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Evaluation of Intelligent Optical Systems (IOS) Colorimetric-Based Organophosphate Vapor Sensor: Part II

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Testing was performed to evaluate the capabilites of an Intelligent Optical Systems (IOS; Torrance, CA) colorimetric-based organophosphate vapor sensor. Sensors were evaluated against varying concentrations (in milligrams per cubic meter) of test agents in humid environments with continuous airflow over the sensors. Repeatability of the sensors and the multi-analyte optical sensor array (MOSA) devices (MOSA Generation 1 boards) was also assessed.

15. SUBJECT TERMS

Intelligent Optical Systems (IOS)	Sensors	Agent detection
Dot sensor	Colorimetric	Vapor generation
Multi-analyte optical sensor array (MOSA)	Generation 1 (GEN1)	Saturator cell

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PREFACE

The work described in this report was authorized under U.S. Army Combat Capabilities Development Command Chemical Biological Center (DEVCOM CBC; Aberdeen Proving Ground, MD) Cooperative Research and Development Agreement IOS 1935C. The work was started in August 2020 and completed in April 2021.

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

This report has been approved for public release.

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EVALUATION OF INTELLIGENT OPTICAL SYSTEMS (IOS) COLORIMETRIC-BASED ORGANOPHOSPHATE VAPOR SENSOR: PART II

1. INTRODUCTION

The Chemical Analysis and Physical Properties (CAPP) Branch of the Research and Technology Directorate at the U.S. Army Combat Capability Development Command Chemical Biological Center (DEVCOM CBC; Aberdeen Proving Ground [APG], MD) was tasked with fulfilling Cooperative Research and Development Agreement IOS 1935C between DEVCOM CBC and Intelligent Optical Systems (IOS; Torrance, CA). Specifically, this work pertains to the sections of Amendment 3 concerning Phase II: Vapor Sensor Array testing. The purpose of this test was to assess the multi-analyte optical sensor array (MOSA) Generation 1 (Gen1) prototypes constructed by IOS to determine the limits and capabilities of the systems with respect to the needs of the chemical, biological, radiological, nuclear, and explosives (CBRNE) Sensor Integration onto Robotic Platforms (CSIRP) program. The intent of the CSIRP program is to integrate small-scale CBRNE sensors onto or into robotics platforms with limited loss in performance. For this particular effort, the goal was to assess the MOSA GEN1 chemical detector while it was mounted to an unmanned aerial system (UAS), determine a baseline capability, and then perform a limited assessment of that capability. Our guidelines for assessment reflected these conditions.

In accordance with the proposed test matrix agreed upon by DEVCOM CBC and IOS, differing analyte concentrations of sarin (GB), soman (GD), phosgene (CG), mustard (HD), and *O*-ethyl-*S*-(2-diisopropylaminoethyl) methyl phosphonothiolate (VX) vapors were evaluated to establish system sensitivity as a function of analyte vapor concentration in a carrier gas matrix at 50% relative humidity (RH).[1–3]

The CAPP branch provided the personnel, labor, facilities, supplies, and equipment to conduct the data collection. CAPP personnel were responsible for preparing the appropriate test conditions and recording laboratory data and results. The Agent Chemistry Branch of the Chemical Sciences Division provided the analyte materials. Both branches performed quality assurance and control (QA/QC) testing and maintained QA/QC records. The testing efforts were conducted at the Advanced Chemistry Laboratory within the DEVCOM CBC R&T Directorate at the Edgewood Area of APG. Testing commenced on 27 August 2020 and was completed on 28 April 2021.

2. SYSTEM DESCRIPTIONS

2.1 Vapor Sensors

The vapor sensors were 4 mm diameter polymer dots. The dots consisted of different formulations, all designed to change color upon exposure to agents. GB dots (batch G115-20201111-2 [G115-2], specifically) are transparent and colorless but turn transparent purple upon exposure to GB. The GD version 1 dots are opaque white (results are not included

here, as agent was detectable at ~34 mg/m^3), and version 2–4 dots are opaque; all are designed to turn purple when GD is present. CG dots are yellow and turn a burnt-orange-salmon color upon exposure to CG. HD dots are yellow and turn green upon HD exposure. VX dots are opaque yellow and turn burnt orange when VX is present. All formulations are designed to change color upon exposure to a specific agent vapor in environments that are at least somewhat humid, as they require some water to react. The dots are meant to be passive sensors, and the color change is intended to be visible to the naked eye. The color changing of the dots is described starting in Section 3.1.

2.2 MOSA GEN1 Boards

Although the dot is meant to be a purely subjective sensor, IOS designed a system to quantify the dot color change that occurs upon exposure. The system is composed of two light-emitting diodes (LEDs) and three photodiodes mounted on a MOSA GEN1 board (Figures 1–3).

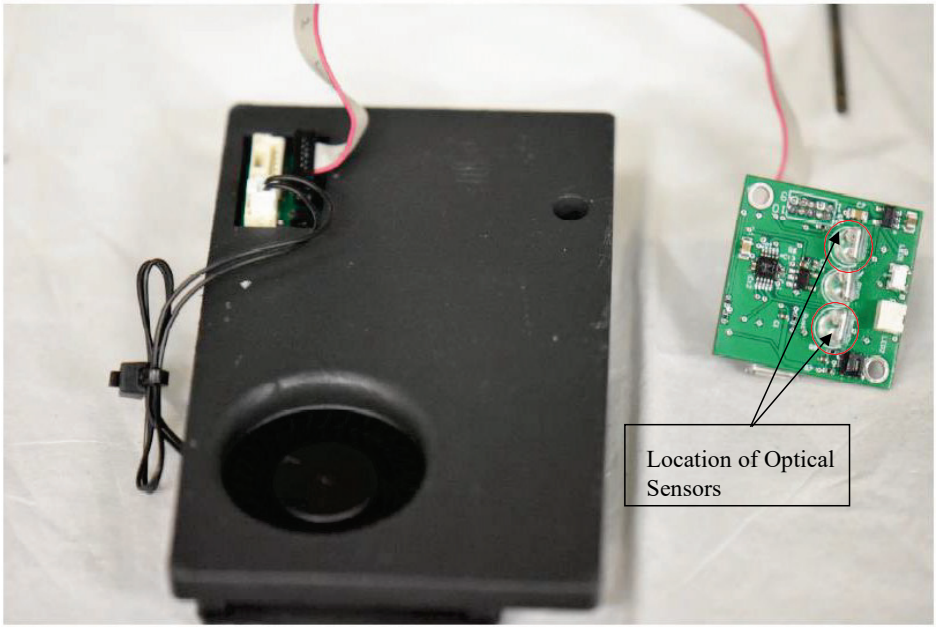

Figure 1. Front of MOSA GEN1 system, where chemical intake occurs; fan is shown. At right: optical sensor circuit board. Color-changing dots are placed on optical sensors.

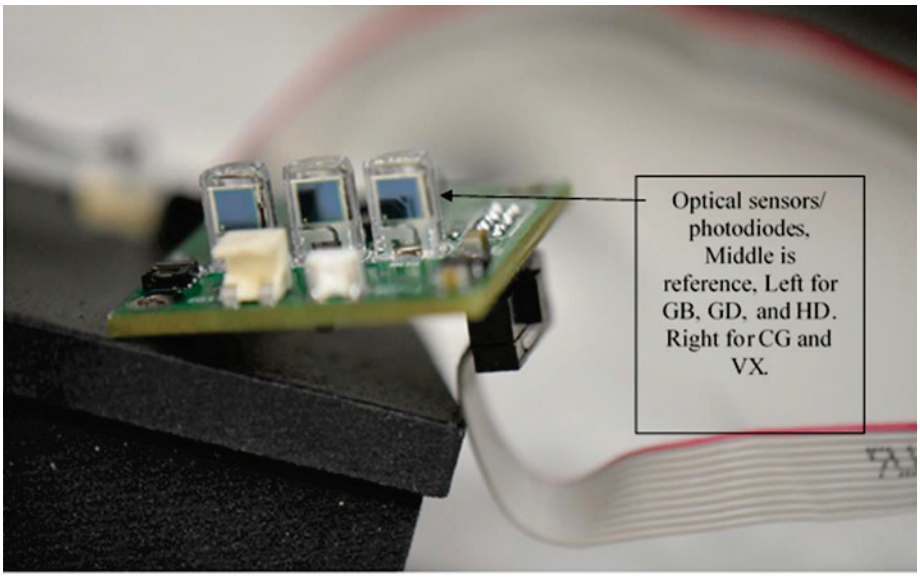

Figure 2. Close-up view of MOSA GEN1 optical sensor circuit board. Color-changing dots are placed on optical sensor head, as indicated by arrow.

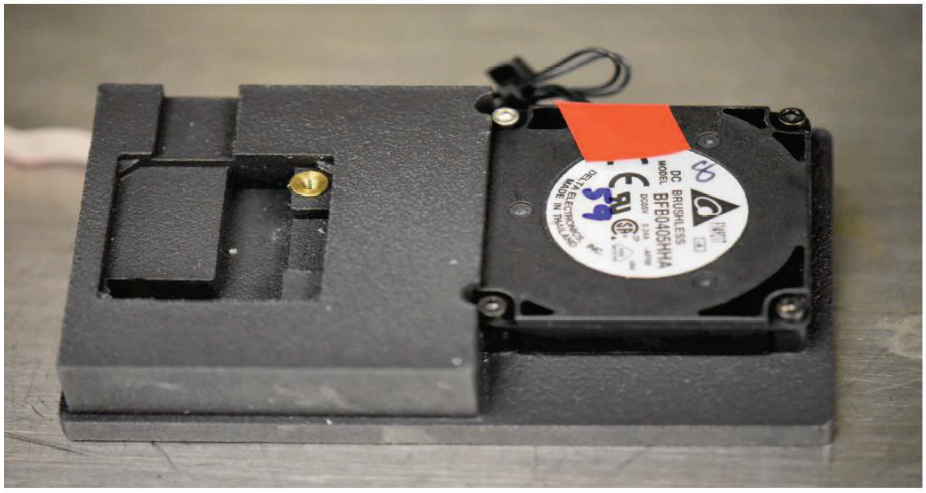

Figure 3. Back of MOSA GEN1 blower fan housing. Fan (right) and slot where optical sensor is installed once dot is attached (left) are shown.

The MOSA GEN1 system is connected to a laptop computer by a universal serial bus (USB) cable. The system is controlled by a graphical user interface (GUI), and the data were used to plot response curves. The GUI opens via a browser window, which allows the user to start and stop the data collection.

As shown in Figure 4, the optical sensor circuit board was installed on the direct-flow pump housing and connected to a laptop computer by an orange USB cable at the bottom right of the device. The direct-flow pump system uses forced air, whereas the MOSA GEN1 system uses fan-drawn air (Figure 1).

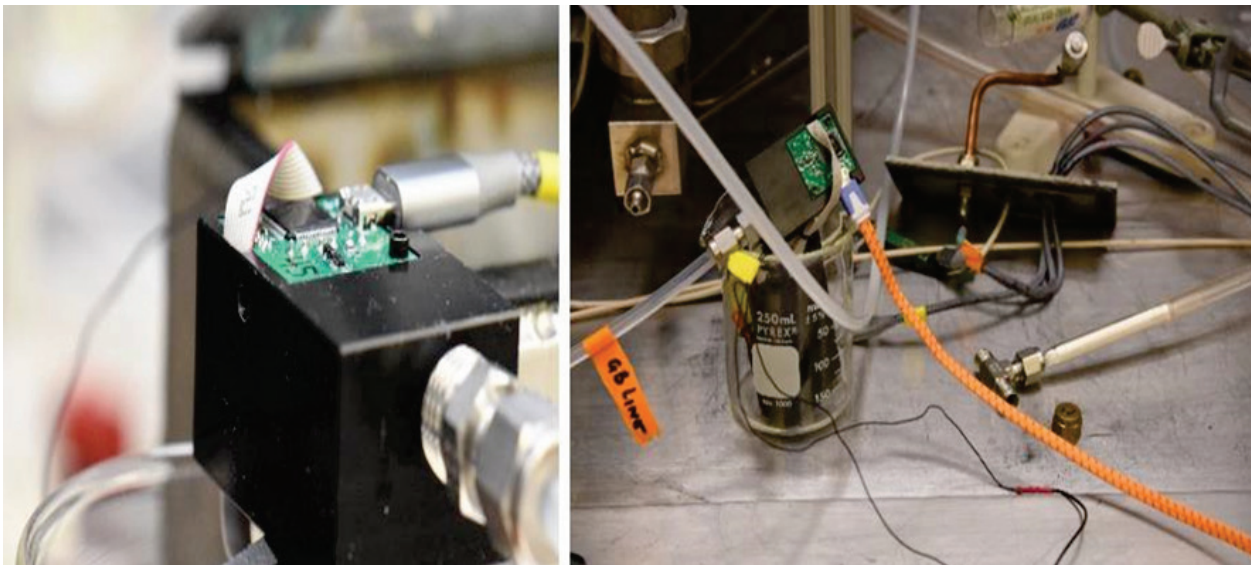

Figure 4. Direct-flow pump, version 4.1 housing (left). This system was designed to fit and convey analyte directly onto the optical sensor circuit board. The optical sensor circuit board is installed on the pump housing and connected to the laptop computer via an orange USB cable (right).

IOS provided predetermined parameter values that were optimized for each dot. All tests were conducted using those provided values. When a user began collecting data, each LED began to flash, and each photodiode recorded the light intensity. To quantify agent detections, a dot was placed onto the left or right photodiode. As the system was exposed to agent, the dot was expected to change color and begin filtering out the light from the LED. This caused the signal recorded in that photodiode to drop, while the signal measured on the uncovered middle photodiode remained constant as a control. The total signal drop in the left or right photodiode relative to the middle photodiode was recorded as the system response and also possibly incurred an alarm.

3. EXPERIMENTAL SECTION

3.1 Testing the Dots

The dots were tested before evaluations were performed on the MOSA GEN1 system. Three to six dots were mounted in a flow chamber, and a maximum of three dots were placed in each row. This was done to allow observation of the dot color-change responses in relation to the vapor source proximity. A 30 s time-lapse image was obtained to gauge mass transfer effects within the chamber. For agents with more than one dot formulation, randomized arrays were used, and the best performers were identified as those with the darkest color. Sometimes, certain liquid agents were deposited on the dots before or after vapor testing for verification of dot performance.

3.2 Test Matrix

To establish baselines for capability and vapor concentration, tests were conducted using a standalone MOSA GEN1 configuration under "ideal" conditions of room temperature and 50% RH. The initial exposure time for all analytes was 10 min. This exposure was preceded and followed by a 10 min (5 min for CG) clean airstream.

The analytes are listed in Tables 1 and 2. Challenge concentrations for GB, CG, HD, and VX were established as shown. A limit of detection (LOD) was reached, and challenges were repeated 10 times.

Table 1. Test Matrix: Blower Fan Housing

Chemical	RH (%)	Concentration			Concentration Test Range for 10 Trials
		1	2	3	
GB	50	0.250 mg/m^3	0.148 mg/m^3	0.061, 0.055 mg/m^3	0.147 ± 0.093 mg/m^3
GD	50	1.41 mg/m^3	2.47 mg/m^3	3.68 mg/m^3	4.17 ± 0.33 mg/m^3
CG*	50	1.42 ppm (5.75 mg/m^3)	0.071 ppm (0.29 mg/m^3)	na	0.0710 ppm (0.29 mg/m^3)
HD	50	0. 450, 0.622 mg/m^3	0.030, 0.130 mg/m^3	1.3; 0.8 mg/m^3	0.854 ± 0.926 mg/m^3
VX	50	0. 0050 mg/m^3	0.0148 mg/m^3	0.0280 mg/m^3	0.0308 ± 0.0078 mg/m^3

*CG was prepared and delivered from gas vendor (see Section 4.2.1) at a specific concentration (12.2 ppm in this case). Flow was adjusted to deliver specific CG concentrations into the test chamber. Only vapor exposures to dots in test chamber were performed at about 0.071 and 1.42 ppm.
na, not available or not applicable.

Table 2. Test Matrix: Enclosure – Direct-Flow Pump Housing

Chemical	RH (%)	Concentration (mg/m^3)			Concentration Test Range for 10 Trials (mg/m^3)
		1	2	3	
GB	50	0.210	0.133	0.054 0.044	0.12

3.3 Test Equipment and Setup

3.3.1 Analyte Vapor Generation

3.3.1.1 Analyte Vapor Generation: Analyte Stream

To generate the analyte vapor, at least 1000 µL of liquid neat analyte was added to a ceramic thimble glass saturator cell (Glassblowers.com; Turnersville, NJ). The main body of the saturator cell contained a porous ceramic thimble, which served to absorb the liquid agent and thereby increase the contact area between the liquid agent and the N_2 carrier gas. The multipass saturator cell was connected to a line that carried a dry N_2 carrier gas. Gas flow was controlled by a

Matheson Gas Products (Montgomeryville, PA) mass flow controller (MFC). The output from the saturator cell was diluted with a humid N_2 carrier gas stream to dilute the analyte stream to the desired concentration and provide sufficient humidity in the carrier stream before it reached the MOSA GEN1 system. Figure 5 shows the saturator cell.

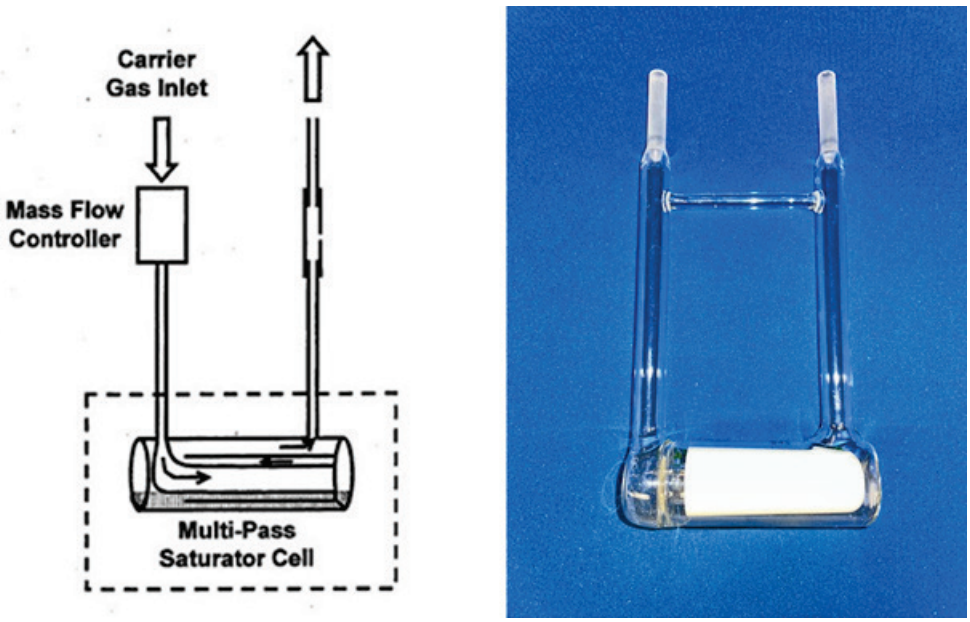

Figure 5. Diagram (left) and photograph (right) of a glass thimble saturator cell.

3.3.1.2 Analyte Vapor Generation: Dilution Flow

The functionality of using the diluted gas stream was twofold, as it allowed for (1) dilution of the analyte concentration from the saturator cell down to the concentration specified for each test; and (2) addition of humidity to the N_2 carrier gas stream. IOS specified that humidity is necessary for the color-change reaction of the dots to occur. Additionally, adding humidity at the dilution step protected the neat-agent liquid analyte from moisture exposure and subsequent hydrolysis, thereby maintaining the high purity of the vapor source.

The overall dilution gas flow was controlled by an Aalborg (Orangeburg, NY) MFC. To achieve the targeted 50% RH, the dilution stream was split into two separate streams, one wet and one dry. The wet humid stream was controlled by a Vertical Owlstone Vapor Generator (V-OVG) integrated with an Owlstone Humidity Generator (OHG-4; Owlstone; Westport, CT) after the stream had passed through an air bubbler stone in a 5 L sealed container approximately half-full with water. The continuous bubbling of the water generated a fully saturated (100% RH) headspace within the container. The dry and saturated flows were then recombined, and the humidity of the combined stream was monitored in real time via a temperature and humidity sensor (model EE03-FT9HC; E+E Elektronik; Engerwitzdorf, Austria). The relative flows between the wet and dry lines were adjustable using two needle valves, which allowed for fine control of the RH. Before it reached the test chamber, the concentrated analyte vapor from the saturator cell was mixed perpendicularly into the RH-controlled dilution flow.

A constant flow of humidified carrier air flowed into the sealed test chamber at a rate of 1 to 2 L/min. This flow rate ensured that during the 60 s exposure time, there was at least one complete air exchange within the test chamber; therefore, all three MOSA units received sufficient exposure to the target analyte. CVCAD Vapor 4.2.vi software (created by Zachary M. Smedley, DEVCOM CBC Respiratory Protection Branch; shown in Figure 6) was used to adjust both the analyte-laden and clean flows such that each was 1 to 2 L/min. As compared to the dilution stream, the flow from the saturator cell was so small that the change in total flow when it was added to the analyte stream had negligible effects on the overall test. Figure 6 shows all of the conditions that resulted in the final gas stream that entered the test chamber and the monitoring of temperature and RH throughout the challenge.

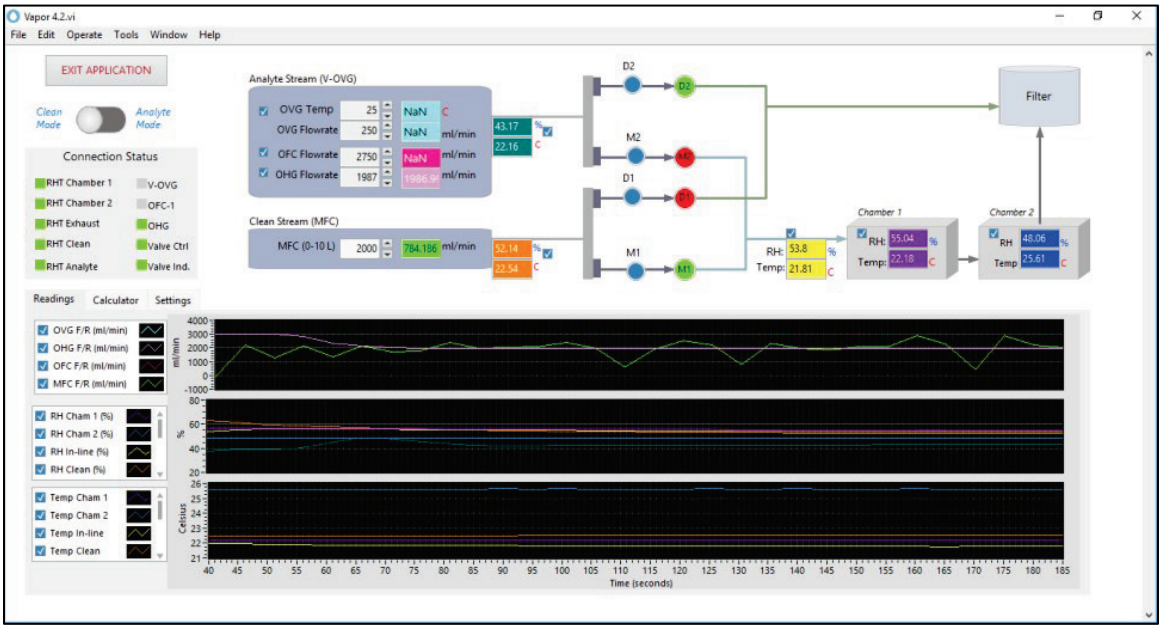

Figure 6. CVCAD Vapor 4.2.vi software.

3.3.2 Clean Flow (Clean Stream)

The dilution gas flow was controlled by an Aalborg MFC. To generate the 50% RH carrier gas specified for the test, the dilution stream was split into two separate streams: one that remained dry, and another that was directed into an air bubbler stone in a 5 L sealed container approximately half-full with water. The continuous bubbling of the water generated a fully saturated (100% RH) headspace within the container. The dry and saturated flows were then recombined, and the humidity of the combined stream was monitored in real time via an E+E Elektronik model EE03-FT9HC temperature and humidity sensor and the CVCAD Vapor 4.2.vi software. Two needle valves allowed for adjustment of the relative flows between the wet and dry lines, which enabled fine control of the RH. A constant flow of humidified carrier air (clean stream) was conveyed to the sealed chamber at a rate of 1 to 2 L/min. Unlike the process described in Section 3.3.1, no challenge analyte was present; therefore, the vapor stream was clean.

3.3.3 Equipment Setup

For safety purposes, all equipment and tubing carrying analyte vapor were kept inside a chemical fume hood with engineering controls. All analyte was contained within ¼ in. Teflon tubing joined with Swagelok connections. The humidified flow generation equipment was stationed outside the hood and piped to the testing rig; one set carried clean humidified N_2 gas (clean stream), and one set carried humidified dilute flow analyte vapor (analyte stream) into the testing chamber. The MOSA GEN1 system was placed in the testing chamber to begin the test phase. The specific streams were connected as needed. All flow rates were verified using a calibrated Defender 520 gas flowmeter (Mesa Labs; Butler, NJ) or a DigiFlow digital gas flowmeter (Netech Corporation; Farmingdale, NY). Sample flow rates were controlled with calibrated MFCs (Matheson Gas Products) and verified before and after sampling with calibrated Defender 520 or DigiFlow flowmeters connected in-line with the sample stream. Further down the pipeline, an adapter was emplaced for sampling the analyte stream after it was released into the test chamber. The sampling process allowed for verification of the challenge concentration. Gas chromatography with flame ionization detection (GC–FID) or flame photometric detection (GC–FPD) and a thermal desorption sampling system, located outside the hood, were used for monitoring the concentration of the generated vapor analyte stream during delivery to the test chamber. A flow chart shows the test process and equipment (Figure 7). For the research, GC–FPD was reported as the primary detection system. The exposure chamber (Figure 8) was 2 L in volume. It was constructed of stainless steel and included safety glass to allow for sample viewing during exposure.

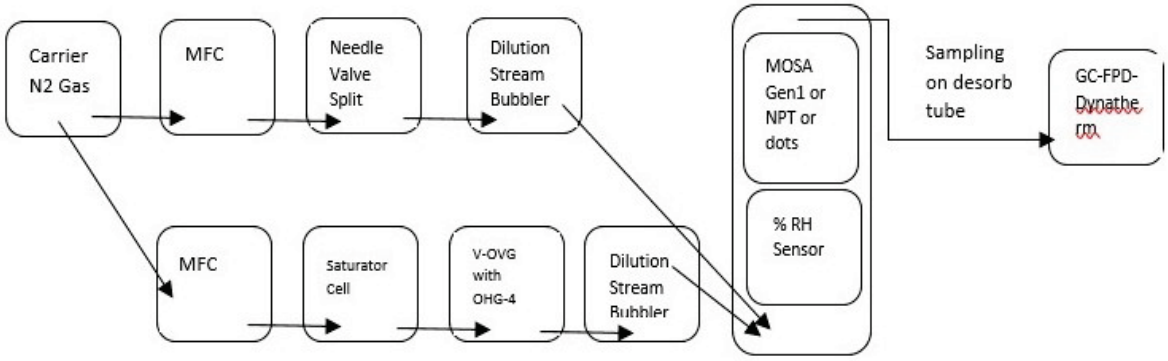

Figure 7. Testing apparatus schematic.

Figure 8. Exposure chamber (2 L volume), front and side views.

3.3.4 Analyte Vapor Sample Analysis

After sufficient time was allowed for the saturator cell to build up the analyte vapor concentration (~30 min), the analyte vapor stream was diluted and allowed to flow into the test chamber. A sample was collected from the test chamber on a sampling sorbent tube (10 mm o.d., 4 5/8 in. long, packed with 20:35 Tenax-TA sorbent) at a rate of 5–500 mL/min for 1–5 min using a vacuum source (SKC; Eighty Four, PA). The sorbent tube was placed in an ACEM model 900 Dynatherm system (CDS Analytical; Oxford, PA), which is a thermal desorption unit (TDU). A vacuum source continuously drew N_2 into the TDU, where the sorbent tube was dried by a clean N_2 purge gas, and the sample was conveyed toward the focusing trap (4.5 in. long, 1/8 in. stainless steel, 6.3 cm of Tenax TA). The sample was then heated and transferred to the focusing trap (which was also heated). The data acquisition analysis cycle was started by the transfer of the collected sample onto the networked 6890 GC column for GC–FPD. This allowed for verification of the concentration at the moment of sampling as well as during the sample (analyte) test and the clearance checks.

The TDU was operated using the following parameters: the desorption tube temperature was maintained at 60 °C; it was purged with N_2 for 1 min, then heated to 350 °C under an N_2 flow at a rate of 80 standard cubic centimeters per minute (sccm) for 3 min. The sample was then transferred to the focusing trap for 3 min, which was maintained at 60 °C. The trap was heated to 375 °C under a 20 sccm N_2 flow for 3 min (4 min for VX) to collect the sample as it was transferred to the GC column of the GC–FPD, to start the data acquisition. The calibration standards (all prepared in hexane) were spiked onto the sorbent tube to calibrate the TDU–GC–FPD system. A linear regression fit ($r^2 = 0.999$) of the standard data was used to calculate the concentration of each chamber sample test and to perform clearance checks.

