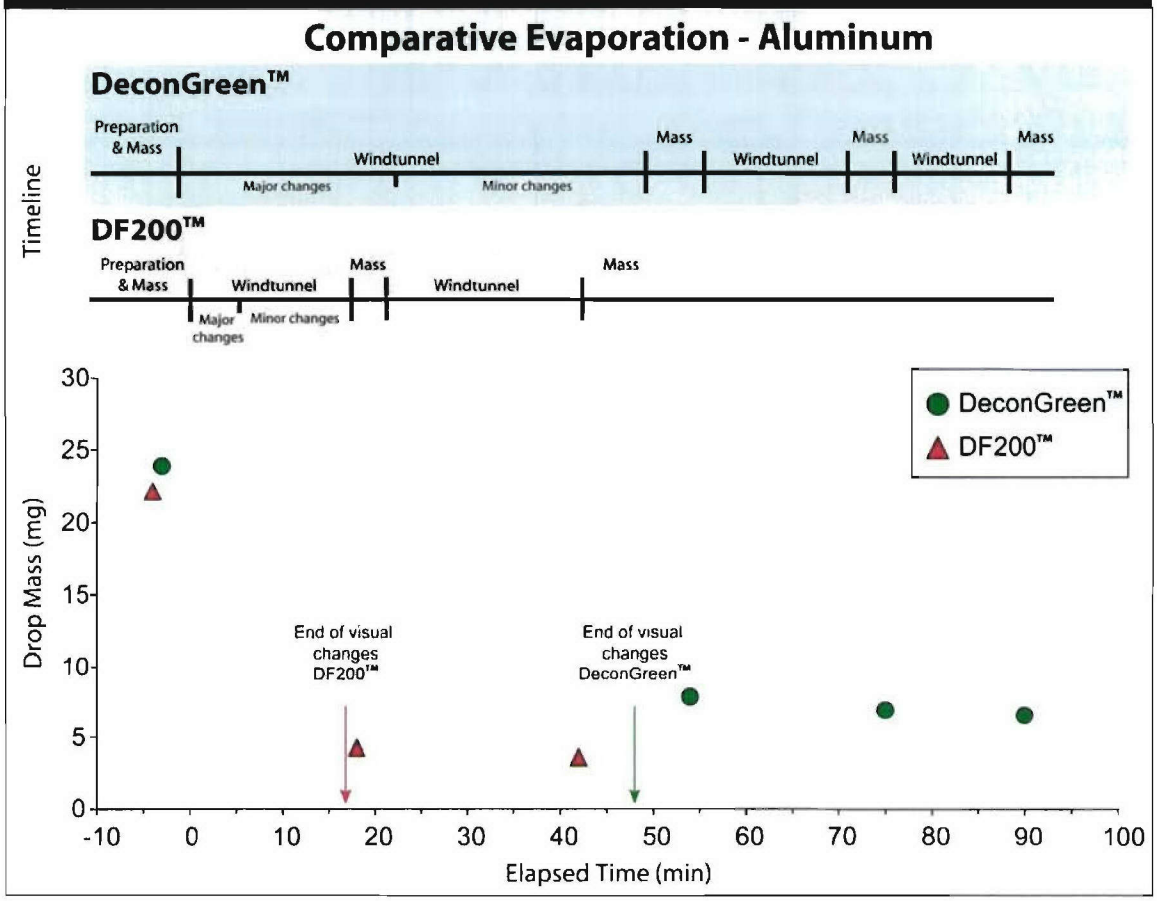
Note: Results for DeconGreen™ on bare aluminum. Timeline, drop mass, digital photographs, and wind tunnel video results all use the same time stamp. The formation of a large foam structure is noted as a major change in the timeline. Time zero is defined at the initial time point for sample placement in the wind tunnel.

Figure 5. DF200™ on Aluminum



Note: Results for DF200™ on bare aluminum. Timeline, drop mass, digital photographs, and wind tunnel video results all use the same time stamp. The evaporation of the liquid phase was noted as the major change in the timeline. Time zero is defined at the initial time point for sample placement in the wind tunnel.