The compound was separated from the sample tube using the GC–FPD analytical system. The sample was analyzed using a Restek Rtx-5 GC capillary column (no. 10269, crossbond diphenyl dimethyl polysiloxane; Restek; Bellefonte, PA), which is medium-polarity, 30 m × 0.32 mm i.d. × 1.5 µm film column. The FPD system was equipped with either a sulfur (for HD) or phosphorus (for GB and VX) selective filter on one side of the burner block, which allowed for detection of sulfur- or phosphorus-containing analytes. Parameters were as follows:

- GC parameters for analysis of analytes: the helium total carrier flow rate was kept constant at 14.7 mL/min.
- GC oven parameters for analysis of specific analytes:
  - GB and GD: total run time was 3 min. Oven temperature ramps were set as follows: initial temperature was 80 °C with a 0.5 min hold time, and Ramp 1 was at 35 °C/min to 150 °C with a 0.5 min hold time.
  - HD: total run time was 3.14 min. Oven temperature ramps were set as follows: initial temperature was 100 °C with a 0 min hold time, and Ramp 1 was at 35 °C/min to 210 °C with a 0 min hold time.
  - VX: total run time was 5.38 min. Oven temperature ramps were set as follows: initial temperature was 100 °C with a 0 min hold time, and Ramp 1 was at 40 °C/min to 275 °C with a 1 min hold time.
- FPD parameters for analysis of analytes: the FPD system was operated at 250 °C, and the flow rates for the combustion gases used to support the system were set as follows: 75 mL/min for hydrogen, 100 mL/min for air, and 15 mL/min for makeup gas.
- FID parameters for analysis of analytes: for GD, the FID system was operated at 250 °C, and the flow rates for the combustion gases used to support the system were set as follows: 44 mL/min for hydrogen, 400 mL/min for air, and 45 mL/min for makeup gas.

All N_2 gas streams supplied were of ultra-high purity. N_2 was obtained from boil-off of house liquid N_2, which was supplied by Praxair Welding Gas and Supply Store (Baltimore, MD) and retained in a bulk storage tank on the premises.

Dynatherm GC–FPD sampling and analysis were not used for CG analysis. The compound CG was prepared and delivered from Praxair Distribution, Inc. (Morrisville, PA) at a specific concentration. Flow and dilution were adjusted to deliver desired concentrations into the test chamber for CG challenges.

3.4 Test and Assessment Procedures

The investigation described in DEVCOM CBC-TR-1744 (part I of this work)[4] indicated that background contamination must be eliminated to fully realize MOSA detection potential. To achieve that objective, a cleaning and clearing protocol was developed and implemented.

3.4.1 Clearance of the Test Chamber before Use

The test chamber was cleared of any contamination by using the following steps, sometimes in a repeated combination:

1. blowing a high flow of N_2 into the chamber for 30–60 min;
2. heating the chamber with a heat gun set to 160–190 °C for 30–60 min; and
3. rinsing the chamber with isopropyl alcohol followed by air drying.

After the test chamber was cleaned, it was sampled to verify the absence of contamination. A 2 L/m vacuum was applied for 1 min; this was followed by GC–FPD analysis of the collected samples, which completed the clearing process. In the case of CG, clearance was determined using the IOS GUI. The presence of a clean airstream with no analyte results in the GUI's transmittance data verified that clearance of the chamber had occurred. The baseline profile showed either a nearly level or increasing upward slope.

3.4.2 Clearance of the MOSA GEN1 before Use

The MOSA GEN1 system was cleared of any analyte by baking it overnight in a 35 °C oven. After the MOSA GEN1 system was baked, it was placed in a closed 1 L jar for 15 min. Headspace was sampled by applying a 2 L/m vacuum for 1 min and then performing GC–FPD analysis. In the case of CG, clearance was determined if the clean-stream GUI transmittance data plot was level or showed increasing slope, as discussed in Section 3.4.1. After a positive indication of chamber cleanliness was obtained or the GUI profile data showed a return to near baseline, it was deemed appropriate to begin the MOSA GEN1 (dots) challenge.

3.4.3 Testing the Dots: Color Change (Liquid Deposits)

A fresh dot was placed on a surface in a horizontal position and exposed to liquid agent. This allowed for or instantly confirmed dot formulation functionality.

3.4.4 Testing the Dots: Color Change (Formulation Selection)

The following steps were performed to test functionality of the dots, to select the best-performing dots when necessary, and to select analyte concentrations for the MOSA GEN1 test challenge:

1. Fresh dots (3–6) were placed on a vertical surface in a horizontal position and then loaded into the test chamber (see Section 4.1.4).
2. The analyte stream was connected to the test chamber at a preselected concentration.
3. A 20–30 min time-lapse video (30 s image interval) was recorded and continued until a physical appearance (color change) was observed. Sometimes longer videos were required.
4. The concentration within the test chamber was verified at various times during the analyte flow by GC–FID or GC–FPD.
5. Steps 1–4 were repeated with increasing concentration increments, depending on physical appearance of the dots (color change).
6. The lowest measured concentration that produced a good color response was selected to perform the MOSA GEN1 test challenge.
7. Unused dots were vacuum-sealed at the end of the day and stored for later use.

3.4.5 Test Matrix (10-10-10 Test; For CG, 5-10-5 Test)

For most analytes (Tables 5–21), a sequential paradigm designated as a 10-10-10 test was followed: 10 min clean stream, 10 min analyte stream, and 10 min clean stream. For CG, the paradigm was a 5-10-5 test. Other sequential paradigms used are noted on the tables. The steps were as follows:

1. Batteries for the power supply box were replaced before each trial run. A USB power plug replaced the battery power supply box during GD testing.
2. A fresh dot was placed on the optical sensor located on the circuit board. Clean gas was flowed over the MOSA GEN1 system for 10 min (5 min for CG). Data recording was initiated via the system GUI at the start of the 10 min. In this configuration, just the clean stream was flowing to the MOSA GEN1 system, which was housed within the test chamber.
3. At the 10 min mark, the analyte stream was connected to the test chamber (replacing the clean stream). The MOSA GEN1 system was exposed to the analyte flow for 10 min. Concentration was verified by TDU–GC–FPD after 5 min of analyte flow.
4. After 10 min of exposure (at the 20 min mark overall, 15 min for CG), the clean stream was reconnected to the test chamber. The final clean stream then flowed over the MOSA GEN1 system for an additional 10 min (5 min for CG), for a total of 30 min (20 min for CG) per trial. Data recording ceased at the end of the 30 min.
5. The start time was recorded in the laboratory notebook, and each trial was matched to the time-stamped data file generated by the MOSA GEN1 system.

Figure 9 is an example of the response data curve generated by the IOS GUI software during an exposure for the system. After each trial, the dot was photographically documented along with an unexposed dot to visually clarify any color change.

6. Concentrations were reduced until an LOD was reached, and the MOSA GEN1 challenge was repeated 10 times.
7. Unused dots were vacuum-sealed at the end of the day and stored for later use.

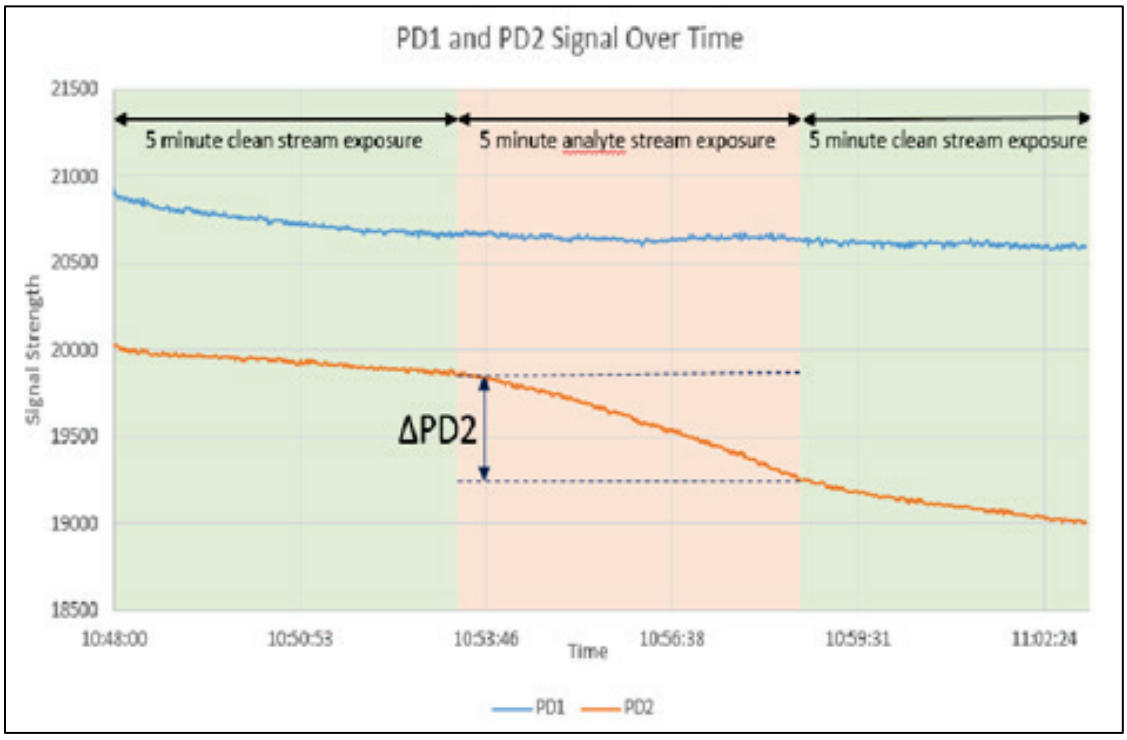

Figure 9. Example of response curve using data produced by the IOS GUI software for the MOSA GEN1 system.

The clearance protocol described in Sections 3.4.1 and 3.4.2 provided a significant improvement in the response baseline and a more accurate and repeatable method for measuring and evaluating dot performance during research and testing. The cleaning process is a critical aspect to performing dot assessments successfully.

4. RESULTS AND DISCUSSION

Provided in this section are direct vapor and liquid challenges for analytes GB, GD, HD, VX, and CG. All vapors with the exception of CG were generated using either the saturator cell approach or delta tube approach (refer to technical report DEVCOM CBC-TR-1744).[4] The analyte CG was purchased as a gas mixture and was used as received. As described in Section 3.3.1, all analyte concentrations were reduced when appropriate by N_2 gas dilution to obtain the desired concentrations.

Using the process presented in Section 3.4, dots were challenged for compounds GB, GD, HD, VX, and, CG. Some results include data for liquid challenges. The majority of the experiments were for vapor challenges. For the initial data collection challenges, dots were horizontally mounted on paper and then placed in the bottom of the chamber. All other challenges were performed by mounting dots vertically on a steel frame and then placing the frame in the chamber.

4.1 GB

The first dot chemistry examined in the assessment and evaluation was the GB dot.

4.1.1 Testing Three SUTs in Tandem (GUI Graph in Appendix A.1)

The test plan for this effort (Appendix B), specified that three SUTs were to be tested in tandem. The dots used were filter-paper based. Tables 3 and 4 summarize the test performed with all three SUTs placed in a sealed 2 L test chamber. For each challenge, corresponding GUI data are located in Appendix A.1.

Table 3. Summary (1 of 2) of Tests Performed for Threat Agent GB with All Three SUTs Placed in a Sealed 2 L Test Chamber

GUI Graph No.	Date	Time	Clean/ Agent/ Clean (min)	Housing	Dot Used (SUT 43/44/45)	Dot Open Date	Concentration (mg/m^3)	Alarm (SUT 43/44/45)	Comment
A.1.1	27-Aug-20	9:32	5/25/5	na	2/3/6	na	44.7	y/y/y	Boards only, no housings
A.1.2	27-Aug-20	10:51	5/15/5	b	2/3/6	na	33	y/y/y	
A.1.3	27-Aug-20	11:48	5/20/5	b	2/3/6	na	36.9	y/y/y	
A.1.4	27-Aug-20	12:36	5/20/5	b	2/3/6	na	43.8	y/y/y	
A.1.5	31-Aug-20	9:58	5/20/5	na	5/7/8	na	17.4	y/y/y	Boards only, no housings
A.1.6	31-Aug-20	10:40	5/20/5	b	5/7/8	na	14.8	y/y/y	
A.1.7	31-Aug-20	11:25	5/20/5	b	5/7/8	na	15.5	y/y/y	
A.1.8	31-Aug-20	12:07	5/20/5	b	5/7/8	na	14.6	y/y/y	Dot 5 fell off
A.1.9	1-Sep-20	10:08	5/20/5	b	1/4/5	na	11.7	y/y/y	
A.1.10	1-Sep-20	11:52	5/20/5	b	1/4/7	na	14.4	y/y/y	
A.1.11	2-Sep-20	10:08	5/20/5	b	4/8/1	na	15.6	y/y/y	
A.1.12	3-Sep-20	4:57	5/20/5	b	2/3/6	na	13.2	y/y/y	
A.1.13	3-Sep-20	5:40	7/20/5	b	2/3/6	na	10.9	y/y/y	
A.1.14	3-Sep-20	6:26	5/20/5	b	2/3/6	na	15.7	y/y/y	
A.1.15	3-Sep-20	9:46	5/20/5	b	70/71/70	na	25.5	y/y/y	RH may have been high, around 68%
A.1.16	3-Sep-20	10:37	5/20/5	b	70/71/70	na	13.3	y/y/y	
A.1.17	3-Sep-20	11:21	5/20/5	b	71/70/71	na	14.4	n/na/y	Dot 70 on SUT44 fell off
A.1.18	3-Sep-20	12:06	5/20/5	b	70/71/71	na	11.7	y/y/y	SUT43 batteries appeared to be dying; SUT43 dot also had a small dark spot on it, unsure if it was a hole poked with the tweezers or something else

Note: GUI graphs are located in Appendix A.1.
b, micro blower housing (requires a testing chamber); n, no; y, yes; na, not available or not applicable; p, pump.
Red type, not the typical 5-20-5 test.

Table 4. Summary (2 of 2) of Tests Performed for Threat Agent GB with All Three SUTs Placed in a Sealed 2 L Test Chamber

GUI Graph No.	Date	Time	Clean/ Agent/ Clean (min)	Housing	Dot Used (SUT 43/44/45)	Dot Open Date	Concentration (mg/m^3)	Alarm (SUT 43/44/45)	Comment
A.1.19	8-Sep-20	10:38	5/20/5	b	2/3/6	na	3.19	y/y/y	One SUT had a blip in the data; batteries may have been dying on SUT43 because system was alarming to CG; noticed a small dark dot on dot 2 on SUT43 after the fact
A.1.20	8-Sep-20	11:24	5/20/5	b	2/3/6	na	4	y/y/y	Changed batteries before this run
A.1.21	8-Sep-20	13:15	5/20/5	b	2/3/na	na	4.7	y/n/na	Ran out of dot 6; SUT44 software GUI was accidentally closed mid-run; GUI and data collection were restarted
A.1.22	8-Sep-20	13:56	5/20/5	b	2/2/2	na	1.98	n/y/y	Ran out of first batch of dot 2, switched to new batch of dot 2; no change in signal seen
A.1.23	8-Sep-20	14:40	5/20/5	b	2/2/2	na	5.15	n/y/y	New batch of dot 2; no change in signal; raised concentration; SUT44 had spikes in signal
A.1.24	9-Sep-20	9:16	5/20/5	b	2/2/2	na	5.28	y/y/y	All three alarmed
A.1.25	9-Sep-20	10:32	5/20/5	b	2/2/2	na	2.8	n/y/n	SUT44 was the only one that alarmed
A.1.26	9-Sep-20	13:15	5/20/5	b	2/2/2	na	2.81	y/y/y	SUT43 unstable baseline and alarming for phosgene as well; all three alarmed
A.1.27	10-Sep-20	10:05	5/20/5	b and p	na/2/2	10-Sep-20	3.3	na/y/y	Batch of dot 2 received 10 September; previous batch may have been affected by a “heat wave”; starting with 10 September batch today; SUT44 was pump, SUT5 was blower
A.1.28	10-Sep-20	11:04	5/20/5	b and p	na/2/2	10-Sep-20	7.05	na/y/y	Blower appeared to perform slightly better than pump

Notes: GUI graphs are located in Appendix A.1.
b, micro blower housing (requires a testing chamber); n, no; na, not available or not applicable; p, pump; y, yes.

4.1.2 GB: Testing Dots from Different Batches, Old (GSen115A LN: RG2000027) versus New (G115A-20200814.3) on 24 September 2020

This experiment was performed to address the inconsistencies that were observed from week to week among dots from the same batch (in some cases, significant differences in response were observed). Six G115 dots (polymer based) of old versus new preparation were randomly arranged and tested by mounting the dots on the sample holder (metal frame) then placing them in the test chamber. The dots were photographed before and after exposure to the test compound (Figure 10).

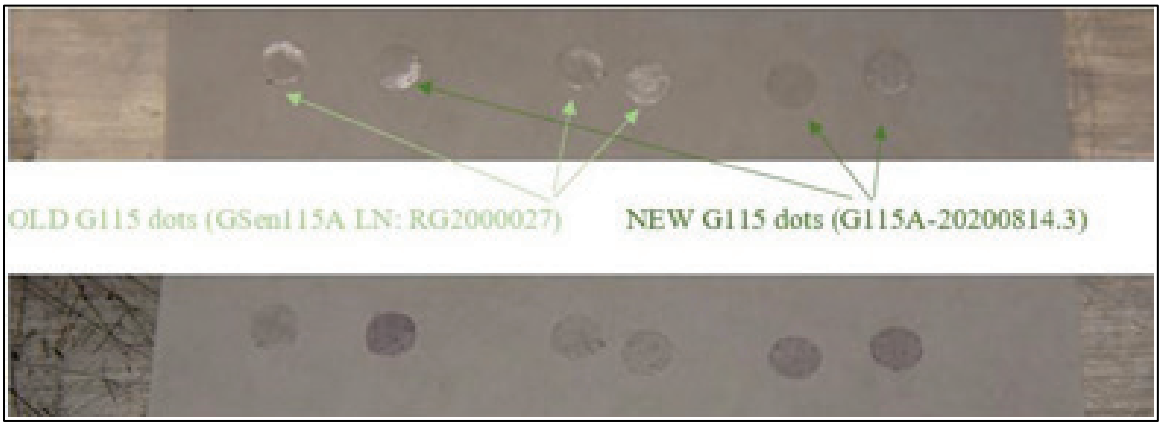

Figure 10. (Top) Three pairs of dots before and after GB exposure. Arrows indicate new versus old dots. (Bottom) Old and new G115 dots after exposure to 12.9 mg/m^3 of GB for 20 min.

For further clarification of dot performance and color intensity, dots were placed on a card then inserted into a 25 mL glass jar. GB (0.2 µL) was placed on the inside surface of the glass jar. The jar was capped tightly, and GB vapor concentration was allowed to equilibrate overnight. Figure 11 shows the resulting color change intensity for the old versus new dots. The dots were arranged side by side in three groups.

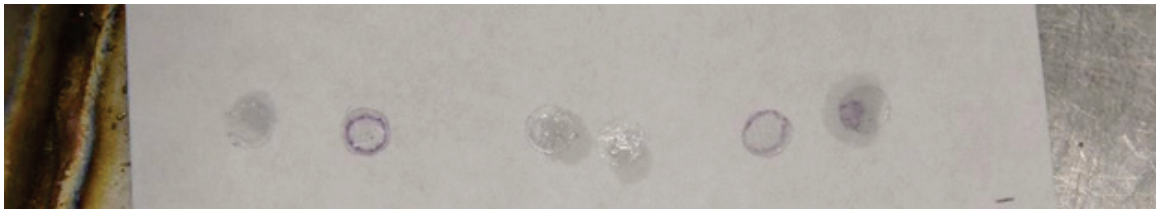

Figure 11. Exposure of 0.2 µL of neat GB to the same old and new G115 dots as shown in Figure 10. Dots were left in a sealed glass jar over the weekend.

Lessons learned from this experiment prompted changes to be made in the test challenge handling protocols. We recognized that (1) dots should be packaged in smaller batches (maybe three or six dots per package); (2) dots should be near room temperature; (3) during shipment, dots should be tracked using a monitoring device (such as a Thermax system [Thermax; Pune, India]); and (4) after use at the end of the day, dots should be vacuum-sealed for storage.

4.1.3 GB: Testing Dots from the Same Batch on 28 September 2020

It was hypothesized that the mass of analyte transferred to each dot as a result of airflow impacted dot performance. This was evaluated by performing a distance from source test (analyte inlet). Three dots were mounted and positioned in the chamber such that the time for the chemical to reach each dot was sequential. Data indicated that the dot nearest the inlet was darker than its adjacent dot (looking left to right). The experiment was repeated and is illustrated in Figure 12. The three G115 (G115A-20200814.3) dots shown in Figure 12 were tested by mounting the dots in the test chamber. A similar response was noted with very slight differences. The response seemed to be related to close proximity to the GB gas flow entering from the left side, as in the horizontally mounted experiment (Section 4.1.2).

In this experiment, the dots were mounted vertically. This mounting change did not alleviate the mass transfer effects within the chamber.

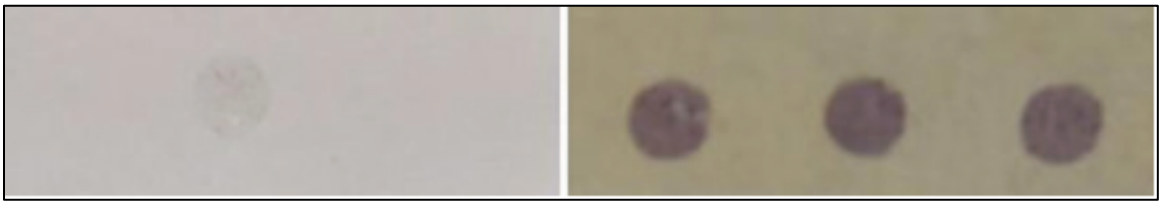

Figure 12. Before (left) and after (right) GB exposure (12.6 mg/m^3 for 20 min) of new G115 dots. In this case, the plate containing the dots was placed on a vertical wall.

Observations from this experiment indicated that mass flow can affect dot performance. As a result, a protocol change was implemented as a pretesting modification before data collection, whereby dots are randomized to determine performance before the statistical test challenge takes place. The result of no uniform dot color change provided evidence that location within the chamber prevented equal results with time. Going forward, the SUTs were tested individually for comparison rather than in groups of three.

4.1.4 GB: Testing Six Different Dots on 30 September 2020—Time-Lapse Data

For this protocol change, six different dots were provided for exposure in the chamber: the polymer-based G127, G125, G115-2, and G115-A1; and the paper-based dot 2 and dot 6. To ensure that the effects of chamber turbulence were known, this experiment was performed using three randomized arrays of those six dots and incorporated time-lapse video using 30 s intervals for images. This also served the purpose of identifying dot performance and, therefore, usefulness. This array approach was repeated in triplicate, as shown in Figures 13–15.

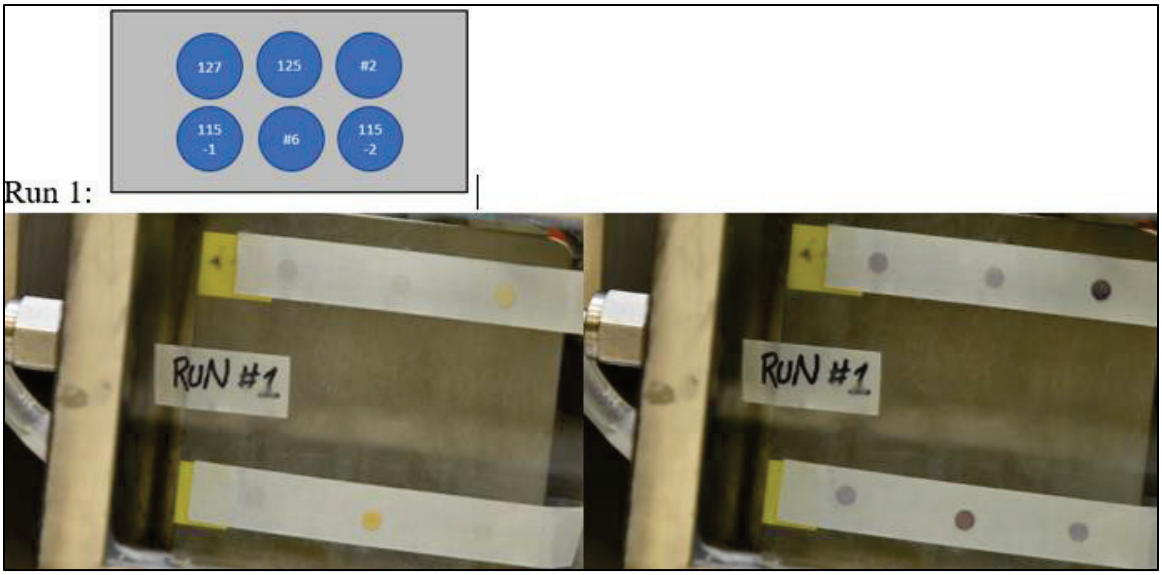

Figure 13. Run 1: (Top) First of three randomized arrays of six dots. (Bottom) Images of dots in chamber before (left) and after (right) GB exposure. GB exposure was 16.4 mg/m^3 for 20 min for six randomly placed dots.

Figure 14. Run 2: (Top) Second of three randomized arrays of six dots. (Bottom) Images of dots in chamber before (left) and after (right) GB exposure. GB exposure was 20.1 mg/m^3 for 20 min for six randomly placed dots.

Figure 15. Run 3: (Top) Third of three randomized arrays of six dots. (Bottom) Images of dots in chamber before (left) and after (right) GB exposure. GB exposure was 21.5 mg/m^3 for 20 min for six randomly placed dots.

This experimental series allowed for selection of the best-performing dot based on the darkest color change. Positive results were observed for all three runs. The most color change was observed in dots 2, 6, and G115-2. On run 3, dot G115-1 was darker also, but this was not consistent on runs 1 and 2. Dot G115-2 was most consistent in its color change; also, its color change occurred at a quicker rate as compared with dots 2 and 6. The protocol changes were adopted for all other initial compound versus "type of dot" evaluations. This also accounted for flow dynamics in the chamber.

4.1.5 Start Testing One SUT at a Time (GUI Graph in Appendix A.2)

The test plan for this effort[4] included the testing of only one SUT at a time. The results from tests performed with only one SUT placed in a sealed 2 L test chamber are summarized in Tables 5–8.

Table 5. Summary (1 of 4) of Tests Performed for Threat Agent GB with Only One SUT Placed in a Sealed 2 L Test Chamber

GUI Graph No.	Date	Time	Clean/Agent/ Clean (min)	Housing	SUT No., Dot Used	Dot Open Date	Concentration (mg/m^3)	SUT No., Alarm (yes/no)	Color Change (yes/no)	Comments
A.2.1	5-Oct-20	13:55	5/20/5	p-df	45, G115-2	30-Sep-20	7.54	45, y	y	GUI detected the moment it was started; housing was not exposed; it stayed red the whole 30 min run; housing was baked out at 110 °C for 1 h, cooled before using
A.2.1	5-Oct-20	14:50	5/20/5	p-df	45, G115-2	30-Sep-20	12.2	45, y	y	GUI detected the moment it was started; housing was not exposed; it stayed red the whole 30 min run
A.2.2	6-Oct-20	7:35	5/20/5	p-df	45, G115-2	30-Sep-20	0	45, y	y	5 min clean stream only
A.2.2	6-Oct-20	8:04	5/20/5	p-df	45, 2	30-Sep-20	0	45, y	n	5 min clean stream only
A.2.3	6-Oct-20	8:30	5/20/5	p-df	G	6-Oct-20	24.14	45, y	y	GUI detected the moment it was started
A.2.3	6-Oct-20	9:31	5/20/5	p-df	45, 2	6-Oct-20	13.7	45, n	y	GUI alarmed off and on the moment it was started
A.2.3	6-Oct-20	10:13	5/20/<5	p-df	45, 2	6-Oct-20	7.32	45, n	y	GUI alarmed off and on the moment it was started; testing was stopped prematurely
A.2.3	6-Oct-20	12:21	25/26/5	p-df	45, G115-2	6-Oct-20	15.32	45, y	y	GUI detected the moment it was started
A.2.4	7-Oct-20	8:07	16/20/10	p-df	45, G115-2	6-Oct-20	0.198	45, y	y	Dot G115-2 faded with time out of chamber
A.2.4	7-Oct-20	9:39	10/10/10	p-df	45, G115-2	7-Oct-20	0.199	45, y	y	
A.2.4	7-Oct-20	10:21	10/10/10	p-df	45, G115-2	7-Oct-20	0.2	45, y	na	

Notes: GUI graphs are located in Appendix A.2.
On 30 September 2020: Performed time-lapse exposure for six dots (G127, G125, G115-2, G115-A1, dot 2, dot 6); G115-2 determined to be best sensor.
Starting 30 September 2020: At end of day, vacuum-sealed all opened dots.
Starting on 7 October 2020: Used clean and analyte line separately.
b, blower (requires a testing chamber); na, not available or not applicable; p-df, direct-flow pump.
Red type, not the typical 5-20-5 test or 10-10-10 test.

Table 6. Summary (2 of 4) of Tests Performed for Threat Agent GB with Only One SUT Placed in a Sealed 2 L Test Chamber

GUI Graph No.	Date	Time	Clean/Agent/ Clean (min)	Housing	SUT No, Dot Used	Dot Open Date	Concentration (mg/m^3)	SUT No., Alarm (yes/no)	Color Change (yes/no)	Comments
A.2.5	8-Oct-20	7:39	10/10/10	b	45, G115-2	7-Oct-20	0.005	45, n	n	Normal transmittance response observed during initial clean, GB, and final clean line connect
A.2.5	8-Oct-20	8:20	10/10/10	b	45, G115-2	7-Oct-20	0.225	45, n	n	Normal transmittance response observed during initial clean, GB, and final clean line connect
A.2.5	8-Oct-20	9:10	10/10/10	b	45, G115-2	7-Oct-20	~1-2	45, n	y	Out of FPD high range; normal transmittance response observed during initial clean, GB, and final clean line connect
A.2.6	8-Oct-20	9:52	10/10/10	p-df	45, G115-2	30-Sep-20	0.011	45, n	n	Normal transmittance response observed
A.2.6	8-Oct-20	10:29	10/10/10	p-df	45, G115-2	30-Sep-20	0.015	45, y	y	Normal transmittance response observed
A.2.6	8-Oct-20	11:05	10/10/10	p-df	45, G115-2	30-Sep-20	0.012	45, y	y	Normal transmittance response observed
A.2.6	8-Oct-20	12:58	10/10/10	p-df	45, G115-2	8-Oct-20	0.010	45, y	y	Package contained a Thermax monitor; normal transmittance response observed
A.2.6	8-Oct-20	13:34	10/10/10	p-df	45, G115-2	8-Oct-20	0.010	45, y	y	Normal transmittance response observed
A.2.6	8-Oct-20	14:09	10/10/10	p-df	45, G115-2	8-Oct-20	0.0099	45, y	y	Normal transmittance response observed
A.2.7	13-Oct-20	12:43	10/10/10	b	45, G115-2	8-Oct-20	0.060	45, n	n	GUI did not alarm throughout the run; transmittance response was not observed
A.2.7	13-Oct-20	13:23	10/10/10	b	45, G115-2	13-Oct-20	0.053	45, n	n	GUI did not alarm throughout the run; transmittance response was not observed
A.2.7	13-Oct-20	14:08	10/10/10	b	45, G115-2	13-Oct-20	0.053	45, n	n	GUI did not alarm throughout the run; transmittance response was not observed
A.2.7	13-Oct-20	14:46	10/10/10	b	45, G115-2	13-Oct-20	0.085	45, n	n	GUI did not alarm throughout the run; transmittance response was not observed
A.2.7	13-Oct-20	15:24	10/10/10	b	45, G115-2	13-Oct-20	0.080	45, n	n	GUI did not alarm throughout the run; transmittance response was not observed

Notes: GUI graphs are located in Appendix A.2.
Starting 8 October 2020: Noted time transmittance response change observed.
b, blower (requires a testing chamber); na, not available or not applicable; p-df, direct-flow pump.

Table 7. Summary (3 of 4) of Tests Performed for Threat Agent GB with Only One SUT Placed in a Sealed 2 L Test Chamber

GUI Graph No.	Date	Time	Clean/Agent/ Clean (min)	Housing	SUT No., Dot Used	Dot Open Date	Concentration (mg/m^3)	SUT No., Alarm (yes/no)	Color Change	Comments
A.2.8	14-Oct-20	7:50	10/10/10	b	45, G115-2	13-Oct-20	0.060	45, y	y	Normal transmittance response observed (why seeing GB now compared to trials on 13-Oct-20)
A.2.8	14-Oct-20	8:30	10/10/10	b	45, G115-2	13-Oct-20	0.056	45, y	y	Normal transmittance response observed
A.2.8	14-Oct-20	9:06	10/10/10	b	45, G115-2	14-Oct-20	0.055	45, y	y	Normal transmittance response observed; slightly erratic transmittance reading during final clean sweep
A.2.8	14-Oct-20	10:10	10/19/10	b	45, G115-2	14-Oct-20	0.156	45, y	y	Normal transmittance response observed; very erratic transmittance reading during initial clean sweep and slightly erratic transmittance reading during final clean sweep; GUI alarmed off and on during initial clean stream
A.2.8	14-Oct-20	10:56	10/10/10	b	45, G115-2	14-Oct-20	0.196	45, y	y	Normal transmittance response observed; very slightly erratic transmittance reading during initial clean sweep; GUI alarmed off and on during initial clean stream
A.2.8	14-Oct-20	11:33	10/10/10	b	45, G115-2	14-Oct-20	0.200	45, y	y	Normal transmittance response observed; GUI alarmed off and on during initial clean stream and alarmed longer during final clean stream with each trial run nos. 57–59
A.2.9	14-Oct-20	12:08	49/na/na	b	na	na	na	na	na	Clean stream only; empty SUT45 was connected, placed in chamber with clean stream; data were collected for 49 min; chamber and SUT cleaned up with N_2 flow over time
A.2.10	14-Oct-20	13:07	10/10/10	b	43, G115-2	14-Oct-20	0.330	43, y	y	Normal transmittance response observed; very slightly erratic transmittance reading during initial clean sweep; GUI alarmed off and on during initial clean stream

Note: GUI graphs are located in Appendix A.2.
b, blower (requires a testing chamber); na, not available or not applicable; p-df, direct-flow pump.
Red type, not the typical 10-10-10 test.

Table 8. Summary (4 of 4) of Tests Performed for Threat Agent GB with Only One SUT Placed in a Sealed 2 L Test Chamber

GUI Graph No.	Date	Time	Clean/Agent/ Clean (min)	Housing	SUT No., Dot Used	Dot Open Date	Concentration (mg/m^3)	SUT No., Alarm (yes/no)	Color Change (yes/no)	Comment
A.2.11	14-Oct-20	13:44	10/10/10	b	G	14-Oct-20	0.0091	45, y	y	Normal transmittance response observed; battery pack changed; erratic transmittance could be coming from the high-concentration GUI still alarming two or three times during clean stream
A.2.11	14-Oct-20	14:21	10/13/10	b	45, 115-2	14-Oct-20	0.009	45, y	y	Normal transmittance response observed
A.2.11	14-Oct-20	15:00	10/10/10	b	45, G115-2	14-Oct-20	0.009	45, y	y	Normal transmittance response observed
A.2.12	14-Oct-20	12:08	445/na/na	b	45, G115-2	14-Oct-20	na	45, y	y	SUT45 with dot left in chamber with no stream; data was collected overnight; GUI alarmed sometime overnight and still alarming 15-Oct-20 morning
A.2.13	16-Nov-20	8:45	10/10/10	b	43, G115-20200924-4	4-Nov-20	0.237	43, n	n	Dots from these batch are no longer good
A.2.13	16-Nov-20	10:04	10/10/10	b	44, G115-20200924-4	16-Nov-20	0.488	44, n	n	Dots from these batch are no longer good
A.2.13	17-Nov-20	14:13	10/10/10	b	43, G115-20201111-2	17-Nov-20	0.030	43, y	y	Dots from these batch are no longer good, alarmed 96 s after analyte connect
A.2.13	18-Nov-20	7:57	10/10/10	b	44, G115-20201111-2	17-Nov-20	0.108	44, y	y	Normal transmittance response observed, alarmed off and on for 5 min after final clean stream connect

Notes: GUI graphs are located in Appendix A.2.
Starting on 14 October 2020: Need to run the GB saturator cell at the desired concentration for at least 30–60 min at start of day.
Starting on 19 October 2020: Chamber was verified below WPL before each trial; refer to Emge and Crouse off-gassing report (Appendix C). Perform cross-contamination test (headspace, GC oven bake-out and overnight); bake SUT overnight at 35 °C and high-flow N_2 in chamber for 30 min.
b, blower (requires a testing chamber); na, not available or not applicable; WPL workplace exposure level.
Red type, not the typical 10-10-10 test.

4.1.6 Lessons Learned from Performing the Tests Described in This Section

The lessons learned from this test series are summarized as follows:

1. Batteries for the MOSA GEN1 need to be changed before each trial run. Spikes will appear in the GUI plot if the batteries are not changed.
2. Dots should be packaged in smaller batches (maybe three or six dots per package).
3. Dots should be near room temperature and should be tracked using a monitoring device such as a Thermax monitor during shipment and with repeated use of the dot bags.
4. Dots should be vacuum-sealed after use (at the end of the day) for storage.
5. For testing dots, liquid and vapor exposures should be performed with time-lapse photography to "test the sensor response first to prove a positive response to the dot chemistry".
6. Contamination issues with the chamber, the MOSA housing, and the clean and analyte shared gas stream lines remain a concern because compounds stick to the walls, and this affects dot performance and the final results. Contamination of the clean and analyte shared stream lines was resolved by using separate clean and analyte lines.
7. Contamination issues with the chamber and housing were resolved, for example, by baking the SUT overnight at 35 °C and using high-flow N_2 in the chamber for 30 min until analysis showed levels were less than the workplace exposure level (<WPL).

4.1.7 GB Test Matrix: Concentrations of 0.044–0.250 mg/m^3

The MOSA GEN1 blower and fan housing (shown in Figures 1–3) versus the direct-flow pump version 4.1 housing (shown in Figure 4; the housing was verified to be below WPL by using the procedure in Section 3.4.2) were tested with GB concentrations ranging from 0.044 to 0.250 mg/m^3. Dots were placed on left optical sensors or photodiodes, and a 10-10-10 test was performed. After testing, dots were removed and photographed (Figure 16). Table 9 summarizes the test matrix as performed with only one SUT placed in a sealed 2 L test chamber to test GB concentrations of 0.044–0.250 mg/m^3.

Table 9. Summary of Test Matrix: Testing of 0.044–0.250 mg/m^3 GB Concentrations

No.	Date	Time	Clean/Agent/ Clean (min)	Housing	Dot Used	Dot Open Date	Concentration (mg/m^3)	SUT No., Alarm (yes/no)	Color Change (yes/no)	Comments
1	27-Oct-20	8:52	10/10/10	b	G115-2	27-Oct-20	0.249	45, y	y	Normal transmittance response observed
2	27-Oct-20	10:55	10/10/10	b	G115-2	27-Oct-20	0.23	44, y	y	Normal transmittance response observed
3	27-Oct-20	12:58	10/10/10	b	G115-2	27-Oct-20	0.25	43, y	y	Normal transmittance response observed; had issues with SUT43 in the past, works now
4	2-Nov-20	10:48	10/10/10	p - V4.1	G115-2	27-Oct-20	0.26	y	y	Normal transmittance response observed; very slight dot color change
5	2-Nov-20	12:53	10/10/10	p - V4.1	G115-2	27-Oct-20	0.226	y	y	Normal transmittance response observed; very slight dot color change
6	3-Nov-20	7:58	10/10/10	p - V4.1	G115-2	27-Oct-20	0.21	y	y	Slight dot color change; transmittance decrease observed 1 min after GB connect
7	4-Nov-20	11:10	10/10/10	b	G115-4	4-Nov-20	0.148	43, y	y	Normal transmittance response observed; dot color change; package came with Thermax sticker
8	4-Nov-20	12:29	10/10/10	p - V4.1	G115-4	4-Nov-20	0.133	y	y	Normal transmittance response observed
9	5-Nov-20	7:55	10/10/10	b	G115-4	4-Nov-20	0.061	43, y	y	Normal transmittance response observed
10	5-Nov-20	9:21	10/10/10	p - V4.1	G115-4	4-Nov-20	0.054	y	y	Normal transmittance response observed
11	5-Nov-20	11:33	10/10/10	b	G115-4	4-Nov-20	0.055	43, y	y	Normal transmittance response observed
12	10-Nov-20	7:16	10/10/10	p - V4.1	G115-4	4-Nov-20	0.044	y	y	Normal transmittance response observed; slight dot color change

Notes: GUI graphs are located in Appendix A.3.
Concentration test (chamber and housing cleared <WPL, and batteries for power supply were replaced before each trial).
b, blower (requires a testing chamber); p - V4.1, direct-flow pump version 4.1.

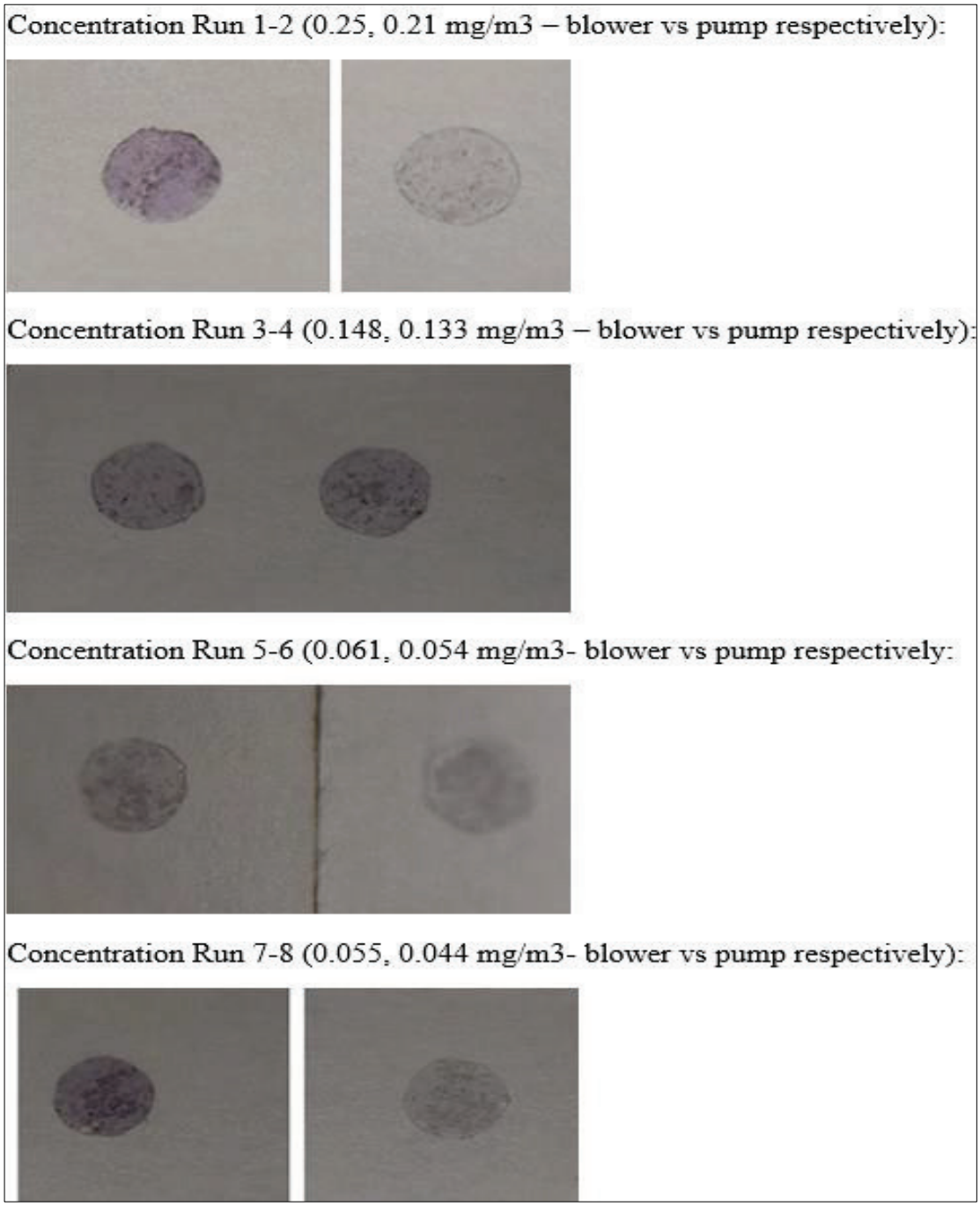

Figure 16. Photographs after exposure for concentration runs 1–8, blower (left side) versus pump (right side). Visual inspection of color profile shows the fan housing performed better than the intake approach for the eight trials tested.

In this series of vapor challenges, the MOSA GEN1 blower and fan housing was determined to be the better of the two housing configurations (as compared to the forced air housing). To conclude the fan housing investigation, the final configuration of the MOSA GEN1 reproducibility experiment was evaluated as the next task for this research effort.

At this point, the exposure low-level concentration was known, the best-performing dot was established, and the protocol for chamber cleaning and clearing time was established; therefore, 10 dot replicate testing could commence. This was critical for the MOSA GEN1 performance to achieve consistency confidence.

4.1.8 GB: 10 or More Trials Using Different MOSA GEN1 Systems at Concentration of 0.147 ± 0.093 mg/m^3

To prove reproducibility of the color changes, all dots were measured and photographically recorded after GB exposure. During the exposure (concentration range of 0.147 ± 0.093 mg/m^3) the GUI alarmed within 21 ± 21 s. For the 10-10-10 test (10 min of clean air, 10 min of GB, 10 min clean air), our GUI data indicated that the dot response was a function of time, except in SUT Trial 2, SUT56. This was later retested in Trial 11 to completion, plus 10 trials were recorded. At the conclusion of all trials performed during the challenge day, the dots tested were recovered from the diode and fixed to a piece of tape, and the trial series was photographed (Figure 17). The visual color change was consistent as documented. For GB, 13 trials were performed over a three-day period. The concentration for each trial was verified with GC–FPD and then reported. Table 10 summarizes the results of the GB trials using different MOSA GEN1 systems.

Table 10. Summary of GB Trials at 0.147 ± 0.093 mg/m^3

No.	Date	Time	Clean/Agent/ Clean (min)	Housing	Dot Used	Dot Open Date	Concentration (mg/m^3)	SUT No., Alarm (yes/no)	Color Change (yes/no)	Comments
1	17-Nov-20	14:40	10/10/10	b	G115-20201111-2	17-Nov-20	0.196	58, y	y	Normal transmittance response observed
2	18-Nov-20	13:06	10/10/10	b	G115-20201111-2	18-Nov-20	0.240	59, y	y	Normal transmittance response observed
3	18-Nov-20	14:39	10/10/10	b	G115-20201111-2	18-Nov-20	0.232	60, y	y	Normal transmittance response observed
4	19-Nov-20	8:04	10/10/10	b	G115-20201111-2	18-Nov-20	0.082	61, y	y	Normal transmittance response observed
5	19-Nov-20	9:28	10/10/10	b	G115-20201111-2	18-Nov-20	0.118	62, y	y	Normal transmittance response observed
6	19-Nov-20	10:52	10/10/10	b	G115-20201111-2	18-Nov-20	0.122	63, y	y	Normal transmittance response observed
7	19-Nov-20	12:36	10/10/10	b	G115-20201111-2	19-Nov-20	0.126	64, y	y	Normal transmittance response observed
8	19-Nov-20	14:08	10/10/10	b	G115-20201111-2	19-Nov-20	0.131	65, y	y	Normal transmittance response observed; new power supply used
9	20-Nov-20	7:36	10/10/10	b	G115-20201111-2	19-Nov-20	0.102	54, y	y	Normal transmittance response observed
10	20-Nov-20	8:54	10/10/10	b	G115-20201111-2	19-Nov-20	0.133	67, y	y	Normal transmittance response observed
11	20-Nov-20	10:36	10/10/10	b	G115-20201111-2	19-Nov-20	0.136	68, y	y	Normal transmittance response observed

Note: GUI graphs are located in Appendix A.4.
b, blower (requires a testing chamber); na, not available or not applicable.

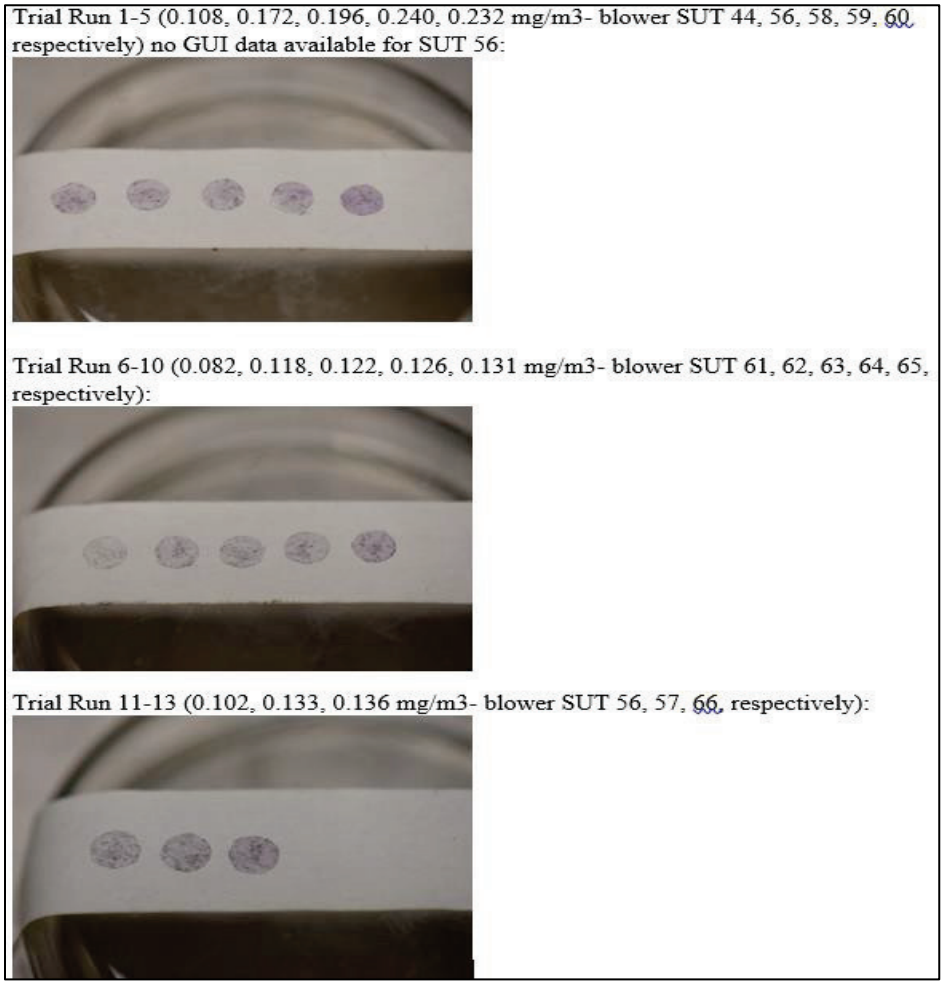

Figure 17. Photographs obtained after exposure for 13 trial runs using GB concentrations of 0.082–232 mg/m^3. Given these dots were photographed after testing, colors may or may not have decreased very slightly due to time passing.

4.2 CG

The next dot chemistry examined in the assessment and evaluation was the CG dot.

4.2.1 CG: Testing the Dots

CG was provided by Praxair Distribution at a concentration of 12.2 ppm (lot no. 300024238001). Sample flow was adjusted to deliver specific concentrations into the test chamber for CG. GUI profiles were not collected at various concentrations. Only vapor exposures to dots in the test chamber were performed at 0.071 and 1.42 ppm. A direct high flow of CG onto the CG dot (Figure 18) resulted in a color change. CG vapor in the chamber was measured (~1.42 and 0.071 ppm), and the resulting impact is shown in Figure 19. Time-lapse video obtained at 30 s intervals is shown in Figure 19 for the 0.071 ppm CG concentration.

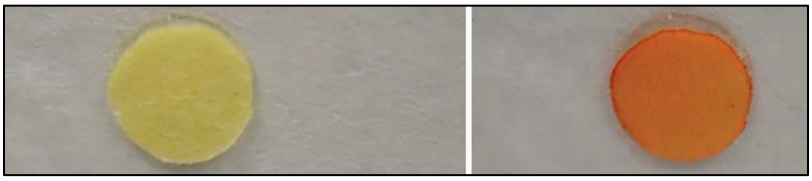

Figure 18. Photographs before (left) and after (right) direct high flow on CG dot.

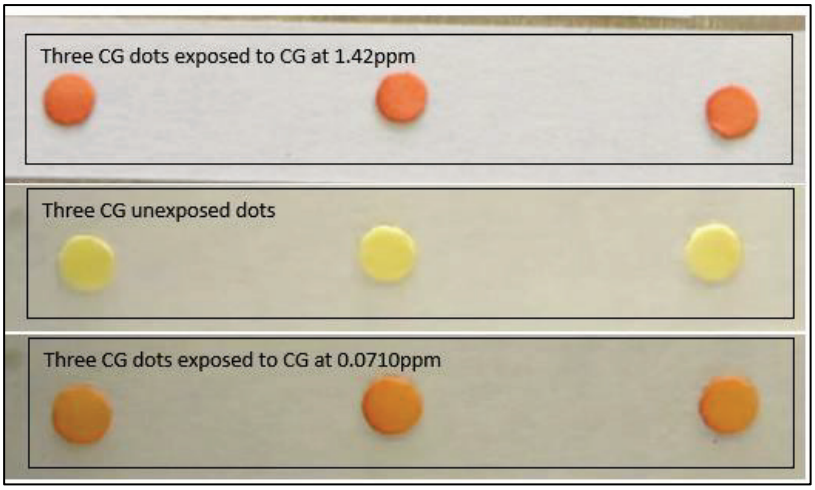

Figure 19. Photographs of CG dot unexposed (middle) and after 6 min (top, 1.42 ppm) and 20 min (bottom, 071 ppm) of CG exposure in chamber.

4.2.2 The TDU–GC–FPD Sampling and Analysis System Was Not Used for CG

The CG compound was prepared and delivered to the exposure chamber as received from the vendor at a specific concentration based on dilution from the primary gas cylinder. Over the course of the experiments, the dilution flow was adjusted as a fraction of the starting concentration to deliver specific CG concentrations into the test chamber.

4.2.3 CG: 10 Trials Using Different MOSA GEN1 Systems at 0.0710 ppm Concentration

The test chamber and housing were cleaned and cleared before use. Dots were placed on right optical sensors and photodiodes. A 5-10-5 test was performed, and GUI data were made available to the IOS after challenge completion. Initial and final clean N_2 streams were 0.3–2.8 min longer than 5 min for Trials 2 and 3. The CG dot colors changed in varying degrees, as shown in Figure 20. The color change was slight to very slight for Trials 4, 5, 8, 9, and 10. Of the 10 trials, 6 alarmed within 17–41 s and 4 alarmed within 3.6–8.5 min after the CG line was connected to the chamber. For Trial 10, the GUI went to green twice during CG line connection and then stayed green until 6 min, 50 s; it did not alarm again until 9 min, 40 s. Table 11 summarizes the 10 CG trials using different MOSA GEN1 systems. Dots performed well during the series of challenges.

Table 11. Summary of 10 CG Trials at 0.0710 ppm

No.	Date	Time	Clean/Agent/ Clean (min)	Dot Open Date	SUT No., Alarm (yes/no)	Color Change (yes/no)	Comments
1	9-Dec-20	10:54	5/10/5	9-Dec-20	56, y	y	CG dot color change
2	9-Dec-20	11:33	5/10/5	9-Dec-20	57, y	y	CG dot color change
3	9-Dec-20	12:50	5/10/5	9-Dec-20	58, y	y	CG dot color change
4	9-Dec-20	13:21	5/10/5	9-Dec-20	59, y	y	CG dot color change very slightly
5	9-Dec-20	13:47	5/10/5	9-Dec-20	60, y	y	CG dot color change slightly
6	9-Dec-20	14:12	5/10/5	9-Dec-20	61, y	y	CG dot color change
7	10-Dec-20	7:30	5/10/5	9-Dec-20	62, y	y	CG dot color change
8	10-Dec-20	8:06	5/10/5	9-Dec-20	64, y	y	CG dot color change very slightly
9	10-Dec-20	8:31	5/10/5	9-Dec-20	65, y	y	CG dot color change very slightly
10	10-Dec-20	8:56	5/10/5	9-Dec-20	66, y	y	CG dot color change slightly*

Note: GUI graphs are located in Appendix A.5.

*SUT63 blower fan housing was used with the SUT66 optoelectronic unit because the USB malfunctioned in the SUT63 optoelectronic unit.