Figure 6. Bare Aluminum Compiled Results



Note: Results comparing evaporation of DeconGreen™ and DF200™ on bare aluminum. Green shading in the timeline and green data points in the drop mass refer to DeconGreen™. Red shading in the timeline and red data points in the drop mass refer to DF200™. The timeline and drop mass results are plotted along the same time axis. The ends of visual changes are noted for both decontamination solutions.

The results for DeconGreen™ on CARC-coated aluminum are documented in Figure 7. After insertion of the coupon into the tunnel, major changes were noted visually for the first 11 min. Major changes noted included the drop boiling and the emergence of a cluster of numerous small foams. Minor changes were noted visually for the next 2 min as the foam structures individually grew and collapsed. The coupon was removed after 18 min and weighed. Upon exposure to the atmosphere, the foam structure rapidly deteriorated. The process of wind tunnel and sample weighing was repeated. No visual changes were noted during subsequent time periods within the wind tunnel. The sample was removed and weighed again. Several hours later, the foam structure noted at the end of the experiment was gone, but small drops of liquid were noted on the surface (data not shown).

Table 3 documents the mass of the drop, the percentage of loss, and the change from the previous mass. As with the other tables, the time is listed as elapsed minutes from the first insertion into the wind tunnel.

Table 3 - DeconGreen™ on CARC

Elapsed Time from Wind Tunnel Insertion	Mass of Drop	Mass Loss	Change from Last Mass
min	mg	%	%
-3	6.5	-	-
42	1.8	72%	-
58	1.7	74%	8%

The results for DF200™ on CARC-coated aluminum are documented in Figure 8. After insertion of the coupon into the tunnel, major changes were noted visually for the first minute. Major changes noted include the evaporation of the liquid phase. Minor changes were noted visually for the next 4.5 min as small individual crystalline growths appeared. The coupon was removed after 12 min and weighed. No changes were noted upon exposure to the atmosphere. The process of wind tunnel and sample weighing was repeated. No visual changes were noted during subsequent time periods within the wind tunnel. The sample was removed and weighed again.

Due to the rapid evaporation of DF200™ on CARC-coated aluminum, the major changes noted in the first minute are not captured in the blue section of Figure 8. Figure 9 displays video still images enhanced with image processing. The background has been subtracted to highlight the liquid phase. Figure 9 focuses on the first 180 sec of evaporation to emphasize the major changes of the sample due to evaporation. The timescale in Figure 9 is in elapsed seconds.

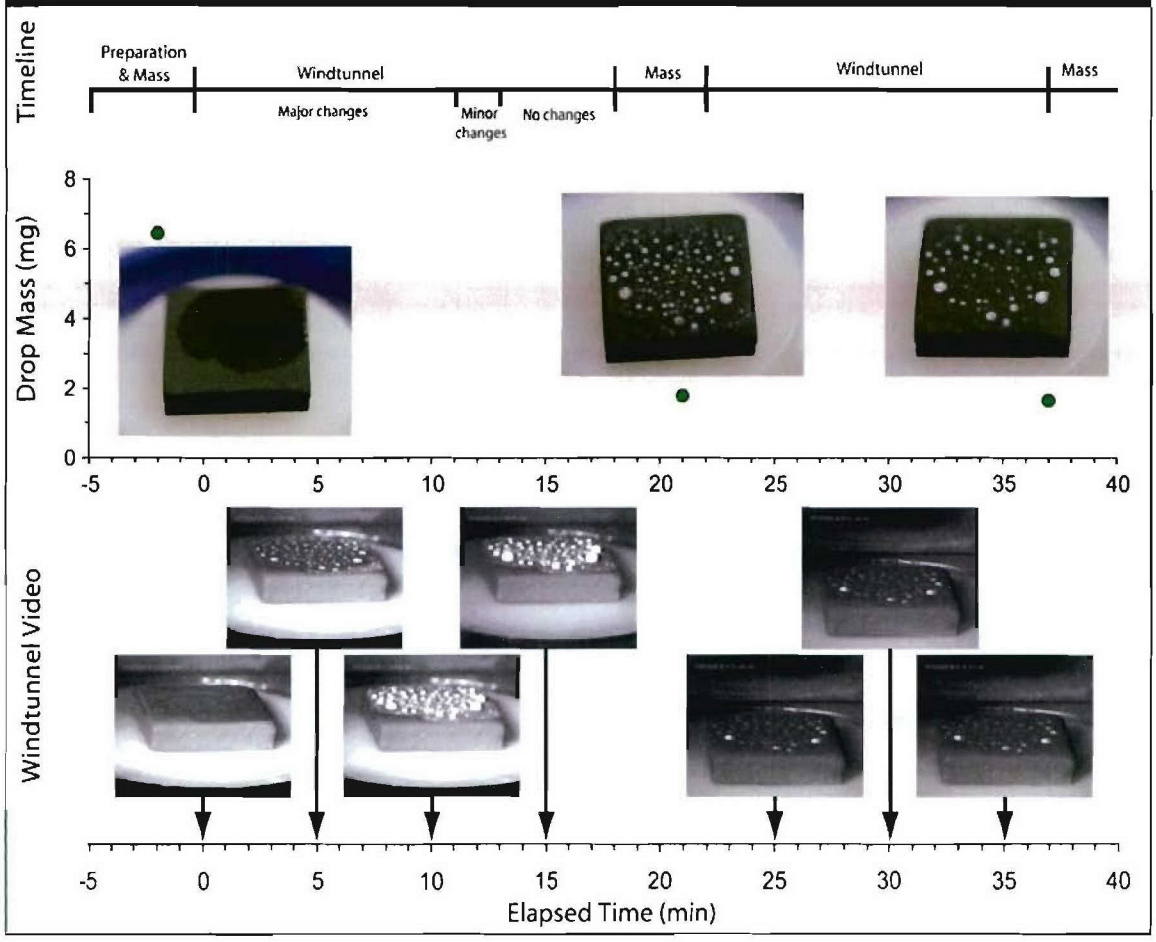
Results comparing evaporation of DeconGreen™ and DF200™ on CARC-coated aluminum are displayed in Figure 10. Green shading in the timeline and green data points in the drop mass refer to DeconGreen™. Red shading in the timeline and red data points in the drop mass refer to DF200™. The timeline and drop mass results are plotted along the same time axis. The ends of visual changes are noted for both decontamination solutions. Major and minor changes noted visually for the DF200™ on CARC-coated aluminum occur in a short period of time.

Table 4 documents the mass of the drop, the percentage of loss, and the change from the previous mass. As with the other tables, the time is listed as elapsed minutes from the first insertion into the wind tunnel.