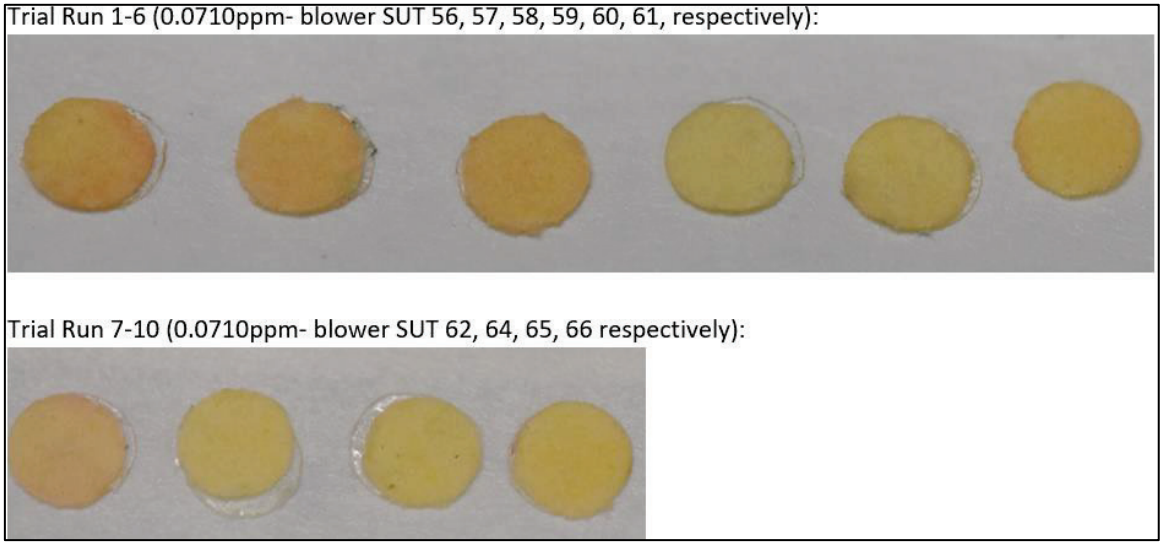

Figure 20. Photographs after exposure for 10 trial runs at 0.0710 ppm concentration. Given these dots were photographed after testing, colors may or may not have decreased very slightly due to time passing.

4.3 HD

The next dot chemistry examined for assessment and evaluation was the chemical agent HD dot.

4.3.1 HD Testing the Dots: Direct and Chamber HD Vapor

HD dots were provided for exposure to direct HD vapor and in-chamber HD vapor (flow entered from the left side), as shown in Figures 21 and 22, respectively. Time-lapse video using 30 s intervals was recorded for Figure 22.

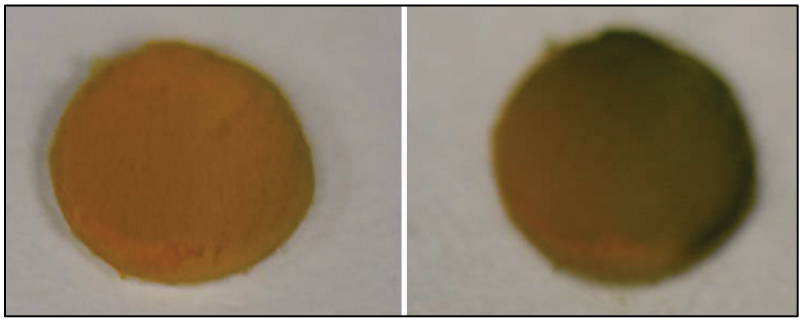

Figure 21. HD exposure results. Photographs before (left) and after (right) HD challenge on HD dot, using high-flow HD directly from the saturator exhaust port at 50 mL/min for 1 min.

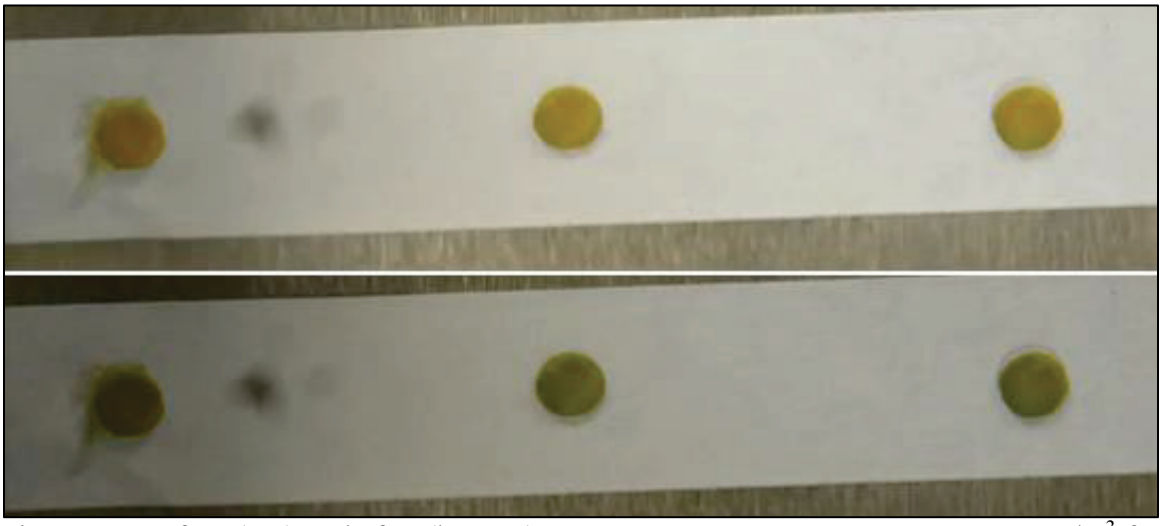

Figure 22. Before (top) and after (bottom) exposure to HD. Exposure was 0.395 mg/m^3 for 10 min for six randomly placed dots. Very slight color changes were seen on a time-lapse video for 0.1 mg/m^3, and no color changes were seen on time-lapse videos for 0.01 and 0.026 mg/m^3.

4.3.2 Aging Unexposed HD Dots

One observation worth noting: after a day, unexposed dots had started to show color on the edges. Figure 23 shows this color change after days 3, 4, and 5. These dots were stored in a Mylar bag with insulation covering the bag during testing, and the bag was vacuum-sealed at the end of the day. We were notified by IOS that HD dots are light-sensitive. Dots were tested with direct vapor to confirm functionality (Figure 24). At or around day 5, HD dots were no longer viable.

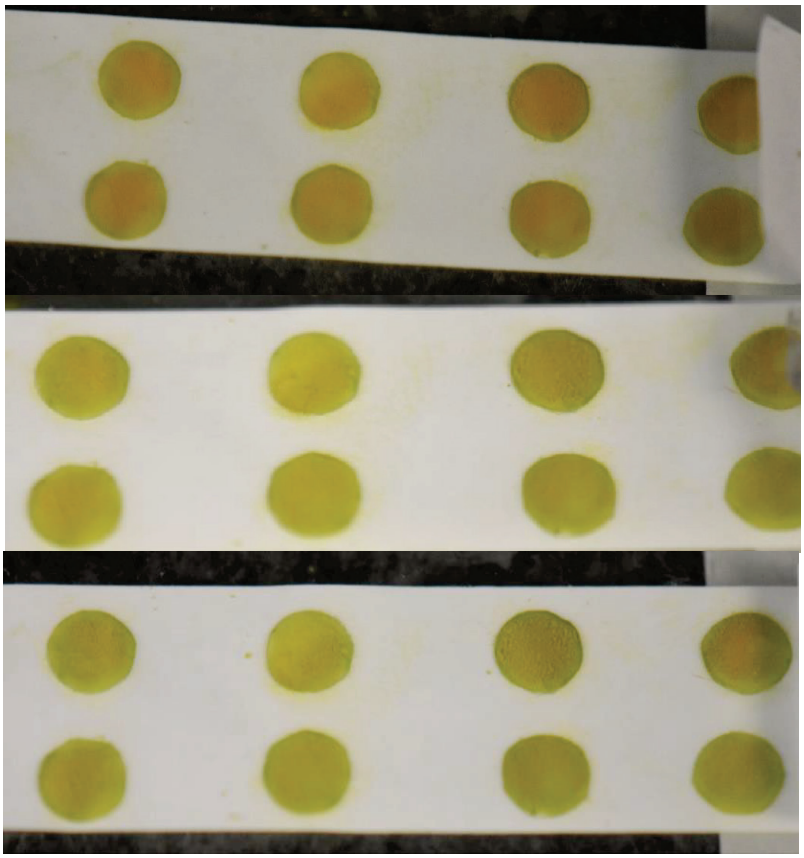

Figure 23. Unexposed HD dots starting to change color on the edges: day 3 (top), day 4 (middle), and day 5 (bottom).

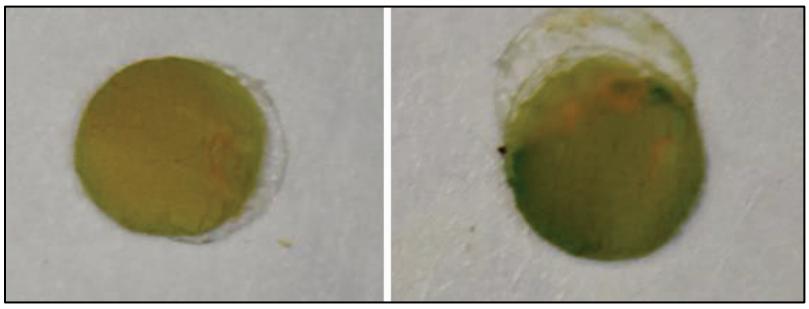

Figure 24. An HD dot before (left) and after (right) exposure to direct HD vapor at a 40 mL/min flow for 1 min.

4.3.3 Testing HD Dots on Clean N_2 Stream

When an HD dot was tested for 30 min using a clean N_2 stream, a very slight color change occurred (Figure 25). The GUI graphs for this are located in Appendix A.6.

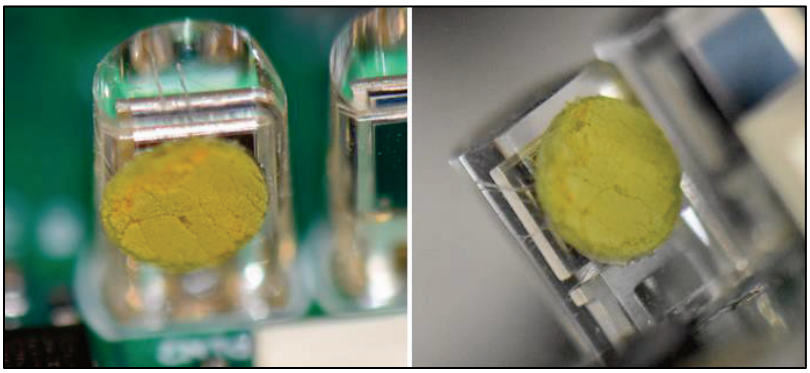

Figure 25. Position of dot on optical sensor for HD trials. Before (left) and after (right) 30 min of clean N_2 stream. A very slight color change occurred.

4.3.4 HD Testing Dots: Time-Lapse Data

The MOSA GEN1 blower and fan housing SUT45 were used for testing (the housing HD concentration was verified to be <0.0008 mg/m^3). An HD dot was placed on the left optical sensor and then exposed in the chamber to HD vapor for 47 min (at increasing, various concentrations: 0.0184, 0.1, 0.144, and ~1.72 mg/m^3). Time-lapse video was recorded at 30 s intervals (Figure 26). A 10-47-10 min test was performed. The GUI graphs for this test are in Appendix A.7. A downward trend was observed from 0 to 67 min. The GUI alarmed off and on the entire time, 0–67 min. There was a very slight decline in transmittance at 24.2, 38.3, 46.3, and 58 min. Table 12 summarizes the HD time-lapse data.

Table 12. Summary of HD Time-Lapse Data

Date	Time	Clean/Agent/ Clean (min)	Dot Open Date	Concentration (mg/m^3)	SUT No., Alarm (yes/no)	Color Change (yes/no)	Time Concentration Was Measured (min)
14-Dec-20	11:31	10.18/47/10	11-Dec-20	0.0184	45, y	y	15
				0.100			30
				0.144			43
				~1.72			48

Note: GUI graphs are located in Appendix A.7.
Red type, not the typical 10-10-10 test.

Figure 26. HD trial of dot before (left) and after (right) 47 min of HD exposure. This test series was performed at concentrations of 0.0184, 0.1, 0.144, and ~1.72 mg/m^3.

4.3.5 HD Test Matrix: Concentrations of 0.03–0.622 mg/m^3 (Dots Received 11 December 2020)

The MOSA GEN1 blower and fan housing were tested at concentrations of 0.03–0.622 mg/m^3 HD (detectable at 0.130–0.622 mg/m^3). The housing concentration was verified to be <0.0008 mg/m^3. Dots were placed on the left optical sensors and photodiodes (Figures 27 and 28), and a 10-10-10 test was performed. GUI data are available in Appendix A.8. It took at most 3 min for the GUI to stabilize at green during the initial clean stream. The GUI alarmed off and on during most of the time that the HD line was connected. The GUI never stabilized to green during the final clean stream. Table 13 summarizes the HD test matrix, which included the use of only one SUT placed in a sealed 2 L test chamber to test concentrations of 0.03–1.3 mg/m^3.

Table 13. Summary of HD Test Matrix: Concentrations 0.03–1.3 mg/m^3 (0.130–1.3 mg/m^3 Detectable)

No.	Date	Time	Clean/Agent/ Clean (min)	Dot Received/ Opened Date	Concentration (mg/m^3)	SUT No., Alarm (yes/no)	Color Change (yes/no)	Comments
1	15-Dec-20	7:52	10/10/10	11-Dec-20	0.45	43, y	y	Color change most likely due to natural change from light; transmittance seen at 10, 22.3 min
2	15-Dec-20	9:35	10/10/10	11-Dec-20	0.622	45, y	y	Color change most likely due to natural change from light; transmittance seen at 10.5, 20.3 min
3	15-Dec-20	11:22	10/10/10	11-Dec-20	0.03	56, n	y	Color change due to natural change from light; stayed green 15+ min; transmittance seen at 20 min; low concentration
4	15-Dec-20	13:09	10/10/10	11-Dec-20	0.13	57, y	y	Color change most likely due to natural change from light; transmittance seen at 22.3 min
5	16-Dec-20	10:44	10/10/10	11-Dec-20	0.600	58, n	n	Was one of the first dots taken from the last spot on the 2nd row white strip; nos. 1–4 were taken from 3rd row; bad dots
6	16-Dec-20	12:31	10/10/10	11-Dec-20	0.420	59, n	n	Was one of the first dots taken from the first spot on the 1st row white strip; nos. 1–4 were taken from 3rd row; bad dots
7	28-Dec-20	10:28	10/10/10	21-Dec-20/ 28-Dec-20	0.059	58, n	y	Color change due to natural change from light; alarmed off/on and steady decreased transmittance; low concentration
8	28-Dec-20	12:10	10/10/10	21-Dec-20/ 28-Dec-20	0.065	58, n	y	Color change due to natural change from light. alarmed off/on and steady decreased transmittance; low concentration
9	28-Dec-20	13:14	5/15/na	21-Dec-20/ 28-Dec-20	1.3	60, y	y	First time seeing a constant alarm during HD exposure; there is a steady decreased transmittance
10	29-Dec-20	11:04	10/0.6/na	21-Dec-20/ 29-Dec-20	0.8	60, na	y	Run stopped prematurely at 10.6 min; was green the whole time; there is a steady decrease in transmittance

Note: GUI graphs are located in Appendix A.8.
na, not available or not applicable.
Red type, not the typical 10-10-10 test.

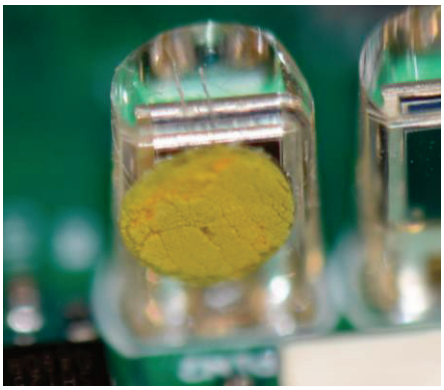

Figure 27. Example photograph of an HD dot before HD exposure.

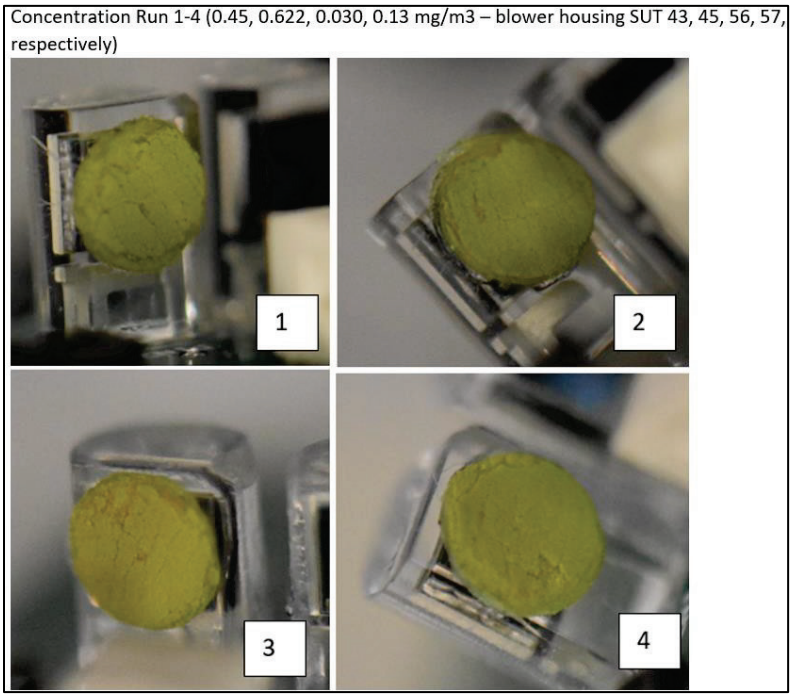

Figure 28. Dots after HD exposure for concentration runs 1–4.

4.3.6 HD Test Matrix: Concentrations 0.8–1.30 mg/m^3 (Dots Received 21 December 2020)

The MOSA GEN1 blower and fan housing were tested at concentrations of 0.059–1.3 mg/m^3 (detectable at 1.30 mg/m^3). The housing HD concentration was verified to be <0.0008 mg/m^3. New HD dots were received. New dots were placed on the left optical sensors and photodiodes (Figure 29). GUI data were incomplete for no. 10 (0.8 mg/m^3). For concentration 1.3 mg/m^3, a final clean run was not performed. The GUI for the 1.3 mg/m^3 concentration using the dots on 21 December 2020 provided the only constant alarm during HD exposure. In the other "alarmed" test, the GUI alarmed off and on during most of the time the HD line was connected. GUI data are available in Appendix A.8. For the dots received on

21 December 2020, transmittance steadily decreased from the start of the test. Table 13 summarizes the data obtained from performing the HD test matrix with only one SUT placed in a sealed 2 L test chamber for the concentrations of 0.03–1.3 mg/m^3.

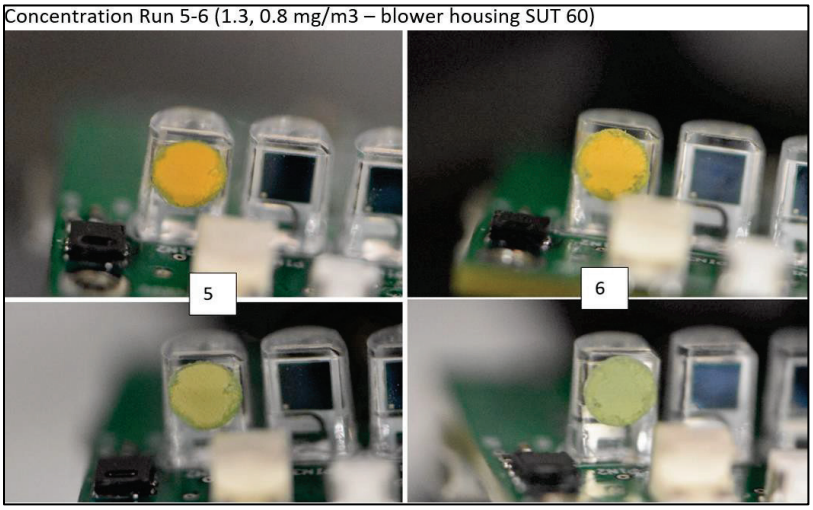

Figure 29. New dots before (top) and after (bottom) HD exposure for concentration runs 5 (no. 9) and 6 (no.10) in blower housing 60.

4.3.7 HD: 10 Trials Using Different MOSA GEN1 at Concentrations of 0.854 ± 0.926 mg/m^3

Dots were placed on the left optical sensors and photodiodes (Figure 30). A 10-10-10 test was performed. In trials 1 and 2, old dots were used. There was a consistent decline in the transmittance data from time 0 with the new dots, but the HD dots (in general) were never able to stabilize at a constant green during the initial clean N_2 stream. The GUI alarmed during the HD line connect and took as long as 2.9–6 min (11 December 2020 dots) and as long as 0.2–1.2 min (21 December 2020 dots) for a constant alarm. The GUI never stabilized to green during the final clean stream. The GUI slope data (versus transmittance data) was a better graph for observing clean versus HD stream changeover, but it was harder to gauge because changes were very subtle. It was harder to perform live observations of the GUI transmittance data graph for clean versus HD stream because the line was always decreasing from time 0, and the changes were very gradual. Table 14 summarizes the 10 HD trials performed, with only one SUT placed in a sealed 2 L test chamber for HD concentrations of 0.854 ± 0.926 mg/m^3. GUI data are available in Appendix A.9.

Table 14. Summary of 10 HD Trials at 0.854 ± 0.926 mg/m^3

No.	Date	Time	Clean/Agent/ Clean (min)	Dot Received/ Opened Date	Concentration (mg/m^3)	SUT No., Alarm (yes/no)	Color Change* (yes/no)	Comment	Constant Alarm Time (min)†
1	16-Dec-20	7:37	10/10/10	11-Dec-20	0.386	56, y	y	Transmittance seen at 10, 20.3 min	6.0
2	16-Dec-20	9:11	10/10/10	11-Dec-20	0.5	57, y	y	Transmittance seen at 12, 20 min	2.9
3	29-Dec-20	8:06	10/10/10	21-Dec-20/ 28-Dec-20	1.78	58, y	y	Color change looks more blue-green; transmittance seen at 740 s, 1220 s	1.1
4	29-Dec-20	9:40	10/10/10	21-Dec-20/ 29-Dec-20	1.38	59, y	y	Transmittance seen at 760 s, 1240 s	0.2
5	29-Dec-20	12:43	10/10/10	21-Dec-20/ 29-Dec-20	1.2	61, y	y	Transmittance seen at 660 s, 1200 s	0.9
6	29-Dec-20	14:11	10/10/10	21-Dec-20/ 29-Dec-20	0.67	62, y	y	Transmittance seen at 740 s, 1230 s	0.9
7	30-Dec-20	7:37	10/10/10	21-Dec-20/ 30-Dec-20	0.796	60, y	y	Transmittance seen at 640 s, 1210 s	0.9
8	30-Dec-20	10:23	10/10/10	21-Dec-20/ 30-Dec-20	0.335	65, y	y	Transmittance seen at 1340 s	0.2
9	30-Dec-20	11:56	10/10/10	21-Dec-20/ 30-Dec-20	0.695	66, y	y	Transmittance seen at 660 s, 1300 s	0.8
10	30-Dec-20	13:24	10/10/10	21-Dec-20/ 30-Dec-20	0.8	64, y	y	Transmittance seen at 680 s, 1220 s	1.2

Note: GUI graphs are located in Appendix A.9.

*Color change seen in dots could be due to natural change from light.

†HD dot (21-Dec-20): There was a consistent decline in transmittance data from time 0 with the new dots, but the HD dots (in general) were never able to achieve a constant green before the exposure run was started.

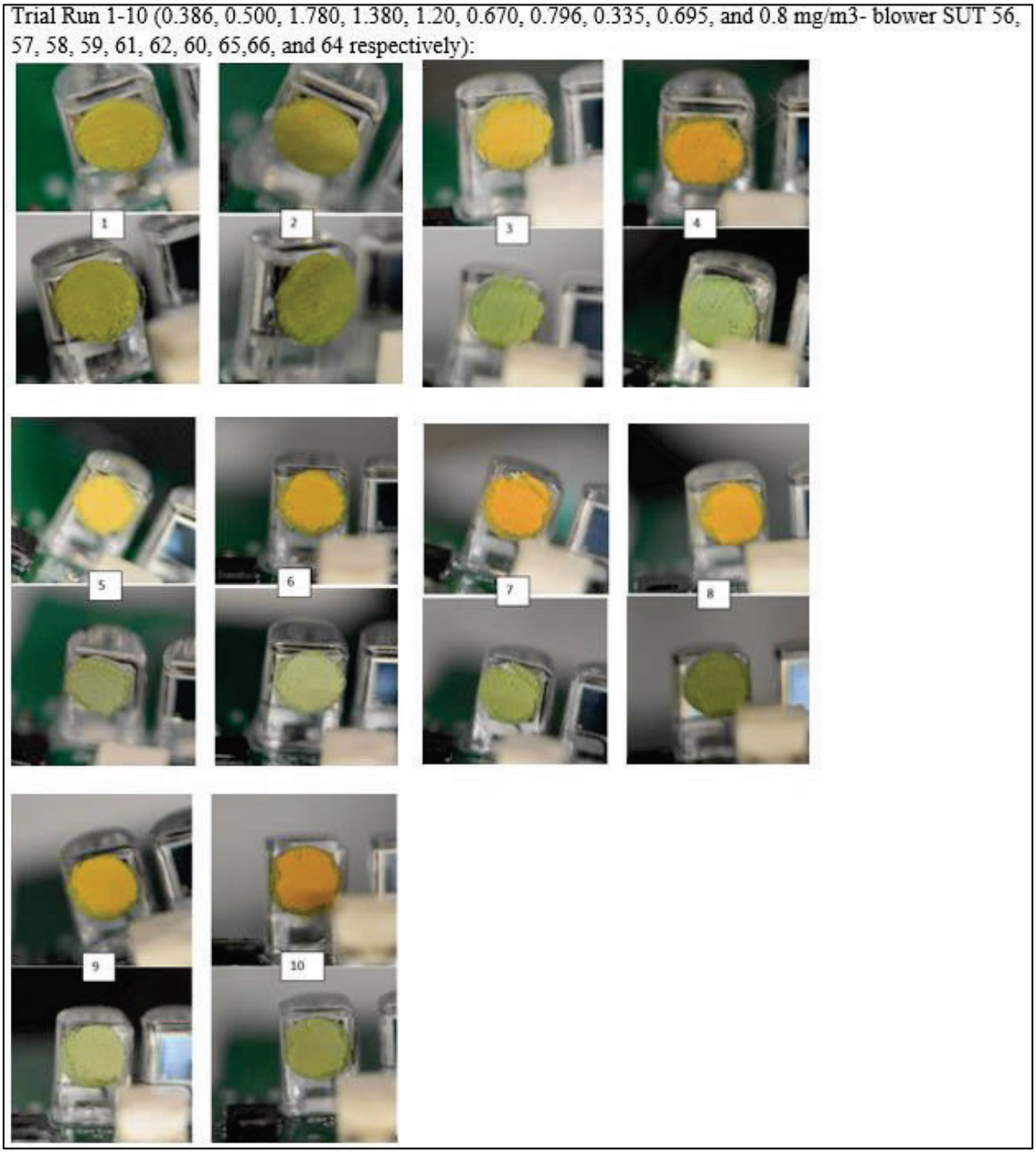

Figure 30. Dots before (top) and after (bottom) HD exposure for 1–10 trial runs at 0.335–1.78 mg/m^3 range in concentration. Five-day-old dots were used in trials 1 and 2.

4.4 VX

Dot chemistry was examined for assessment and evaluation of the chemical agent VX.

4.4.1 VX Testing the Dots: Direct VX Vapor in Jar and Liquid

VX dots were provided for ambient vapor and direct liquid exposure (Figures 31 and 32, respectively). High vapor concentration was used for dots challenged with 2 µL of neat VX placed in the bottom of a glass jar. A time-lapse video with 30 s intervals was recorded (Figure 31). For the liquid-exposed dot from Figure 32, it was observed that color faded with time (Figure 33).

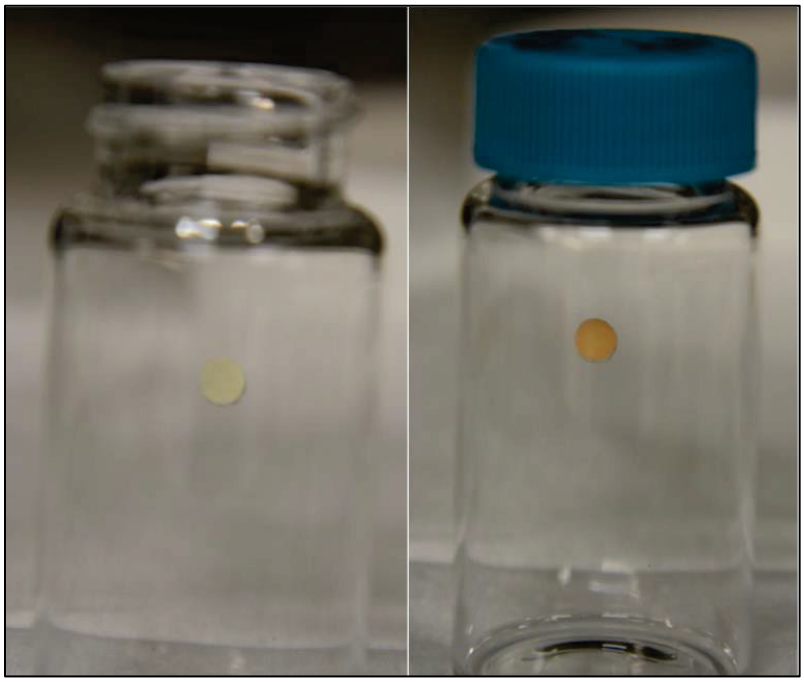

Figure 31. VX dot placed inside wall of a 20 mL glass jar;
100 µL of VX deposited on the jar bottom.

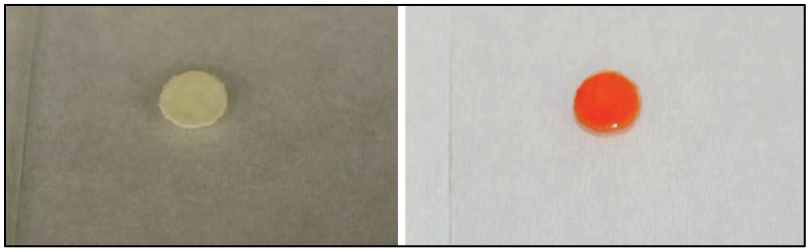

Figure 32. An ~4 mm liquid drop of VX was deposited directly onto the VX dot.

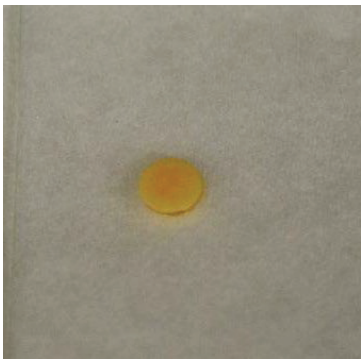

Figure 33. A VX dot photographed 30 min after liquid deposit.
Color faded with time and VX liquid exposure.

4.4.2 Testing VX Dots on Clean N_2 Stream

VX dots were tested with 10 min of a clean N_2 stream. The GUI graphs are located in Appendix A.10.

4.4.3 VX Testing the Dots: Time Lapse

The MOSA GEN1 blower and fan housing SUT43 was used for this test. The housing was verified to be clear of VX. A VX dot was placed on the right optical sensor and then exposed in the chamber. VX is a "sticky" compound, and previously, issues with HD clearance arose; therefore, a new chamber was set up. This setup allowed for complete clearance of analyte from the chamber. The new chamber is shown in Figures 34–36.

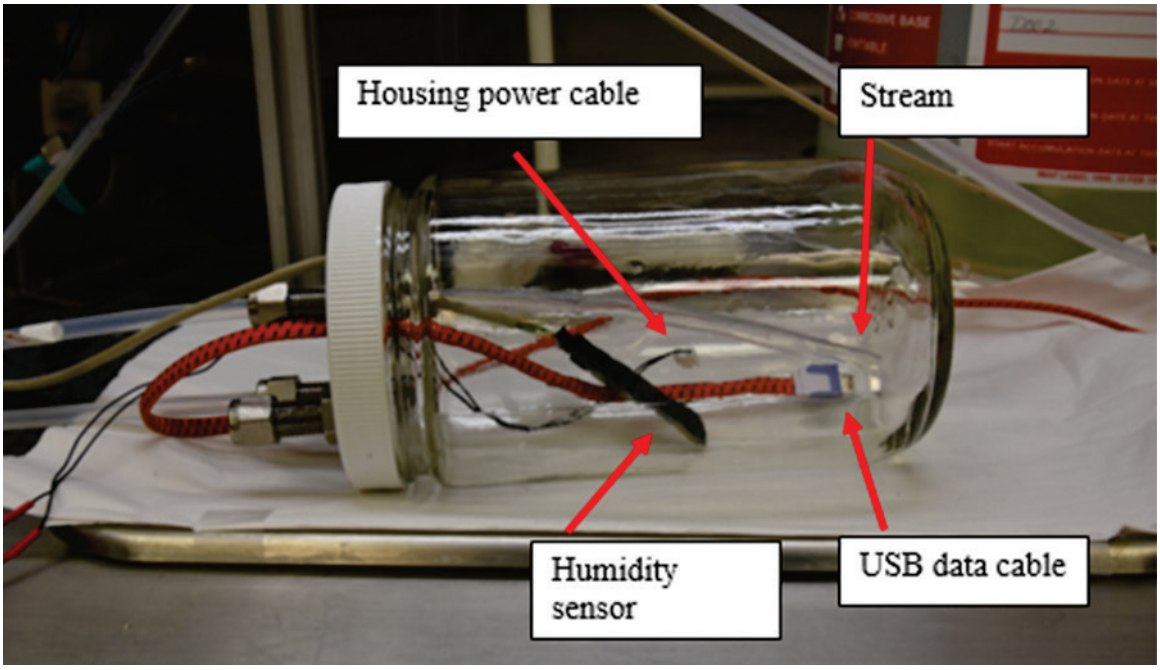

Figure 34. New chamber setup (side view).

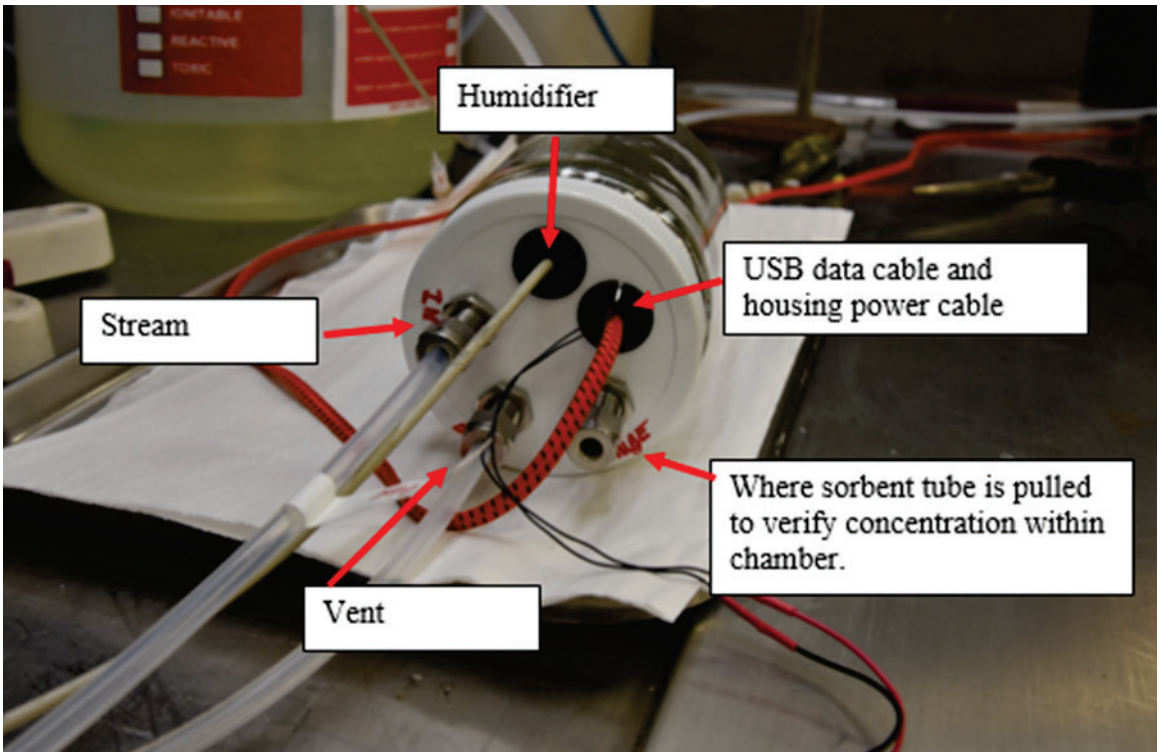

Figure 35. New chamber setup (cap view).

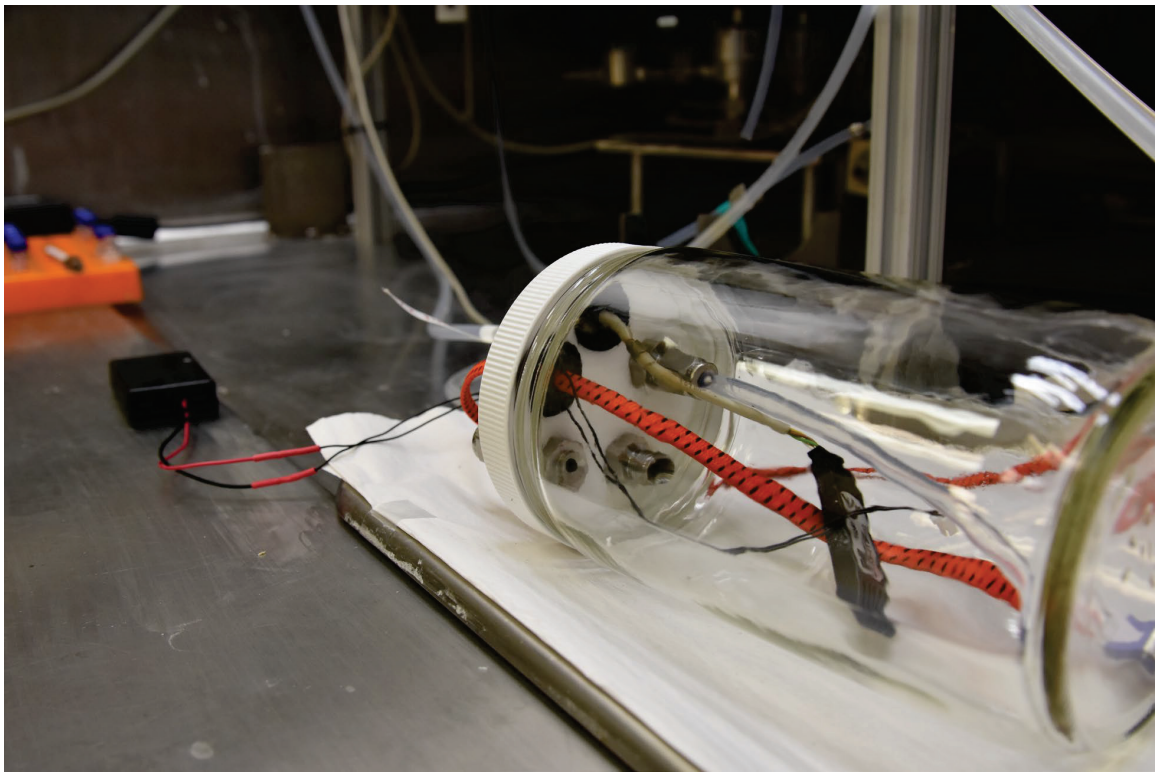
Figure 36. New chamber setup (under-cap view).

Based on the positive response to the compound, VX dots were exposed to VX vapor (with flow entering from the right side) in a chamber at various concentrations, as follows:

1. Dots were exposed to VX in the chamber for 20 min: at 0.0067 mg/m^3 for 5 min, and at 0.0075 mg/m^3 for 15 min. No visible color changes were observed.
2. Time-lapse recordings of three VX dots exposed to VX in chamber were continued for an additional 20 min, at 0.028 mg/m^3 for 35 min. No visible color changes were observed.
3. Time-lapse recordings of three VX dots exposed to VX in the chamber were continued for an additional 60 min, at 0.056 mg/m^3 for 100 min. Slight color changes were observed (Figure 37; time-lapse video at 30 s intervals were recorded for this image). The contrast with an unexposed dot makes the color change easier to see.
4. Table 15 summarizes the VX time-lapse data.
5. The SUT43 blower fan housing was used during the time-lapse setup. 100 min of data were collected during the time lapse. Transmittance was observed at a 0.0075 mg/m^3 concentration. Transmittance increased around 70 min, which cannot be explained. The GUI did not alarm during the run. The GUI graphs are located in Appendix A.11.

Table 15. Summary of VX Time-Lapse Data

Date	Time	Test Type	Dot Open Date	Concentration (mg/m^3)	SUT No., Alarm (yes/no)	Color Change (yes/no)	Comments	Time Concentration Was Measured (min)
20-Jan-21	11:37	0–20 min time lapse 1	12-Jan-21	0.00665	43, n	y, at end of run time	Three dots (12-Jan-21) exposed in chamber; another concentration was 0.0075 at 15 min	5
		20–40 min time lapse 2		0.028			Continue same three dots (12-Jan-21) exposure in chamber	35
		40–100 min time lapse 3		0.056			Continue same three dots (12-Jan-21) exposure in chamber	100

Note: GUI graphs are located in Appendix A.11.

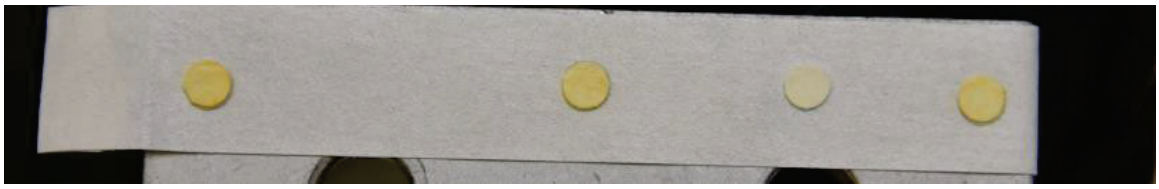

Figure 37. Three VX dots were exposed to VX in the chamber for a total of 100 min. Concentration of chamber was 0.056 mg/m^3 at 100 min. Slight color change was observed when an unexposed dot was placed (third from left) for contrast.

VX is a very nonvolatile compound. Maximum flow (300 mL/min) was reached on the MFC, which provided a concentration of 0.0308 mg/m^3. The only other way to increase the VX concentration would be to use a high-flow MFC and heat the saturator cell.

4.4.4 VX Test Matrix: Concentrations 0.0050, 0.0148, and 0.0280 mg/m^3

The MOSA GEN1 blower and fan housing were tested at VX concentrations of 0.0050, 0.0148, and 0.0280 mg/m^3. The housing was verified to be clear of VX. Dots were placed on the right of the optical sensors and photodiodes (Figure 38). A 10-10-10 test was performed. Transmittance changed during the VX connection, but the change was very gradual. No color changes were observed, although it is possible that the color in the edges of the dots changed. For concentration runs 2 and 3, dots were placed next to an unexposed dot for contrast (Figures 39 and 40, respectively). The GUI did not alarm during the runs. For concentration run 3, transmittance decreased during the initial clean line by about 0.1% for 10 min. A very slight color change was observed using side-by-side comparison with a nonexposed dot. Table 16 summarizes the VX test matrix, which was performed with only one SUT placed in a sealed 1 L test chamber for concentrations of 0.0050, 0.0148, and 0.028 mg/m^3. The GUI data are available in Appendix A.12.

Table 16. Summary of VX Test Matrix: Concentrations 0.005–0.028 mg/m^3

No.	Date	Time	Clean/Agent/ Clean (min)	Dot Open Date	Concentration (mg/m^3)	SUT No., Alarm (yes/no)	Color Change (yes/no)	Comment
1	21-Jan-21	11:39	10/10/10	12-Jan-21	0.005	43, n	n	Transmittance changed during VX connect but very gradually
2	21-Jan-21	13:30			0.0148	45, n	n	Transmittance changed during VX connect but very gradually; possibly the edges may have changed color
3	25-Jan-21	7:50			0.028	45, n	y	Very slight color change

Note: GUI graphs are located in Appendix A.12.

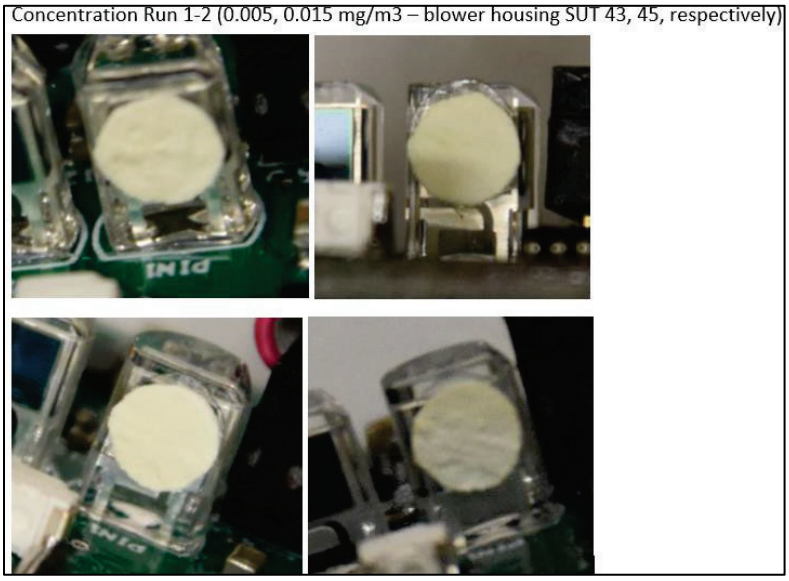

Figure 38. Dots before (left) and after (right) VX exposure
for concentration runs 1 (top) and 2 (bottom).

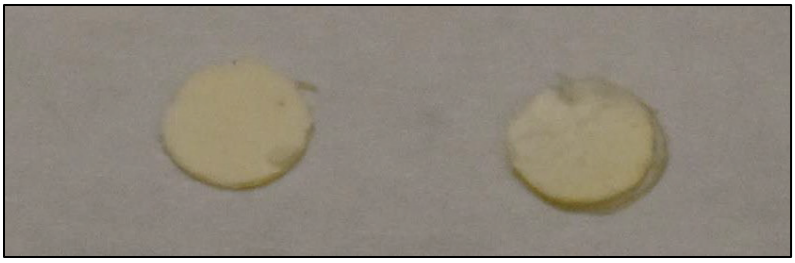

Figure 39. Dots before (left) and after (right) VX exposure for concentration run 2.

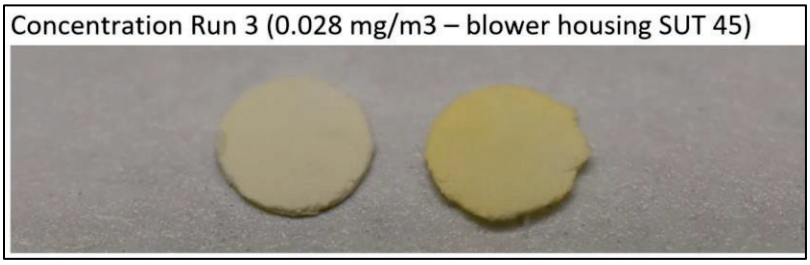

Figure 40. Dots before (left) and after (right) VX exposure for concentration run 3.

4.4.5 VX: 10 Trials Using Different MOSA GEN1 at 0.0308 ± 0.0078 mg/m^3 Concentration

Dots were placed on the right of the optical sensors and photodiodes, and a 10-10-10 test was performed. Transmittance changed during the cleaning and VX connection. The GUI did not alarm during the 10 trial runs. The color change was very slight for all 10 trials (Figures 41 and 42). The contrast is better viewed in real time than in photographs. A huge *O,O'*-diethyl methylphosphonothionate (DEMPS) impurity appeared during sampling from the VX saturator cell that decreased each day. This DEMPS decrease could be the reason for the improved GUI data that were collected from the 10 units with each passing day. Table 17 summarizes the results from the 10 VX trials performed with only one SUT placed in a sealed 1 L test chamber with an approximately 0.0308 ± 0.0078 mg/m^3 VX concentration. GUI data are available in Appendix A.13.

Table 17. Summary of VX Trials at 0.0308 ± 0.0078 mg/m^3

No.	Date	Time	Clean/Agent/ Clean (min)	Dot Open Date	Concentration (mg/m^3)	SUT No., Alarm (yes/no)	Color Change (yes/no)	Comment
1	22-Jan-21	7:57	10/10/10	12-Jan-21	0.0316	56, n	y	Very slight color change
2	22-Jan-21	9:13	10/10/10	12-Jan-21	0.0314	57, n	y	Very slight color change
3	22-Jan-21	10:19	10/10/10	12-Jan-21	0.0250	58, n	y	Very slight color change
4	22-Jan-21	11:20	10/10/10	12-Jan-21	0.0298	59, n	y	Very slight color change
5	22-Jan-21	13:00	10/10/10	12-Jan-21	0.0292	60, n	y	Very slight color change
6	22-Jan-21	14:08	10/10/10	12-Jan-21	0.0294	61, n	y	Very slight color change
7	25-Jan-21	8:51	10/10/10	12-Jan-21	0.0284	62, n	y	Very slight color change
8	25-Jan-21	9:50	10/10/10	12-Jan-21	0.0333	64, n	y	Very slight color change
9	25-Jan-21	12:27	10/10/10	12-Jan-21	0.0318	65, n	y	Very slight color change
10	25-Jan-21	13:33	10/10/10	12-Jan-21	0.0386	66, n	y	Very slight color change
11	28-Jan-21	13:53	10/10/10	12-Jan-21	0.0306	66, y	y	Very slight color change; new/updated GUI alarms

Note: GUI graphs are located in Appendix A.13.

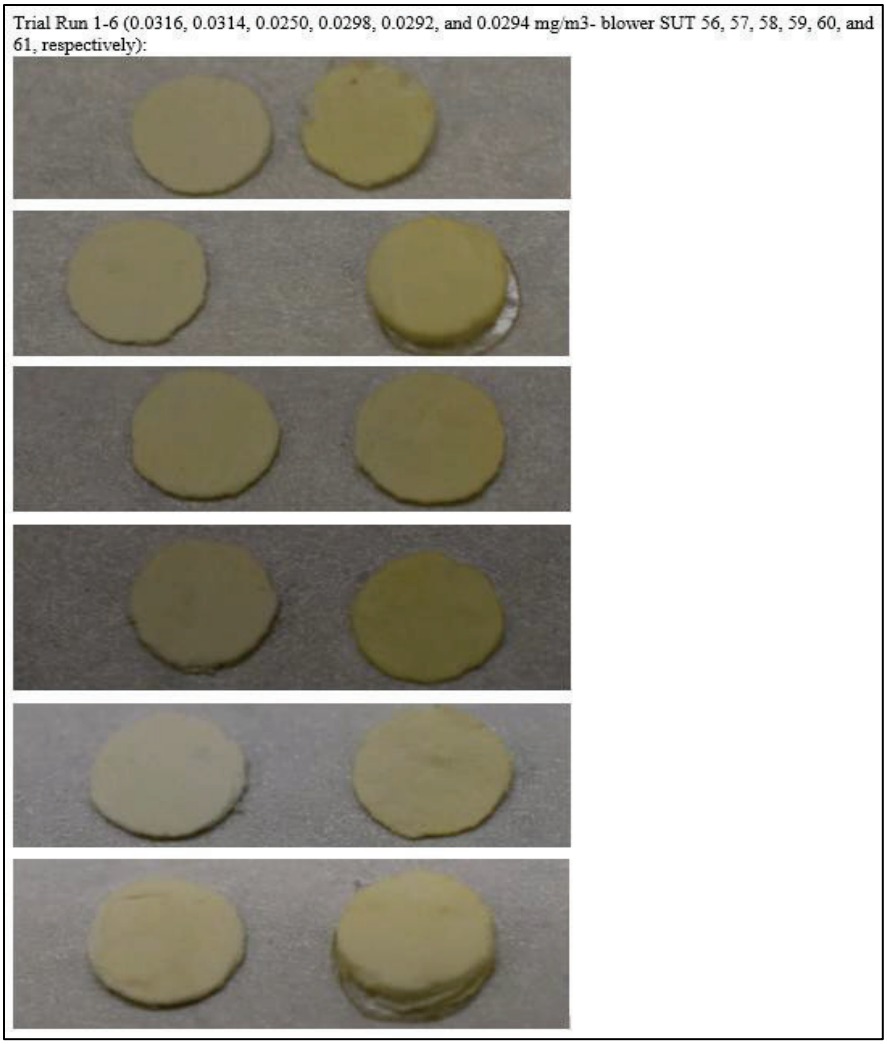

Figure 41. Dots unexposed (left) and exposed (right) to VX during trial 1–6 runs at a 0.0250–0.0316 mg/m^3 range in concentration.

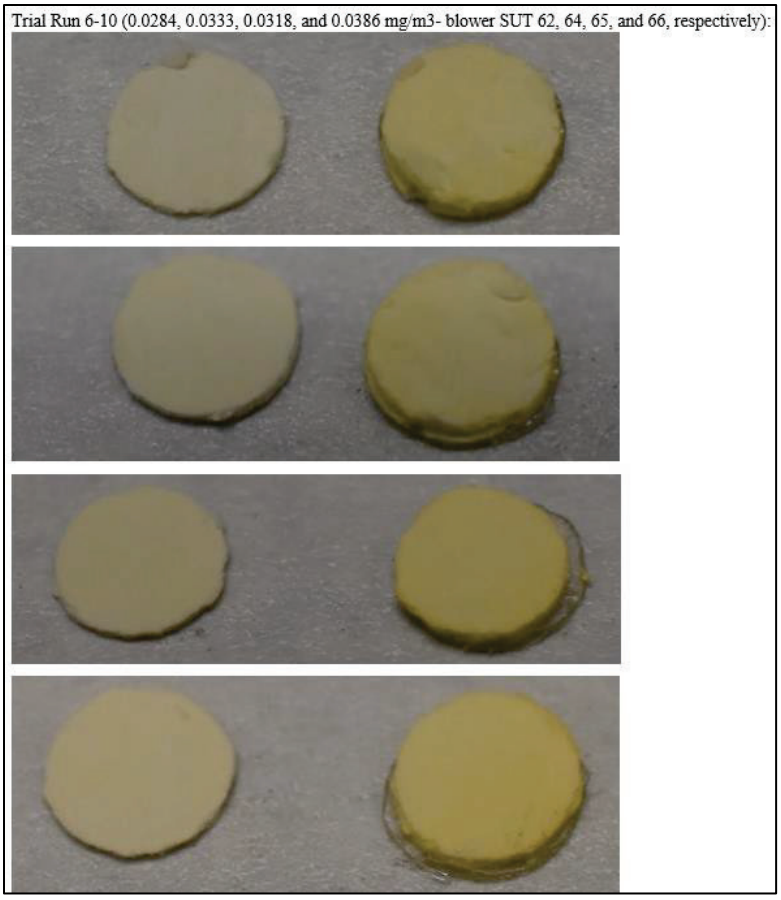

Figure 42. Dots unexposed (left) and exposed (right) to VX during trial 6–10 runs at a 0.0284–0.0386 mg/m^3 range in concentration.

4.4.6 Updated GUI for VX Testing

The GUI was updated, and one trial was performed using the update. The GUI alarmed at 90–140 s during the initial clean stream and also during all of the VX stream. The GUI alarmed for a bit during the changeover to the VX line. The GUI turned green once as the VX line was connected, and it stayed alarmed for 28 s afterward. Changes were observed in transmittance and slope during the VX connection. During the final clean stream, the GUI initially alarmed and then returned to green at 43 s after the final clean line connection. A very slight color change was observed in a side-by-side comparison with the nonexposed dot (Figure 43).

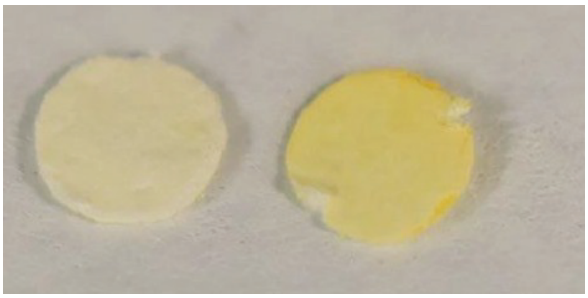

Figure 43. Dots unexposed (left) and exposed (right) to VX for one trial using updated GUI and 0.0306 mg/m^3 VX concentration.

4.5 GD

Performance of the GD dot was assessed and evaluated. Several attempts were made to identify a high-quality functional GD dot. GD is unique in that the material is stable to hydrolysis as compared to GB. Alteration of the GB dot chemistry would be required to understand that stability and achieve the desired performance. For this material, the protocol was well established and was followed as outlined.

4.5.1 GD Testing the Dots: Time-Lapse Video

Four GD dots (one opaque dot, two 240La,150B dots, three 240Eu,150B dots, and four LaZn 20210426 dots) were provided for ambient vapor exposure in a high vapor concentration. Dots were challenged in a glass jar with 200 µL of neat GD placed on the top portion of the jar (Figure 44). A time-lapse video using 30 s intervals was recorded. Color changes were observed for all dots except dot 1, the opaque dot.