Table 4 - DF200™ on CARC

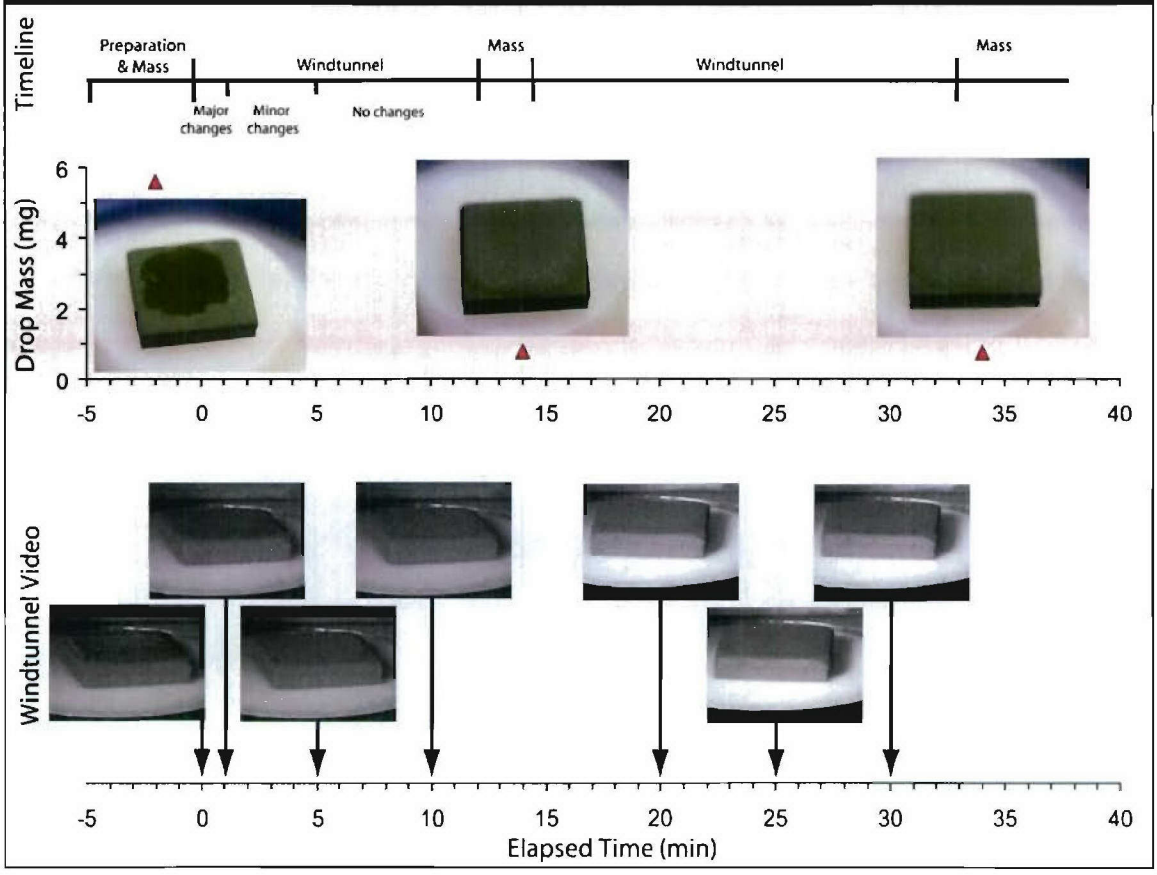
Elapsed Time from Wind Tunnel Insertion	Mass of Drop	Mass Loss	Change from Last Mass
min	mg	%	%
-2	5.6	-	-
14	0.8	86%	-
34	0.7	87%	5%

Figure 7. DeconGreen™ on CARC



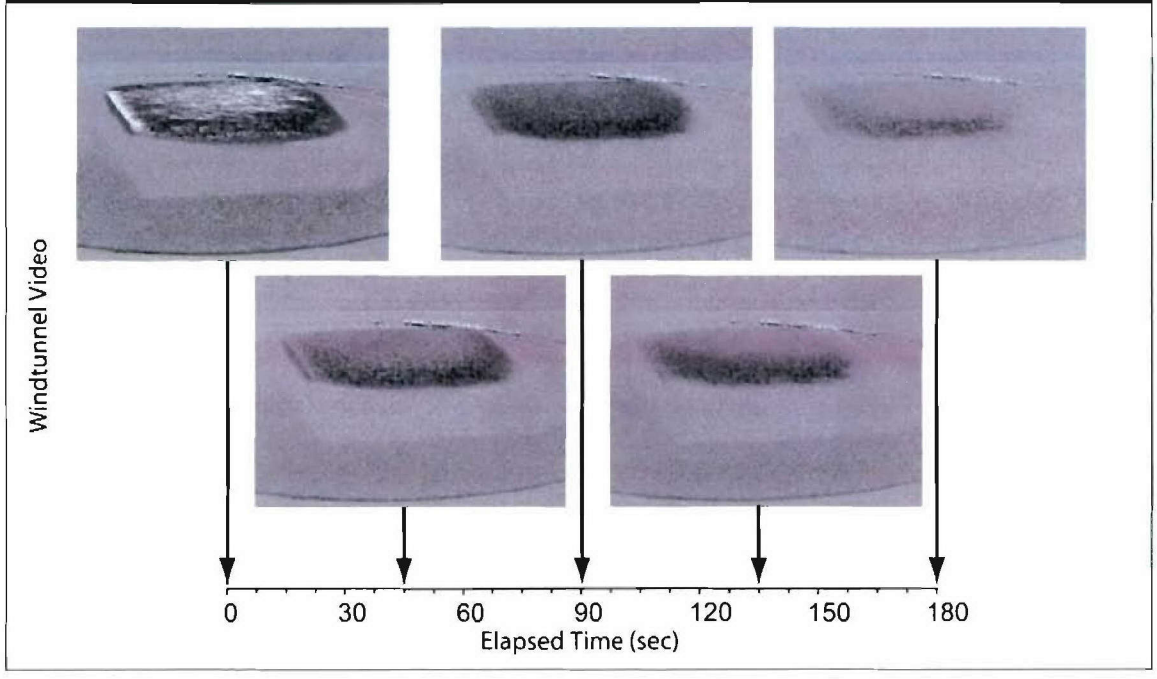
Note: Results for DeconGreen™ on CARC-coated aluminum. Timeline, drop mass, digital photographs, and wind tunnel video results all use the same time stamp. The evaporation of the liquid phase and emergence of structured foam clusters were noted as the major change in the timeline. Time zero is defined at the initial time point for sample placement in the wind tunnel.

Figure 8. DF200™ on CARC



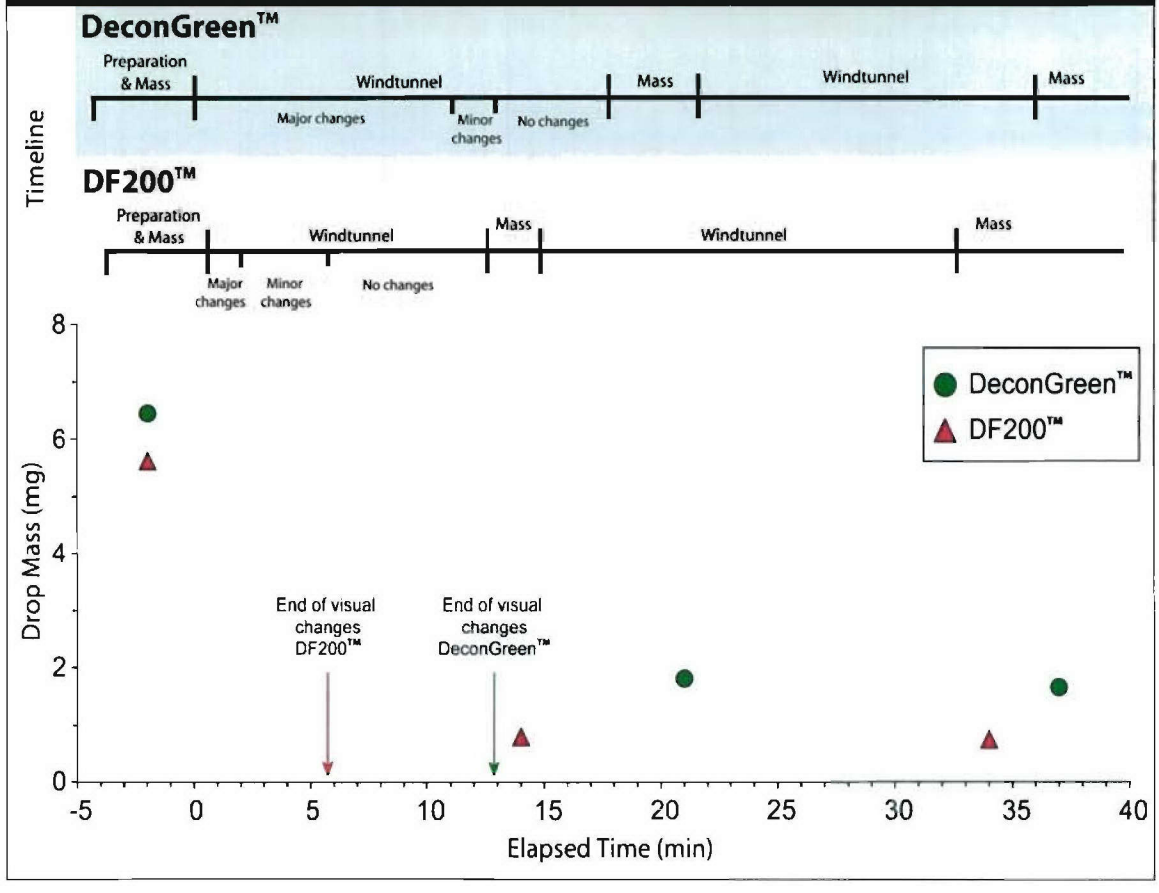
Note: Results for DF200™ on CARC-coated aluminum. Timeline, drop mass, digital photographs, and wind tunnel video results all use the same time stamp. The evaporation of the liquid phase was noted as the major change in the timeline. Time zero is defined at the initial time point for sample placement in the wind tunnel.