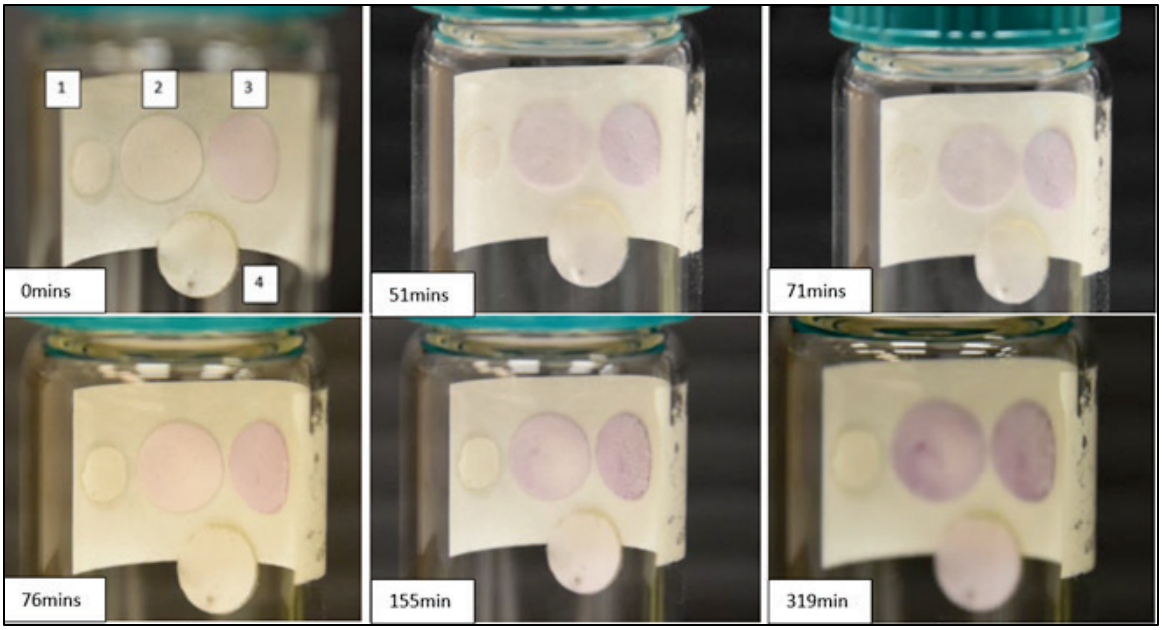

Figure 44. An array of four different, random dots in a 20 mL glass jar with 200 µL of GD, exposed for 319 min.

An array of those same four different dots were tested with GD vapor in the chamber (Figure 45). Flow entered from the right side, and the exposure time was 30 min. The concentration was 29.5 mg/m^3 at 20 min and 29 mg/m^3 at 30 min. A time-lapse video using 30 s intervals was recorded (Figure 45). Color changes were observed at 20–30 min. The testing and time-lapse data for the four dots are summarized in Table 18.

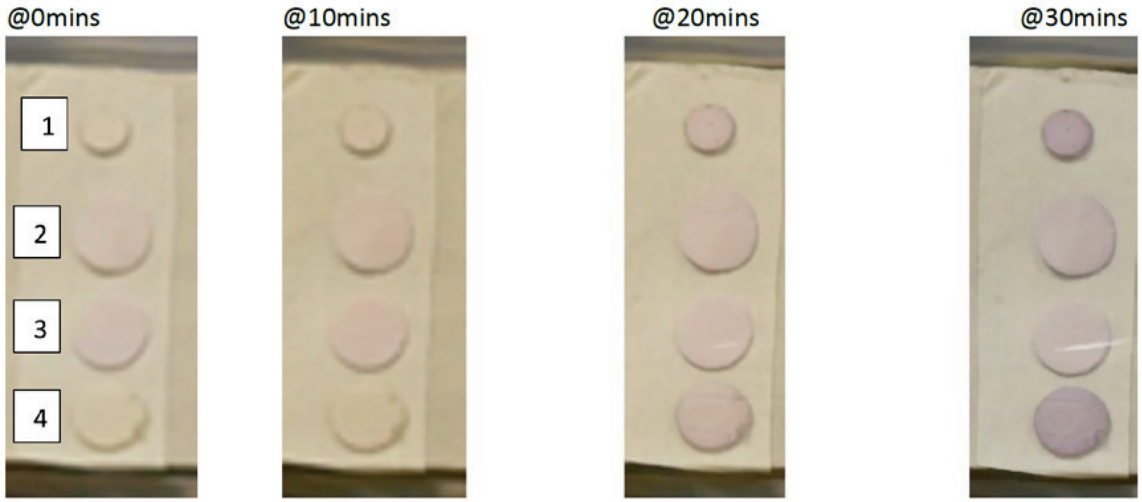

Figure 45. Four dots exposed to GD vapor in the chamber for a total of 30 min.

Dots 2–4 (shown in Figure 45) were observed as being responsive to GD vapor and ambient GD vapor in the jar. Each was tested three times with the MOSA GEN1 using the new GUI. Dots were placed on the left of the optical sensors and photodiodes of the MOSA GEN1 blower and housing. A 10-10-10 test was performed. The transmittance and slope changed during the GD connection for all dots. Color changes were observed for all dots. Unexposed and exposed dots are shown in Figure 46. The GUI analysis for the three dots is summarized in Table 19. GUI data are available in Appendix A.14.

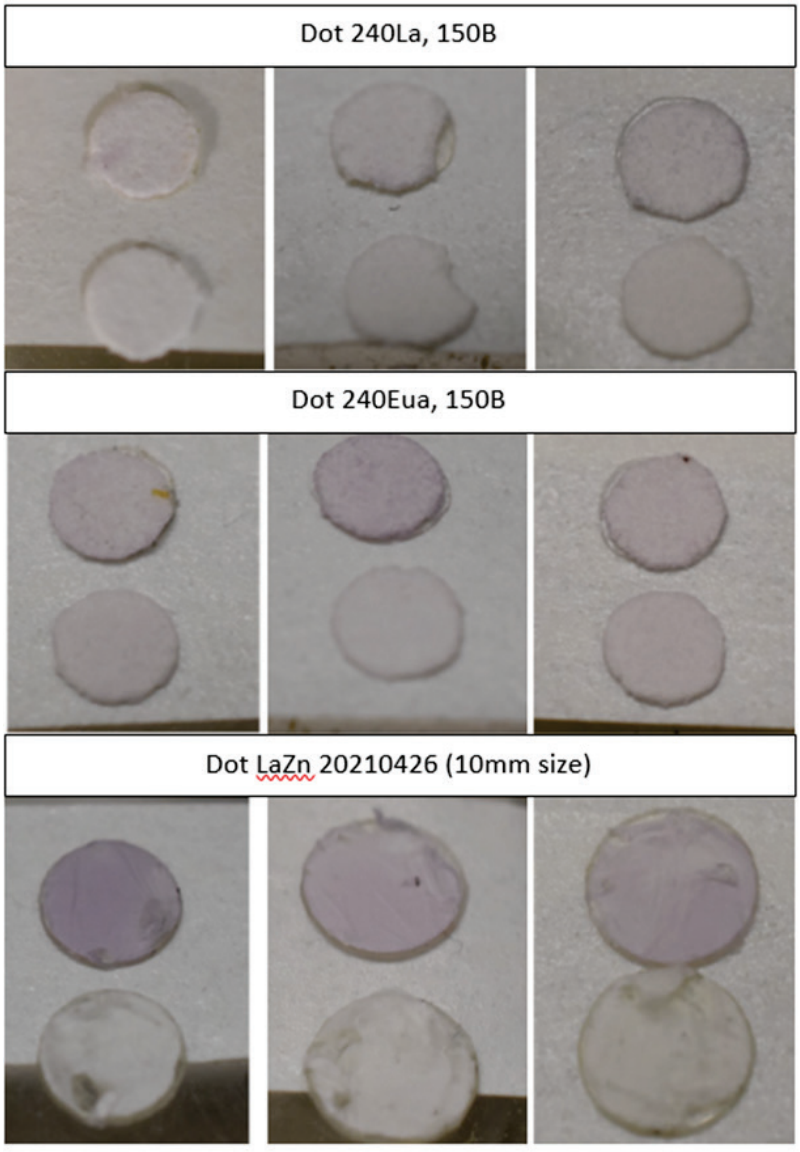

Figure 46. Dots 2–4, unexposed (bottom) and exposed (top) to GD.

Table 18. Summary of GD Time-Lapse and Ambient Vapor Data for Four Dots

Analysis Date	Analyte	Test Type	Total Run Time	Test	Concentration during Exposure (mg/m^3)	Time Concentration Was Measured (min)	Comments
28-Apr-21	GD	Ambient vapor exposure	Up to overnight	4 dots exposed to vapor from 100 µL neat GD in 20 mL jar: • dot 1, opaque dot received 30-Mar-21; • dot 2, 240La,150B received 26-Apr-21; • dot 3, 240Eu,150B received 26-Apr-21; • dot 4, LaZn 20210426 received 28-Apr-21 (was incorrectly labeled LaEu)	na	na	Dots 2 and 3 were quicker to change color; no color change for dot 1 (Figure 44)
28-Apr-21	GD	0–30 min time lapse		Same four GD dots exposure in chamber	29.5	10	Dot 4 was quicker to change color; all changed color (Figure 45)
					29	30	

na, not available or not applicable.

Table 19. Comparison of GUI for Three Selected GD Dots*

No.	Date	Time	Clean/Agent/ Clean (min)	Dot Used	Dot Open Date	Concentration (mg/m^3)	SUT No. Alarm (yes/no)	Color Change (yes/no)	Comment
1	29-Apr-21	10:25	10/10/10	240La,150B	30-Apr-21	12.47	62, y	y	Ever-so-slight color change; initial clean, alarmed off and on; final clean, one alarm blip but mostly stayed green
2	29-Apr-21	11:06	10/10/10	240Eu,150B	30-Apr-21	12.65	66, y	y	Ever-so-slight color change; initial clean, alarmed and went to green at 480 s
3	29-Apr-21	13:19	10/10/10	LaZn 20210426	30-Apr-21	13.1	64, y	y	Color change; sensor life at end of exposure time was 40%
4	29-Apr-21	13:57	10/10/10	240La,150B	30-Apr-21	12.29	61, y	y	Ever-so-slight color change; initial clean, alarmed once; final clean, one alarm blip but mostly stayed green
5	29-Apr-21	14:34	10/10/10	240Eu,150B	30-Apr-21	14.1	60, y	y	Color change; initial clean, alarmed off and on; final clean, alarmed off and on until end of run
6	30-Apr-21	07:39	10/10/10	LaZn 20210426	30-Apr-21	11.16	59, y	y	Color change; sensor life at end of exposure time was 59%
7	30-Apr-21	08:17	10/10/10	240La,150B	30-Apr-21	11.44	62, y	y	Slight color change; final clean, alarmed at 1780 s and stayed alarmed
8	30-Apr-21	08:55	10/10/10	240Eu,150B	30-Apr-21	11.85	66, y	y	Slight color change; final clean, alarmed once
9	30-Apr-21	09:35	10/10/10	LaZn 20210426	30-Apr-21	10.58	64, y	y	Color change; sensor life at end of exposure time was 59%

Note: GUI graphs are located in Appendix A.14.
*Concentration test: chamber and housing cleared <WPL, and batteries for power supply were replaced before each trial (Figure 46).

4.5.2 GD Test Matrix: 1.41, 2.47, and 3.68 mg/m^3 Concentrations

The MOSA GEN1 blower and fan housing were tested at various concentrations (1.41, 2.47, and 3.68 mg/m^3). The fan housing was verified to be clear of GD. The dot (LaZn 20210426) was placed on the left of the optical sensors and photodiodes. A 10-10-10 test was performed. Transmittance and slope changed during the GD connection for the 2.47 and 3.68 mg/m^3 concentrations. No color change was observed for the 1.41 mg/m^3 concentration. The GUI did not alarm during the run. A vague color change was observed for the 2.47 mg/m^3 concentration, and a slight color change was observed for the 3.68 mg/m^3 concentration. The GUI did alarm during these runs (Figure 47). Table 20 summarizes the data for the performance of the GD test matrix with only one SUT placed in a sealed 1 L test chamber for these three concentrations. GUI data are available in Appendix A.15.

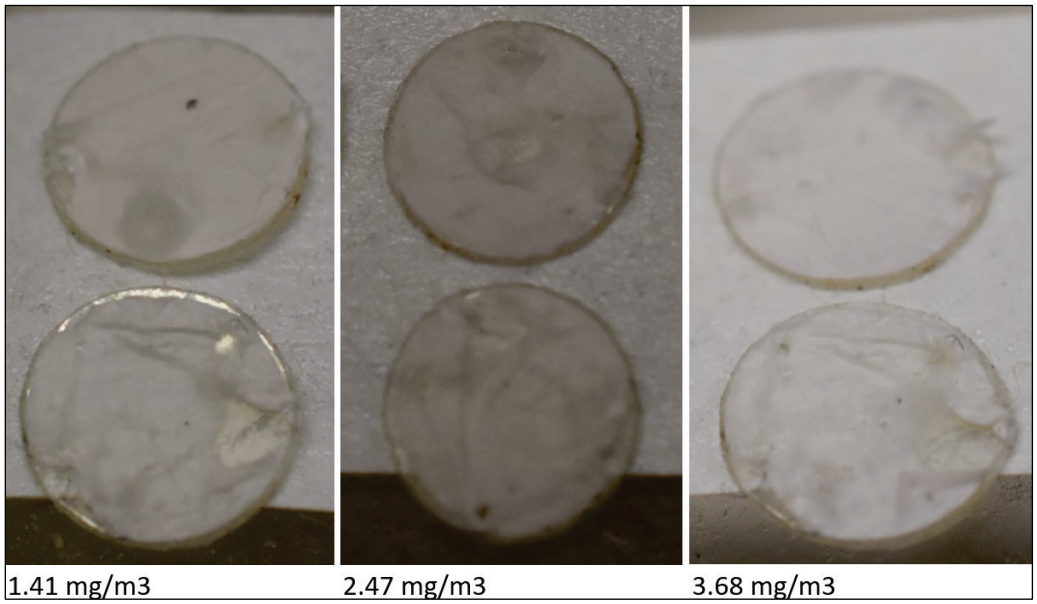

Figure 47. Unexposed (bottom) and exposed (top) dots for three GD concentrations: 1.41 mg/m^3 (left), 2.47 mg/m^3 (middle), and 3.68 mg/m^3 (right)

Table 20. Summary of GD Test Matrix: 1.41, 2.47, and 3.68 mg/m^3 Concentrations*

No.	Date	Time	Clean/Agent/ Clean (min)	Dot Used	Dot Open Date	Concentration (mg/m^3)	SUT No., Alarm (yes/no)	Color Change (yes/no)	Comment
1	3-May-21	8:25	10/10/10	LaZn 20210426	30-Apr-21	1.41	43, n	n	No transmittance response observed
2	3-May-21	09:58	10/10/10	LaZn 20210426	30-Apr-21	2.47	45, y	†	Color change was vague; normal transmittance response observed
3	3-May-21	10:58	10/10/10	LaZn 20210426	30-Apr-21	3.68	56, y	y	Normal transmittance response observed

Note: GUI graphs are located in Appendix A.15.
*Concentration test: chamber and housing cleared <WPL and batteries for power supply were replaced before each trial (Figure 47).
†Color change was not clear.

4.5.3 GD Response Measurement for 10 Trials Using MOSA GEN1 at 4.17 ± 0.33 mg/m^3 Concentration

The GD dots were placed on the left of the optical sensors and photodiodes, and the standard 10-10-10 min test was performed. The GUI response data available for transmittance changed during the clean air stream and the GD connection. For all tests in the 10 trial runs, the GUI alarmed during the GD line connection. The color changes were very slight to definite for all trials (Figure 48). For the 10 trials, an indisputable visual color response was apparent for all dots. For all exposed dots, the contrast was better viewed when the dots were placed against a white background or next to an unexposed dot. Table 21 summarizes the 10 GD trials performed with only 1 SUT placed in a sealed 1 L test chamber for the 4.17 ± 0.33 mg/m^3 concentration. The GUI data are available in Appendix A.16.

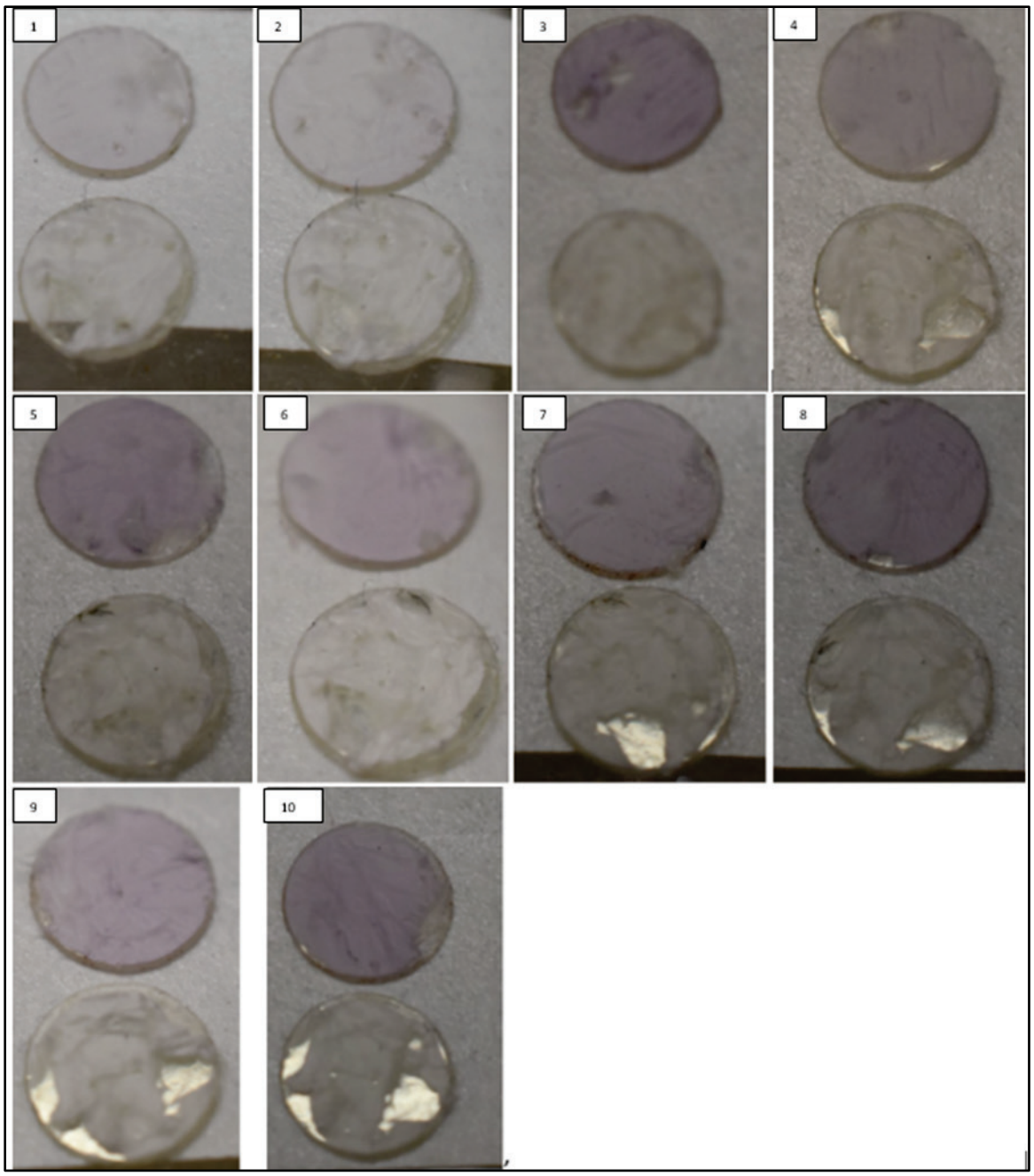

Figure 48. Unexposed (bottom) and exposed (top) for the 10 LaZn 20210426 dots exposed to GD vapor.

Table 21. Summary of 10 GD Trials at 4.17 ± 0.33 mg/m^3 Concentration*

No.	Date	Time	Clean/Agent/ Clean (min)	Dot Used	Dot Open Date	Concentration (mg/m^3)	SUT No., Alarm (yes/no)	Color Change (yes/no)	Comment
1	3-May-21	13:14	10/10/10	LaZn 20210426	30-Apr-21	3.84	58, y	y	Slight color change; initial clean, alarmed off and on and went to green 300 s; final clean, cleared out at 2.8 min
2	3-May-21	13:55	10/10/10	LaZn 20210426	30-Apr-21	4.17	59, y	y	Slight color change; initial clean, alarmed off and on and went to green 500 s; final clean, cleared out at 3.3 min
3	4-May-21	07:50	10/10/10	LaZn 20210426	30-Apr-21	4.2	57, y	y	Color change; final clean, cleared out at 7 min
4	4-May-21	08:55	10/10/10	LaZn 20210426	30-Apr-21	4.4	60, y	y	Color change; final clean, cleared out at 3 min
5	4-May-21	09:37	10/10/10	LaZn 20210426	30-Apr-21	4.4	61, y	y	Color change; final clean, cleared out at 2.8 min
6	4-May-21	10:57	10/10/10	LaZn 20210426	30-Apr-21	4.15	63, y	y	Color change; final clean, cleared out at 3.5 min
7	4-May-21	12:43	10/10/10	LaZn 20210426	3-May-21	4.42	62, y	y	Color change; final clean, cleared out at 5.3 min
8	4-May-21	13:21	10/10/10	LaZn 20210426	3-May-21	3.99	64, y	y	Color change; final clean, cleared out at 8.5 min
9	4-May-21	13:58	10/10/10	LaZn 20210426	3-May-21	4.15	65, y	y	Color change; final clean, cleared out at 5 min
10	4-May-21	14:35	10/10/10	LaZn 20210426	3-May-21	4	66, y	y	Color change; final clean, cleared out at 4.8 min

Note: GUI graphs are located in Appendix A.16.
*Concentration test: chamber and housing cleared <WPL, and batteries for power supply were replaced before each trial. Started using USB power cord for Trials 7–10 (Figure 48).

5. FINAL DISCUSSION AND CONCLUSION

5.1 Discussion

Presented are the results for the compounds CG, GB, GD, HD, and VX. Several pre-evaluations were performed to ascertain the best-performing dot formulation for the chemical targeted for detection. After the exposure concentrations were established for the dots, the minimum detectable concentrations and specific concentrations required to perform the repeatability and reliability trials were determined. This specific assessment was performed using 10 trials; 1 SUT was used for each trial and respective sensor dot. The selected concentrations (listed in Table 1) represent the finalized concentrations used in the 10 trial challenges of the MOSA GEN1 (Figures 2 and 3).

For the test matrix performed with GB, the repeatability trials (data shown in Appendix A.4) revealed that the MOSA GEN1 produced repeatable detection results. The repeatability assessment of CG illustrated a similar MOSA GEN1 performance (Appendix A.5). The data are arranged first by table number, beginning with Table 3, and correspond with GUI graphs (response representation) in Appendix A. As an example, from Table 3, the GUI graphs for the threat compound are shown in Appendix A.1.1. Beginning in August 2020, each trial alarm status was reported as yes, no, or not applicable, based on MOSA GEN1 performance. At the end of the challenge, the dot was removed, and the color was documented by still-image photography and assigned a figure number when applicable.

5.2 Conclusion

For all chemicals tested, the specific dot for the threat compound performed well. In some dot cases, slight variations existed; however, they were well within experimental error between similar dots. Modifications were made to the assessment protocol to resolve contamination conflicts and improve reproducibility, the MOSA GEN1 system configuration, and construction materials.

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2. Buchanan, J.H.; Buettner, L.C.; Butrow, A.B.; Tevault, D.E. *Vapor Pressure of VX*; ECBC-TR-068; U.S. Army Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD, 1999; UNCLASSIFIED Report (ADA371297).

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4. Pardoe, I.; Crouse, K.; Sohrabi, A.; Emge, D.; Ellzy, M. *Evaluation of Intelligent Optical System (IOS) Colorimetric-Based Organophosphate Vapor Sensor: Part I*; DEVCOM CBC-TR-1744; U.S. Army Combat Capabilities Development Command Chemical Biological Center: Aberdeen Proving Ground, MD, 2022; UNCLASSIFIED Report.

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ACRONYMS AND ABBREVIATIONS

APG	Aberdeen Proving Ground
CAPP	Chemical Analysis and Physical Properties
CBRNE	chemical, biological, radiological, nuclear, and explosives
CG	phosgene
CRADA	Cooperative Research and Development Agreement
CSIRP	CBRNE Sensor Integration onto Robotic Platforms
DEMPS	*O,O'*-diethyl methylphosphonothionate
DEVCOM CBC	U.S. Army Combat Capabilities Development Command Chemical Biological Center
FID	flame ionization detection
FPD	flame photometric detection
GB	sarin
GC	gas chromatography
GD	soman
GEN1	Generation 1
GUI	graphical user interface
HD	mustard
IOS	Intelligent Optical Systems
LED	light-emitting diode
LOD	limit of detection
MFC	mass flow controller
MOSA	multi-analyte optical sensor array
QA/QC	quality assurance and control
RH	relative humidity
sccm	standard cubic centimeters per minute
SUT	system under test
TDU	thermal desorption unit
UAS	unmanned aerial system
USB	universal serial bus
VX	*O*-ethyl-*S*-(2-diisopropylaminoethyl) methyl phosphonothiolate
WPL	workplace exposure level

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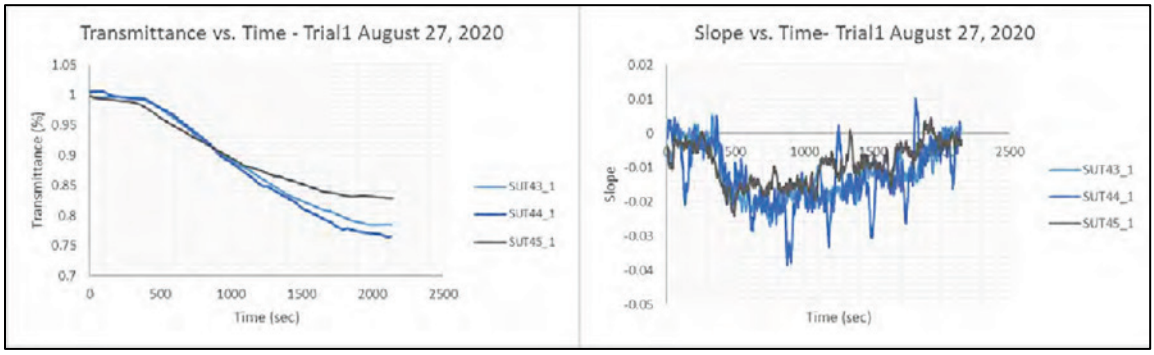
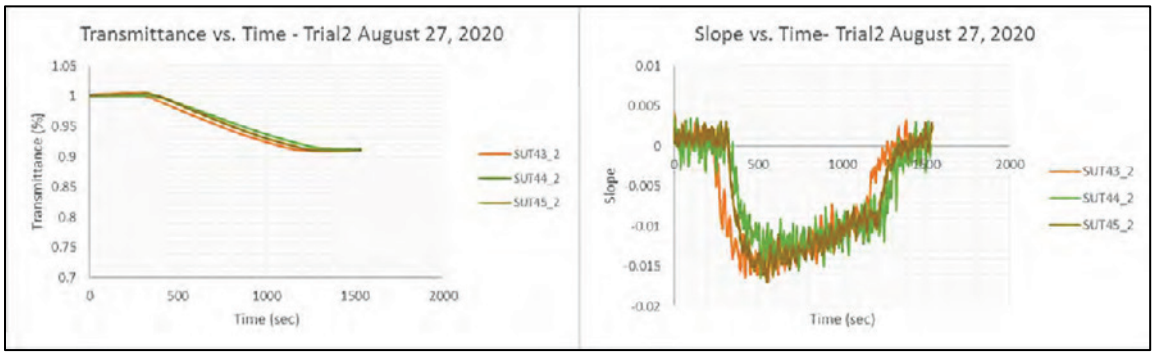
APPENDIX A:
GRAPHICAL USER INTERFACE (GUI) GRAPHS

A.1 Test Performed with All Three Systems Under Test (SUTs) in Tandem (Tables 3 and 4 in the Main Report)

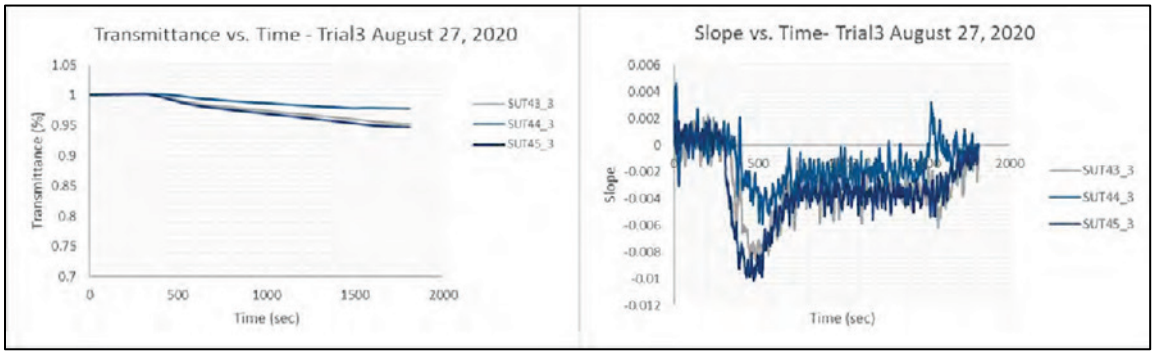
A.1.1 Trial 1: 27 August 2020 (Table 3)



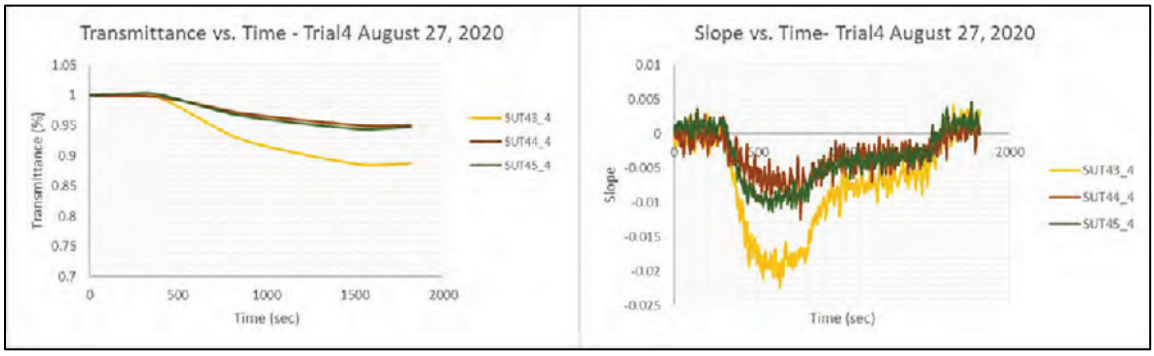
A.1.2 Trial 2: 27 August 2020 (Table 3)



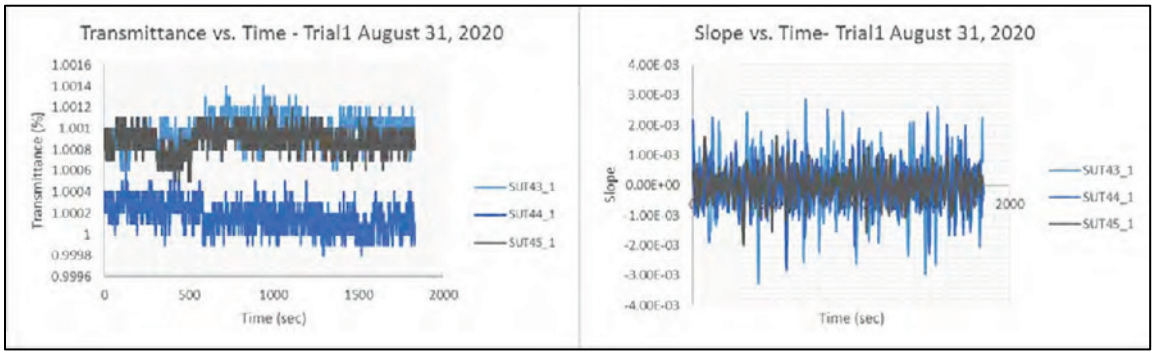
A.1.3 Trial 3: 27 August 2020 (Table 3)



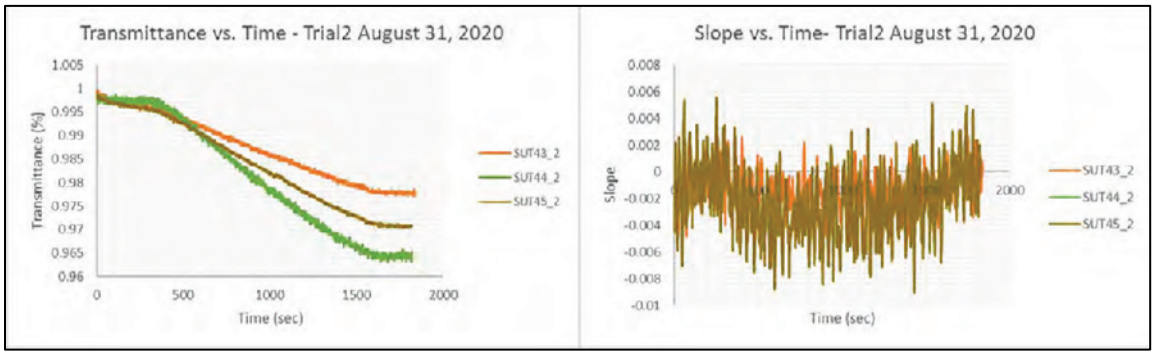
A.1.4 Trial 4: 27 August 2020 (Table 3)



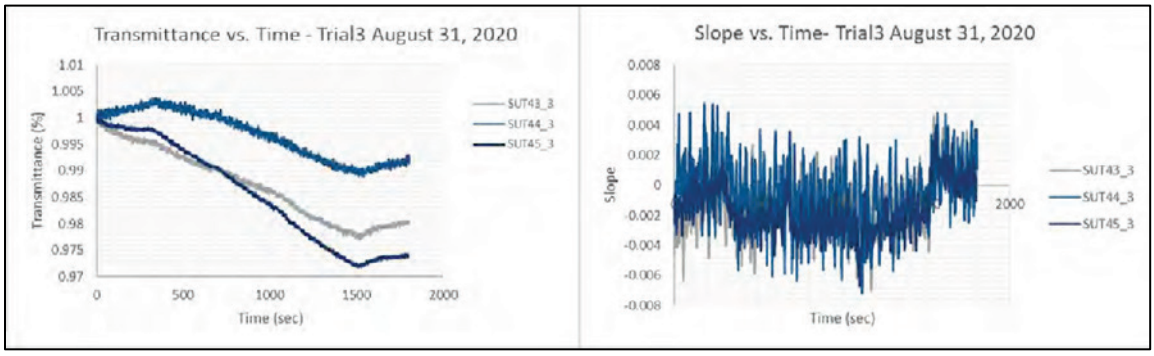
A.1.5 Trial 1: 31 August 2020 (Table 3)



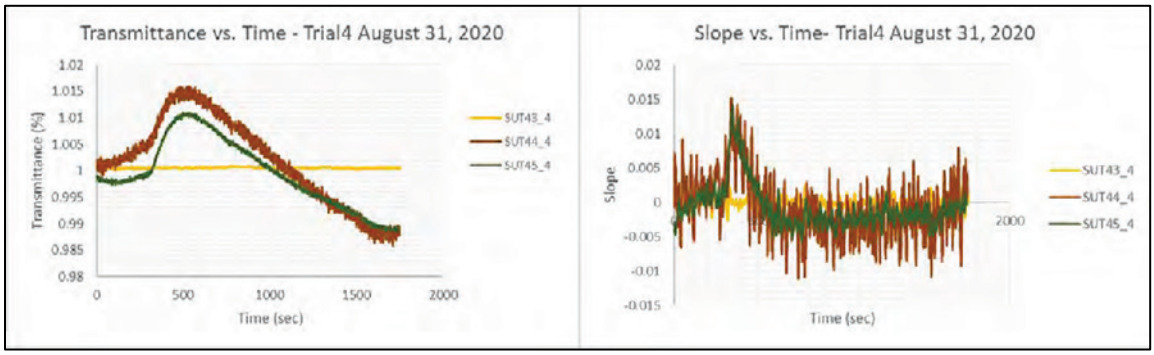
A.1.6 Trial 2: 31 August 2020 (Table 3)



A.1.7 Trial 3: 31 August 2020 (Table 3)

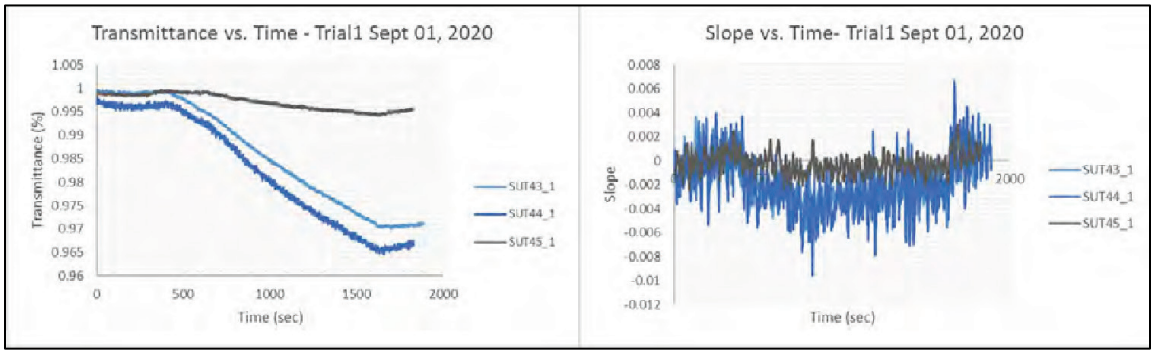


A.1.8 Trial 4: 31 August 2020 (Table 3)*

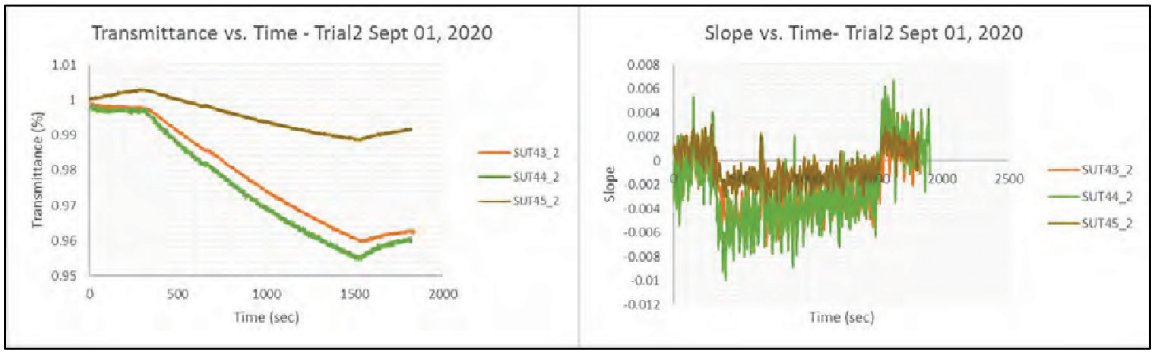


*Dot 5 (SUT43) fell off.

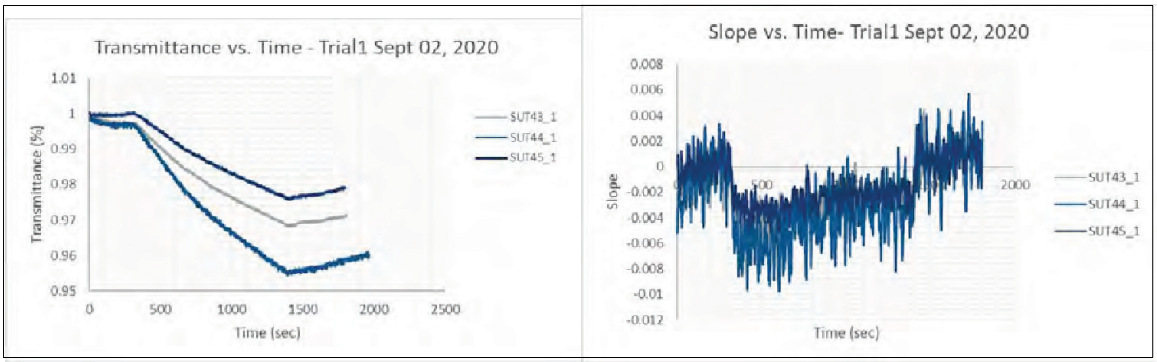
A.1.9 Trial 1: 1 September 2020 (Table 3)



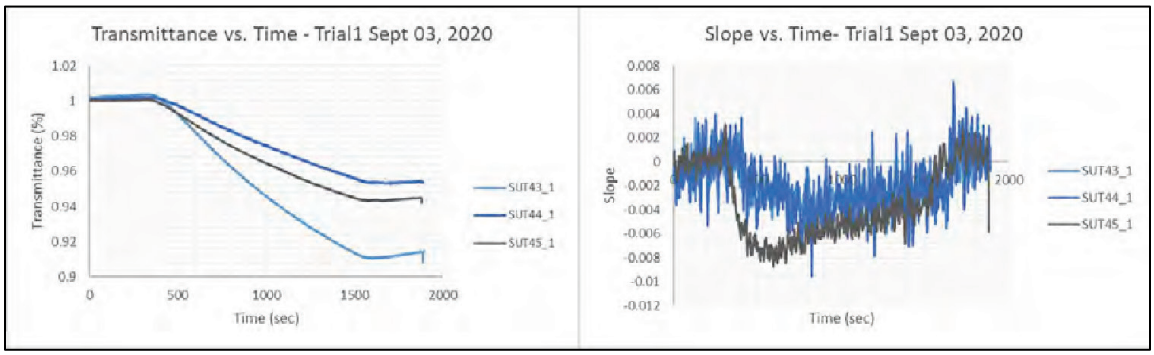
A.1.10 Trial 2: 1 September 2020 (Table 3)



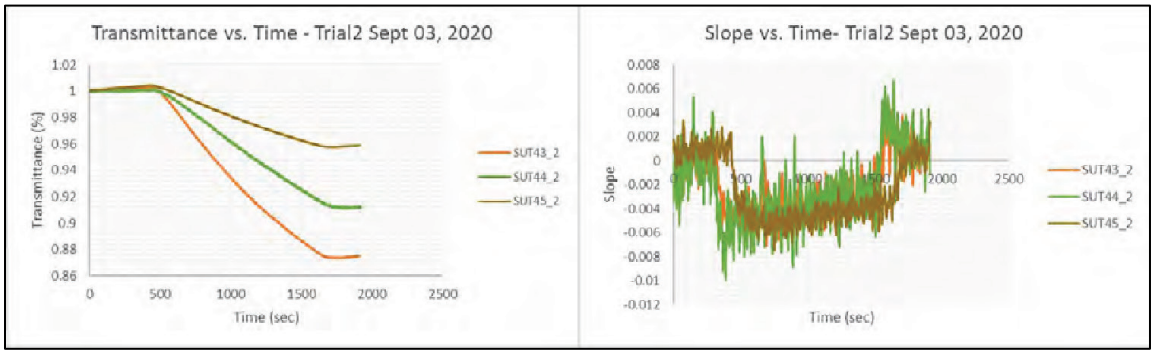
A.1.11 Trial 1: 2 September 2020 (Table 3)



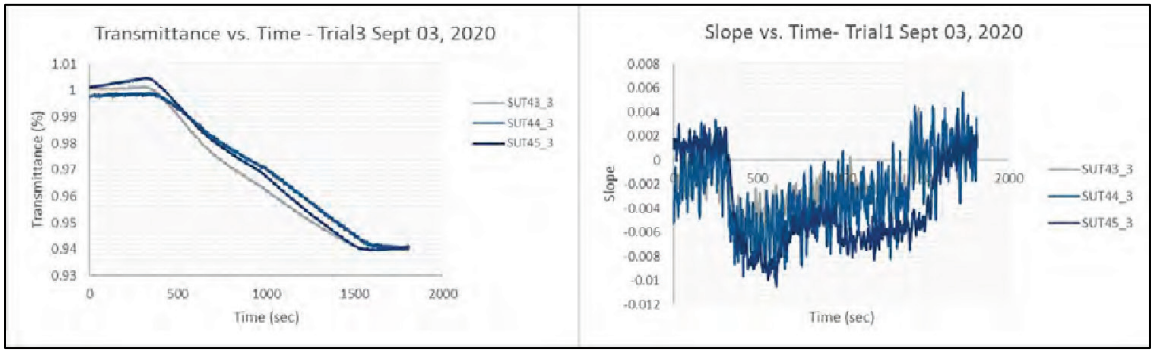
A.1.12 Trial 1: 3 September 2020 (Table 3)



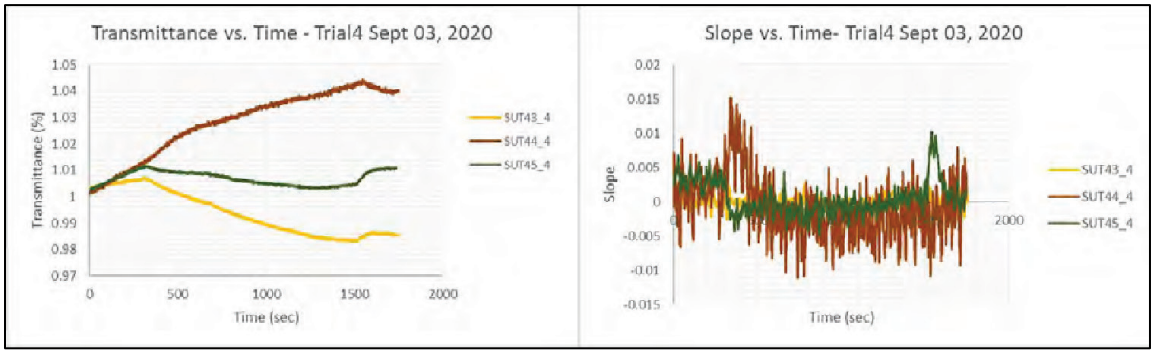
A.1.13 Trial 2: 3 September 2020 (Table 3)



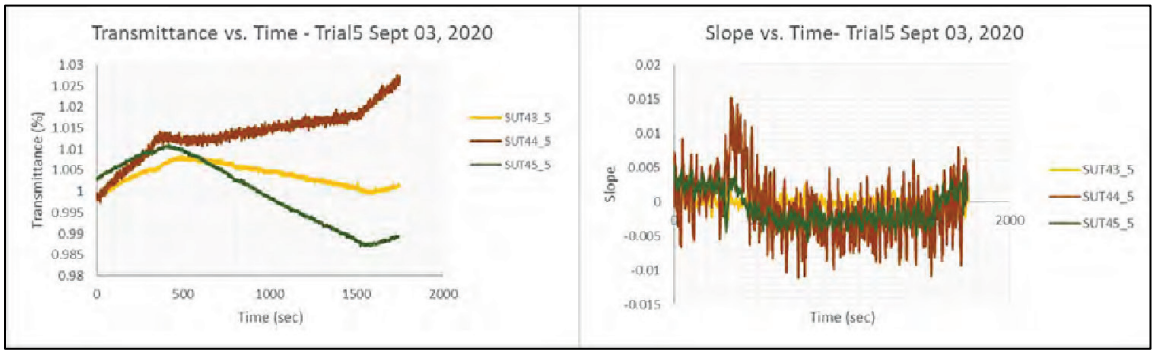
A.1.14 Trial 3: 3 September 2020 (Table 3)



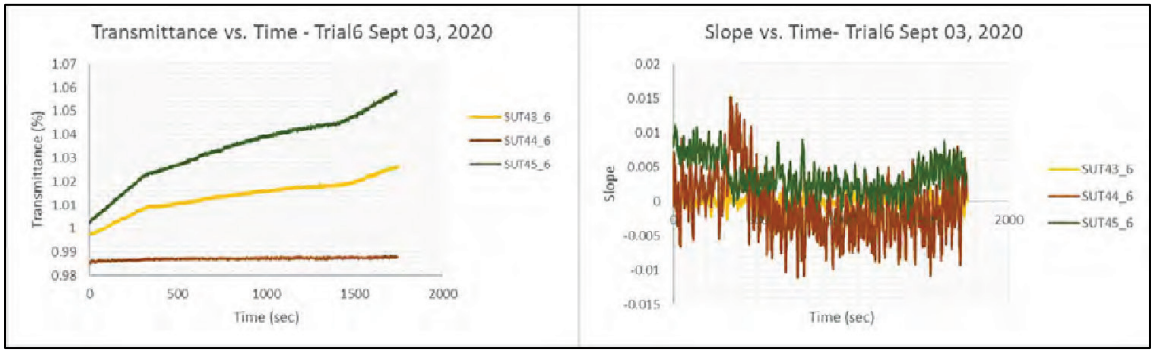
A.1.15 Trial 4: 3 September 2020 (Table 3)



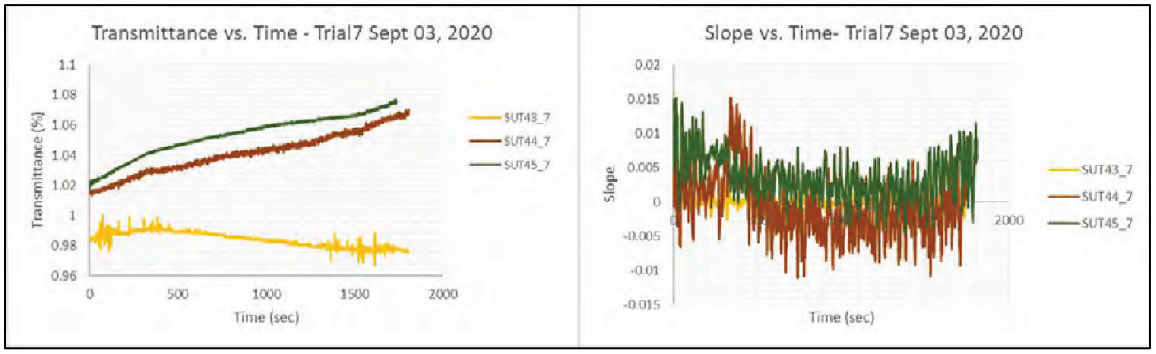
A.1.16 Trial 5: 3 September 2020 (Table 3)



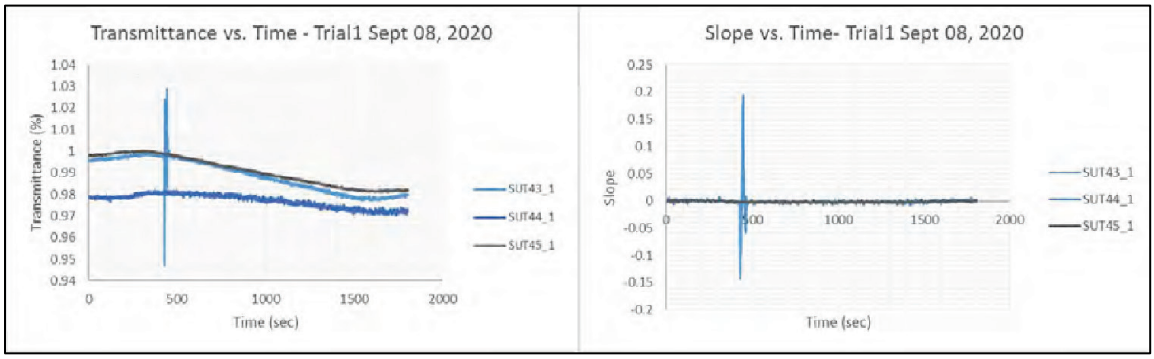
A.1.17 Trial 6: 3 September 2020 (Table 3)



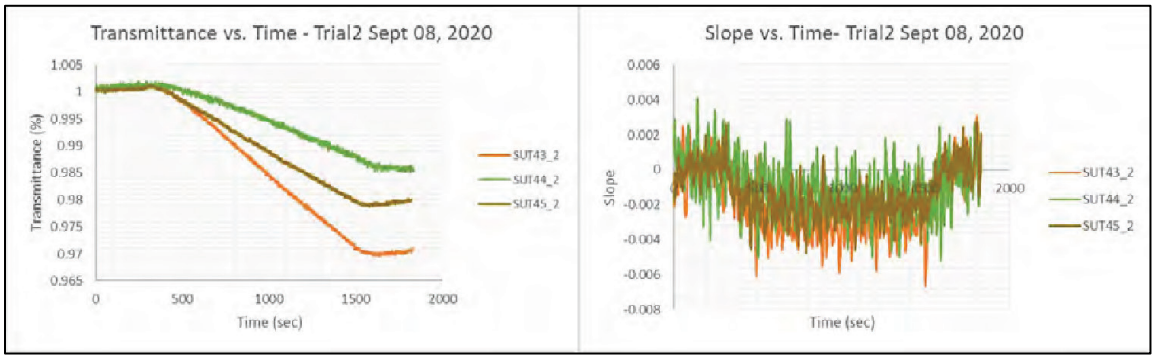
A.1.18 Trial 7: 3 September 2020 (Table 3)



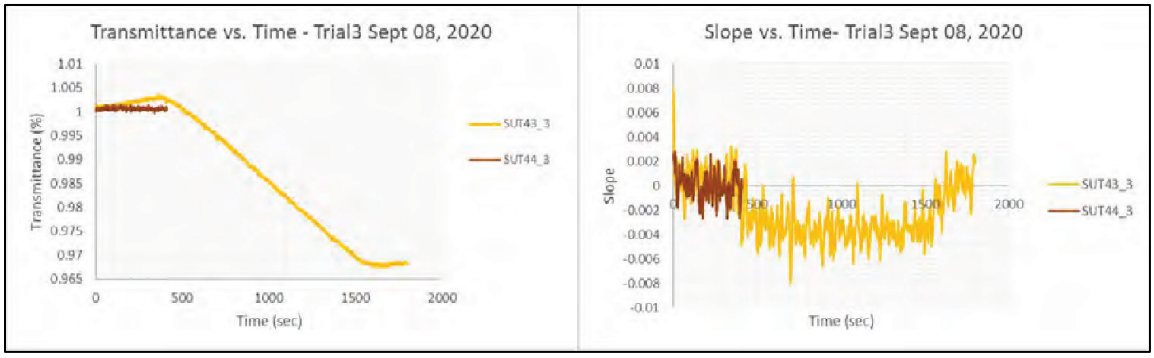
A.1.19 Trial 1: 8 September 2020 (Table 4)



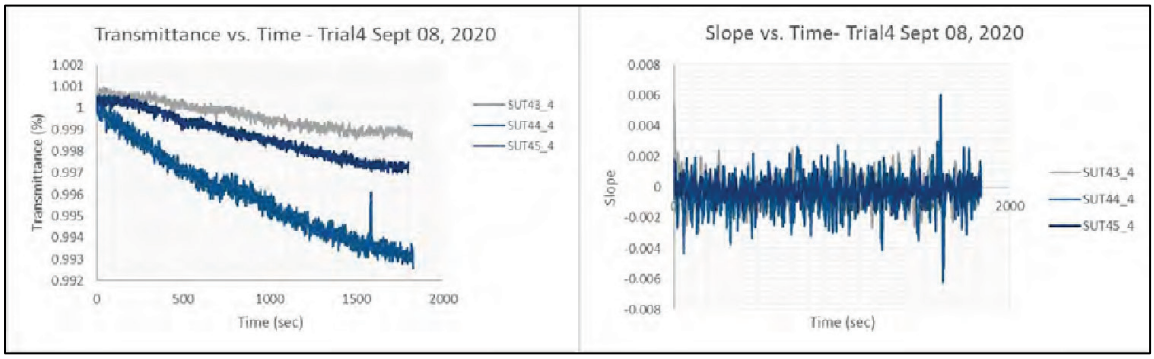
A.1.20 Trial 2: 8 September 2020 (Table 4)



A.1.21 Trial 3: 8 September 2020 (Table 4)

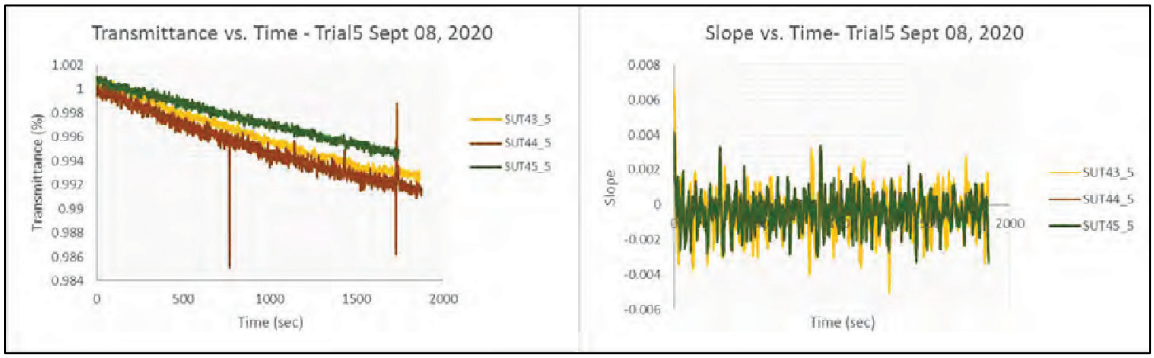


A.1.22 Trial 4: 8 September 2020 (Table 4)*



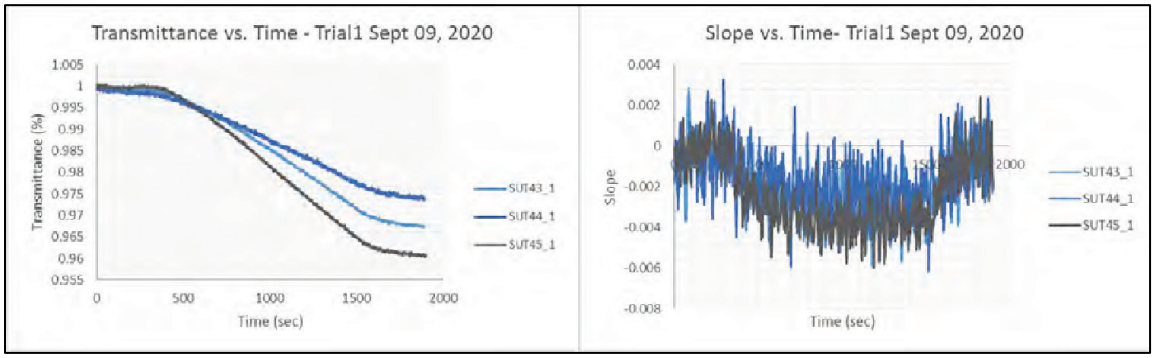
*Intelligent Optical Systems (IOS) said the new (bad) batch was due to a heat wave in California.