Figure 9. Enhanced Images of DF200™ on CARC



Note: Video stills of DF200™ on CARC-coated aluminum enhanced with image processing. The background has been subtracted to highlight the evaporation during the first 180 sec. Major and minor changes are highlighted.

Figure 10. CARC Compiled Results



Note: Results comparing evaporation of DeconGreen™ and DF200™ on CARC-coated aluminum. Green shading in the timeline and green data points in the drop mass refer to DeconGreen™. Red shading in the timeline and red data points in the drop mass refer to DF200™. The timeline and drop mass results are plotted along the same time axis. The ends of visual changes are noted for both decontamination solutions.

5. DISCUSSION

The goal of these experiments was to determine the evaporation rate for DeconGreen™ and DF200™ from operationally relevant surfaces with a given set of environmental conditions. The purpose of this work is to establish the persistence of the two decontamination solutions. These decontamination solutions contain mixtures of volatile and non-volatile components. An assumption is made that effective decontamination requires the decontaminant to remain on the surface and in a liquid state. Upon evaporation of the volatile compounds and the drying of the solution, the decontaminant is considered exhausted.

The evaporation process was measured with several independent techniques. Samples were contained within the controlled environment of the wind tunnel. The samples were monitored with video analysis to document changes to decontamination solution and periodically weighed to determine mass loss. The environmental conditions chosen represent an arid or desert climate with low humidity and a temperature in the range of 32 - 49 °C.[1]

The results displayed in Figure 6 show that DF200™ evaporates faster than an equal volume of DeconGreen™ by a factor of 2.8 times from bare aluminum. The results displayed in Figure 9 show that DF200™ evaporates faster than an equal volume of DeconGreen™ by a factor of 2.9 times from CARC-coated aluminum. These rates are determined by noting visual changes. The rapid evaporation of DF200™ has been noted previously.[2]

The components of DeconGreen™ formed a skeletal foam structure within the wind tunnel conditions. As described in Section 4, liquid drops were noted for both DeconGreen™ samples several hours after the completion of the experiment. These liquid drops are attributed to the collapse and deliquesce of the foam structures due to the ambient room humidity. This assertion is supported by a steady rising mass noted during the weighing periods, which is described discussed below. The rising mass is attributed to adsorption of water vapor during the foam collapse.

The adsorption of water vapor posed a challenge to obtaining an accurate mass for each sample during weighing periods. The balance is designed measure microgram quantities. However, it was not possible to distinguish between normal settling of the mass and an increase of the mass by water sorption. Therefore, the recorded mass was rounded to the nearest 0.1 mg. Water adsorption also affected the time chosen to declare the sample exhausted. The experimental test plan originally called for periodic weighing until the mass stayed within 10%. Due to the water sorption and the small quantity of residuals remaining on the surface, consecutive masses within 0.5 mg established the end of the experiment.

The deliquescing of the foam structures suggest that DeconGreen™ may evaporate more slowly in a humid environment. Future studies would be needed to test this hypothesis. Foam structures were not observed for the DF200™ samples during

evaporation; however, the presence of crystalline materials was documented. A rising mass was also noted for the DF200™ samples during weighing periods. However, a liquid phase was not observed post evaporation. This observation suggests the crystalline material to be hygroscopic; however, the structures remained solid and did not collapse as the foam.

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ACRONYMS

APG	Aberdeen Proving Ground
CARC	Chemical Agent Resistive Coating
DF200™	Decontamination Foam 200
ECBC	Edgewood Chemical Biological Center
IAW	in accordance with
m/s	meters per second
mg	milligram
min	minutes
µL	microliter

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