A.1.23 Trial 5: 8 September 2020 (Table 4)*

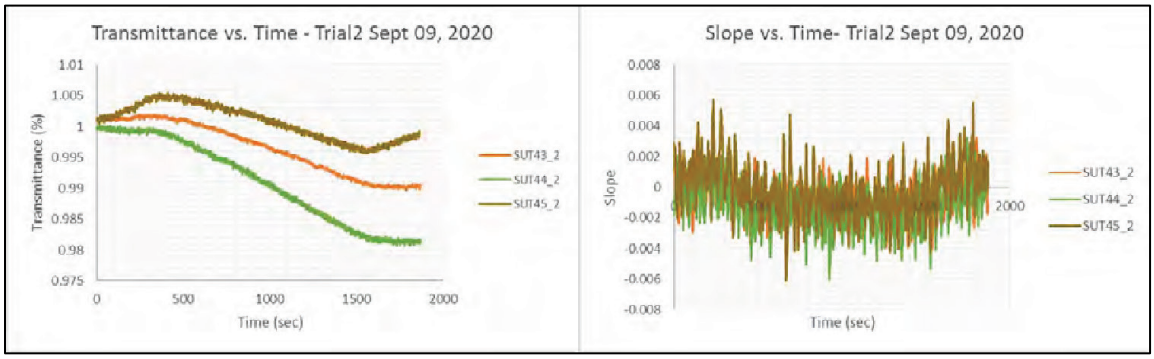


*IOS said the new (bad) batch was due to a heat wave in California.

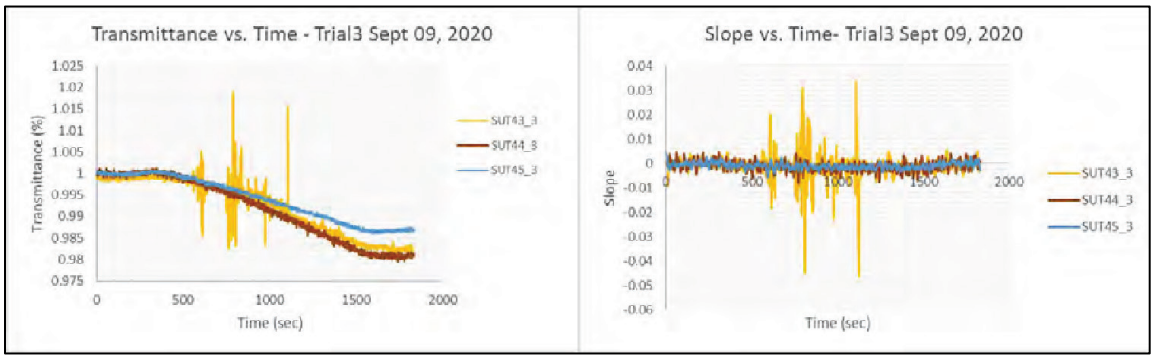
A.1.24 Trial 1: 9 September 2020 (Table 4)



A.1.25 Trial 2: 9 September 2020 (Table 4)

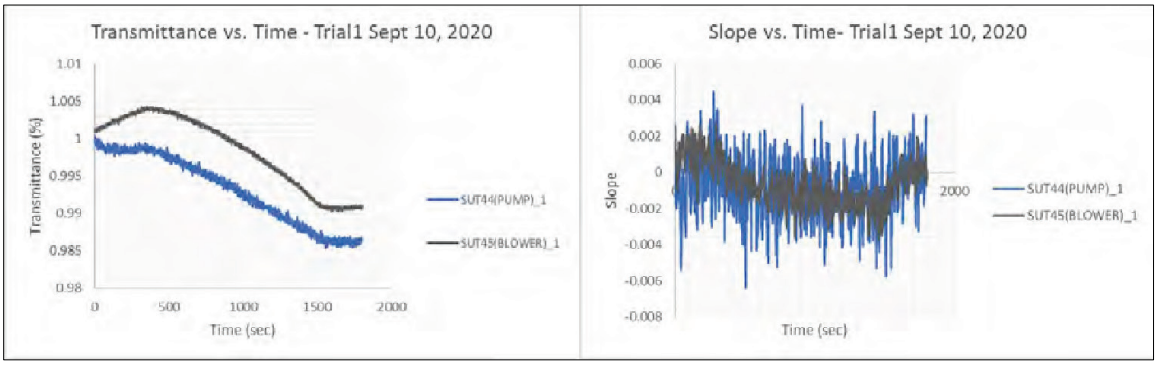


A.1.26 Trial 3: 9 September 2020 (Table 4)*



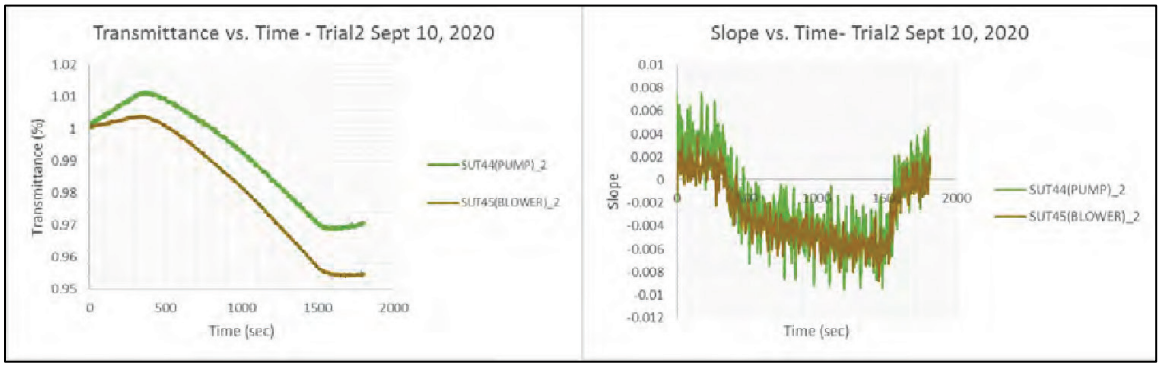
*IOS suspected the power/voltage regulator.

A.1.27 Trial 1: 10 September 2020 (Table 4)*†



*SUT44, pump; SUT45, blower.
†Used Parafilm wrap on the pump.

A.1.28 Trial 2: 10 September 2020 (Table 4)*†‡

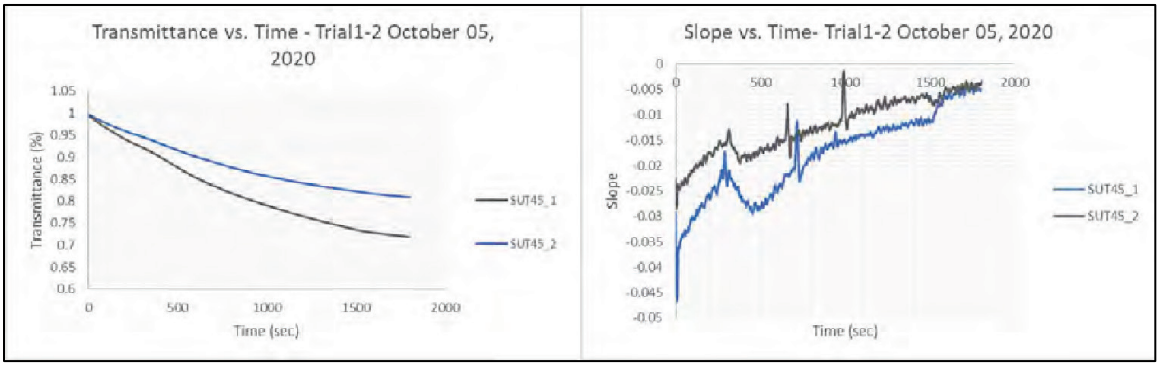


*SUT44, pump; SUT45, blower.
†Used Parafilm wrap on the pump.
‡IOS: "The pump was envisioned to solve the flow rate issue."

A.2 Performed with Only One SUT Placed in a Sealed 2 L Chamber (Tables 5–8)

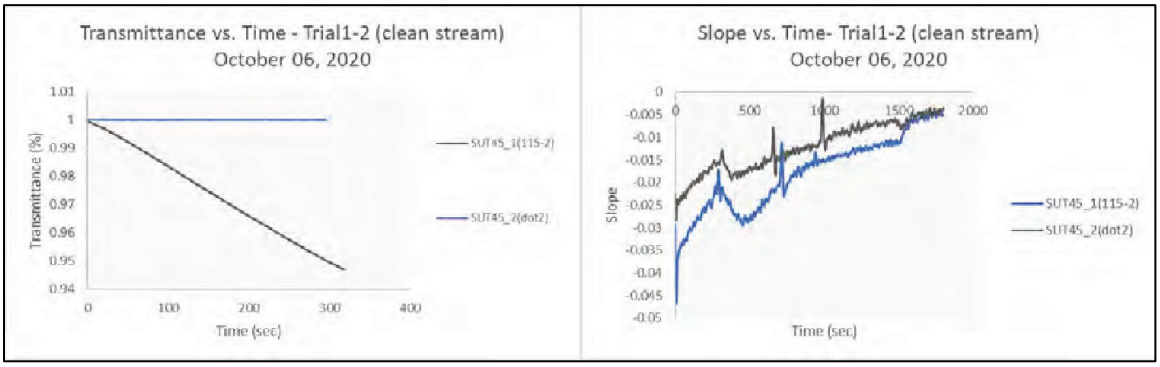
A.2.1 Trial 1-2: 5 October 2020, Direct-Flow/Pump Housing SUT45 (Table 5)

Cross-contamination issue.



A.2.2 Trial 1-2: 6 October 2020, Direct-Flow/Pump Housing SUT45 Clean Stream Only (Table 5)

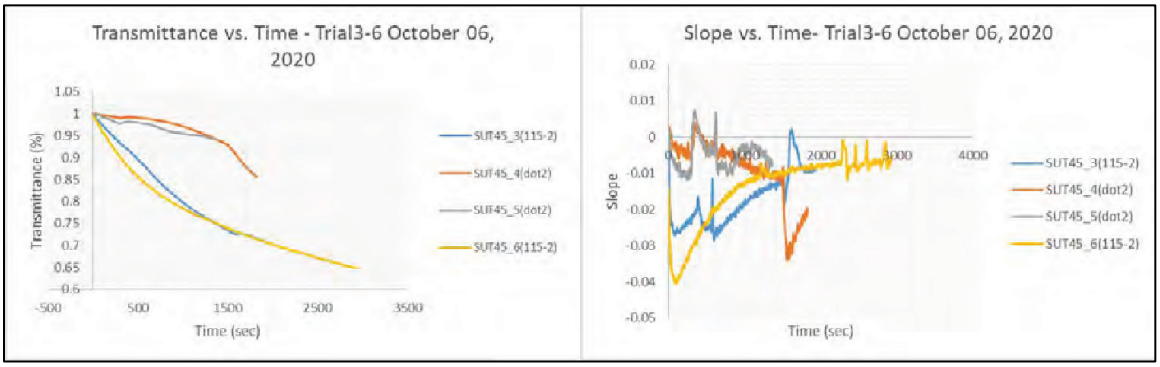
Cross-contamination issue; GUI alarmed throughout the run.



A.2.3 Trial 3-6: 6 October 2020, Direct-Flow/Pump Housing SUT45 (Table 5)

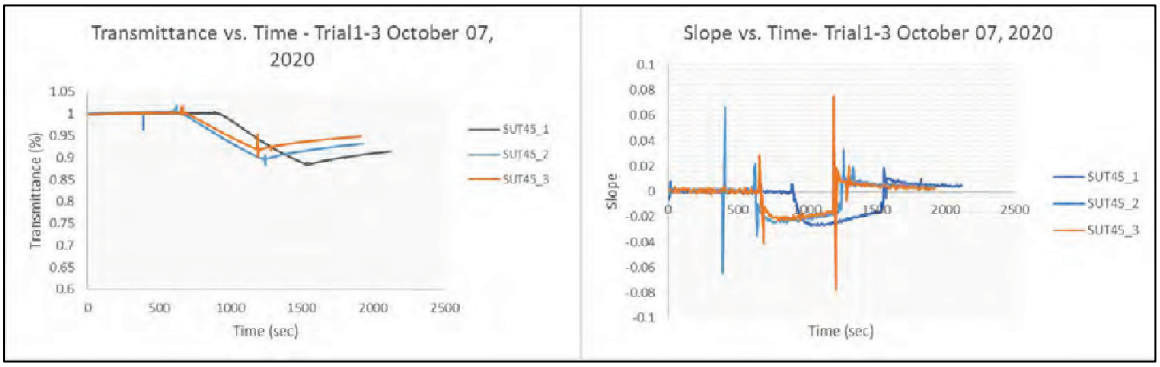
Cross contamination issue.

Trial 3: GUI alarmed red at start of run during the clean stream.
Trial 4: Cross-contamination issue. GUI alarmed red at 2 min, 15 s of run during the clean stream and off at 2 m, 50 s. This occurred continuously during the whole run.
Trial 5: GUI alarmed red at 30 s of run during the clean streamand off at 8 m, 19 s. This occurred continuously during the whole run. Test was stopped prematurely.
Trial 6: Cross-contamination issue. GUI alarmed red at start of run during the clean stream.

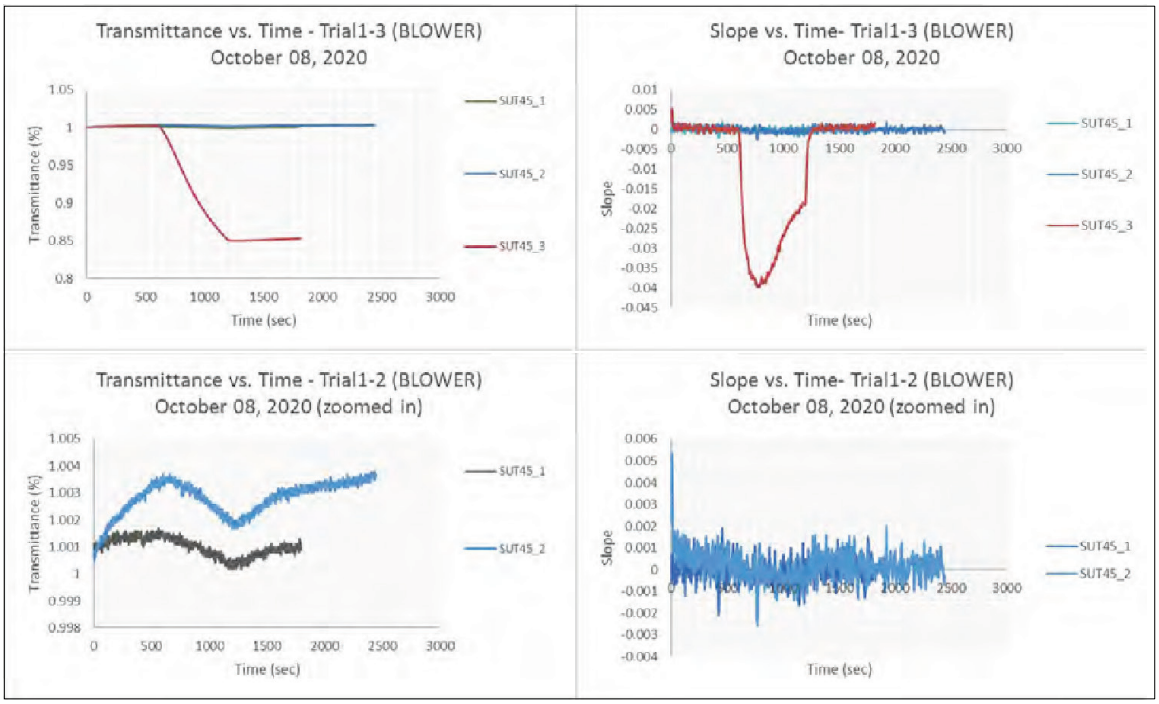


A.2.4 Trials 1–3: 7 October 2020; Direct-Flow/Pump Housing SUT45 (Table 5)

Direct-flow housing baked out overnight at 80 °C then cooled to room temperature. Started using clean and analyte line separately. GUI alarmed red at start of sarin (GB) (only) line connection and continued to alarm until the clean stream (only) line was reconnected.

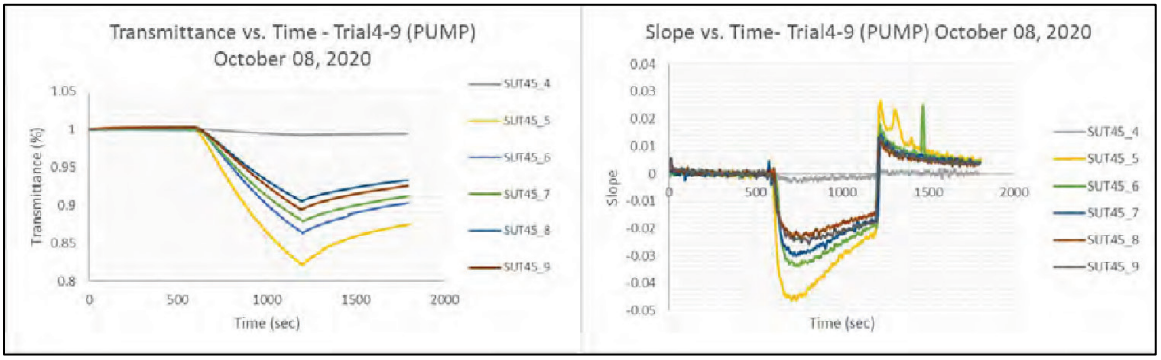


A.2.5 Trials 1–3: 8 October 2020; Blower Fan Housing SUT45 (Table 6)



A.2.6 Trials 4–9: 8 October 2020; Direct-Flow/Pump Housing SUT45 (Table 6)

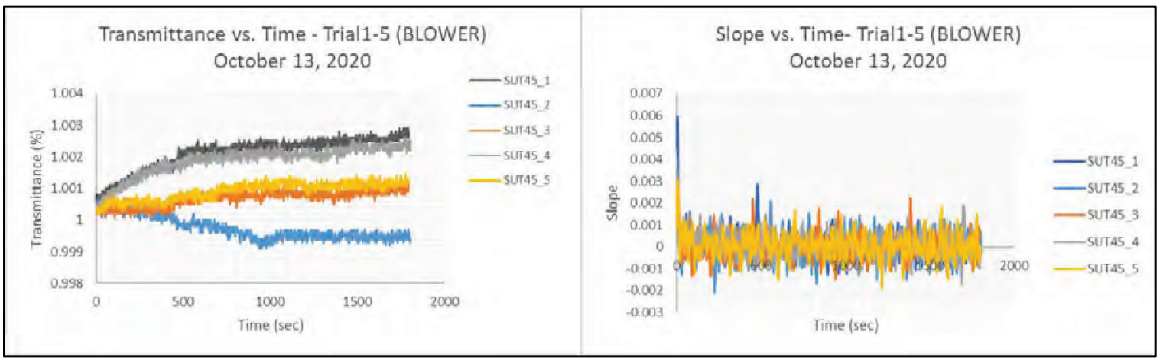
N_2 flow was directed on the housing before the dot was applied.



A.2.7 Trials 1–5: 13 October 2020; Blower Housing SUT45 (Table 6)

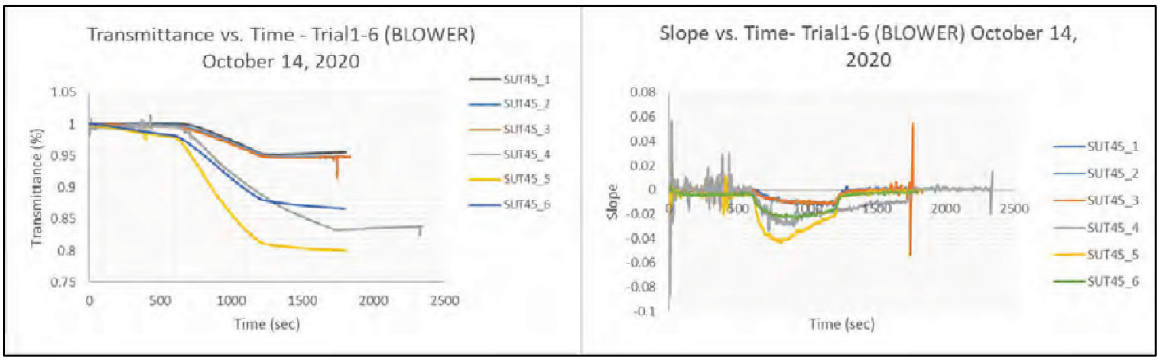
GUI did not alarm throughout the run. Transmission response was not observed. No color change.

Trial 4: Increase GB flow, 0.085 mg/m^3 (started collecting from chamber).

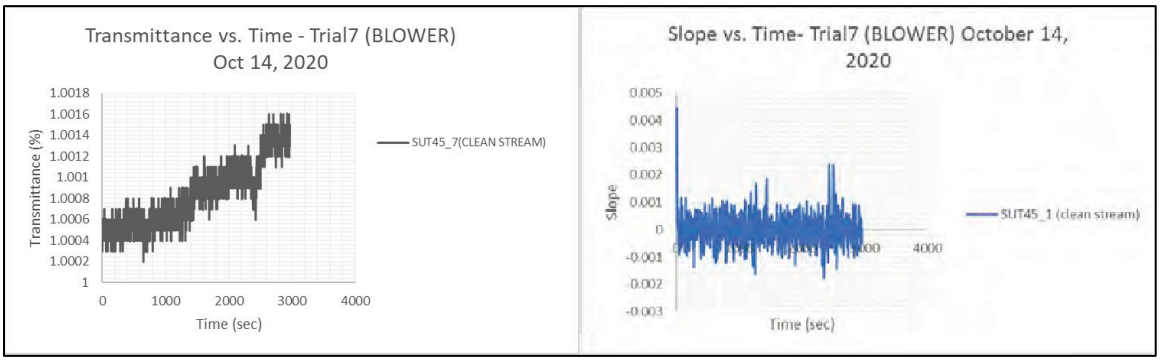


A.2.8 Trials 1–6: 14 October 2020; Blower Housing SUT45 (Table 7)

Trials 4–6: GUI alarmed off and on during clean stream and alarmed longer during final clean stream with each trial run. Color change observed. Increased GB flow.

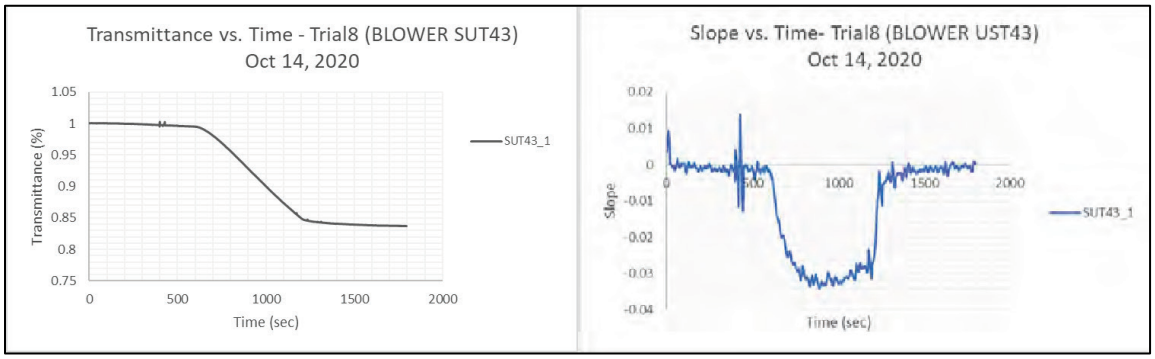


A.2.9 Trial 7: Clean Stream Only (Table 7)



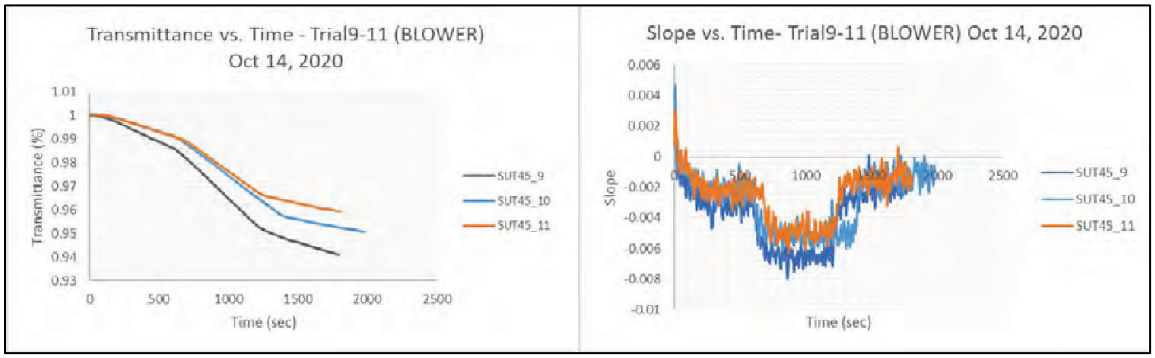
A.2.10 Trial 8: 14 October 2020; Blower Housing SUT43 (Table 7)

Color change observed. It was a chamber-contamination issue.



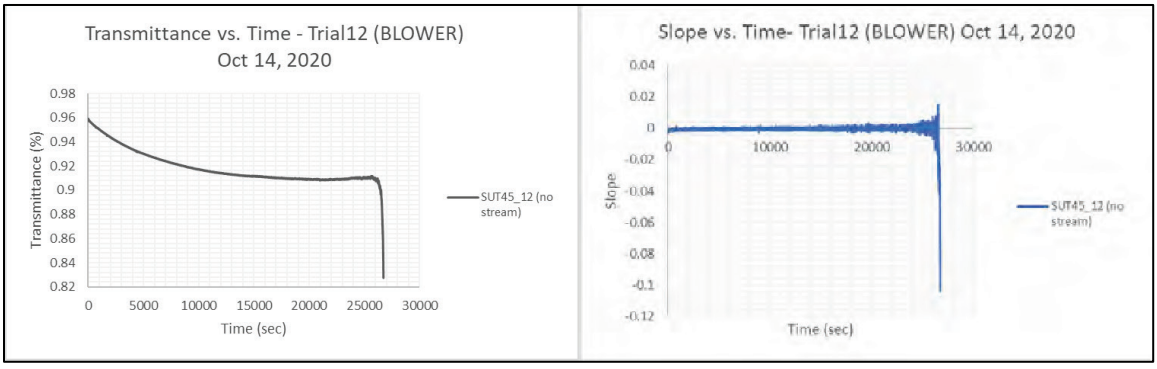
A.2.11 Trials 9–11: 14 October 2020; Blower Housing SUT45 (Table 8)

Switched to battery pack for SUT45. It was a chamber-contamination issue. GUI was not alarming or alarmed less during clean stream because the concentration was lower.



A.2.12 Trial 12: 14 October 2020 No Stream (Table 8)

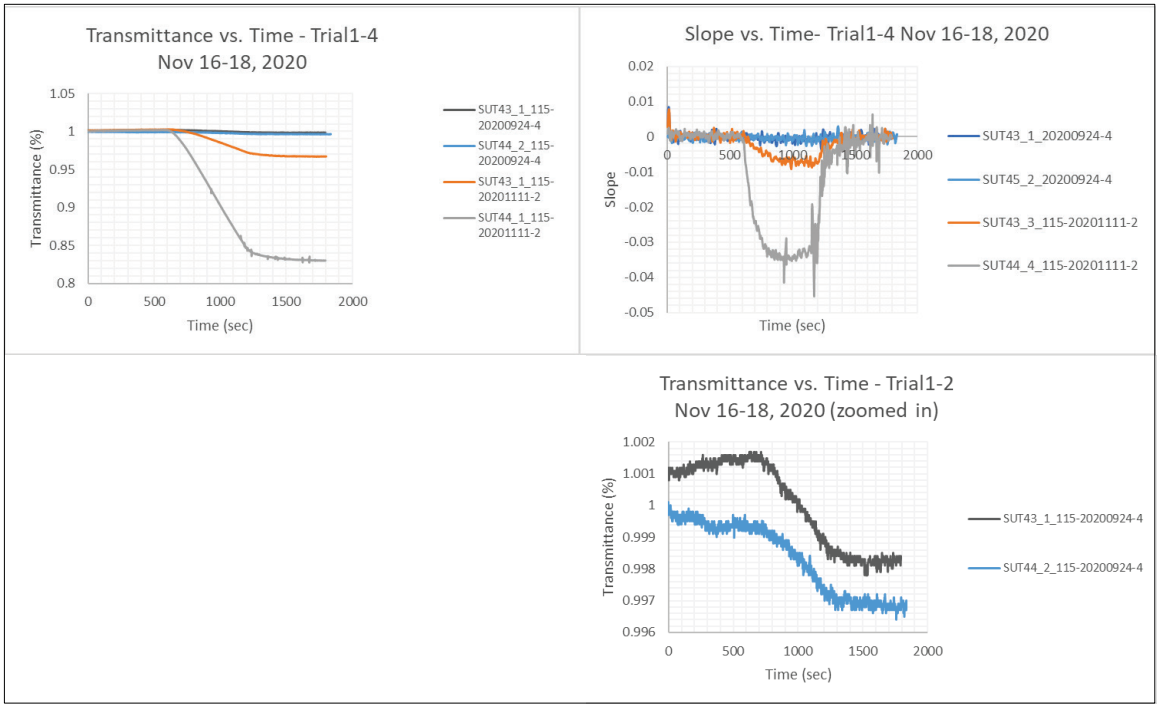
19 October 2020: D. Emge and K. Crouse started performing contamination test (refer to Appendix C). This led to the performance of a clearance of the test chamber and housing before each trial.



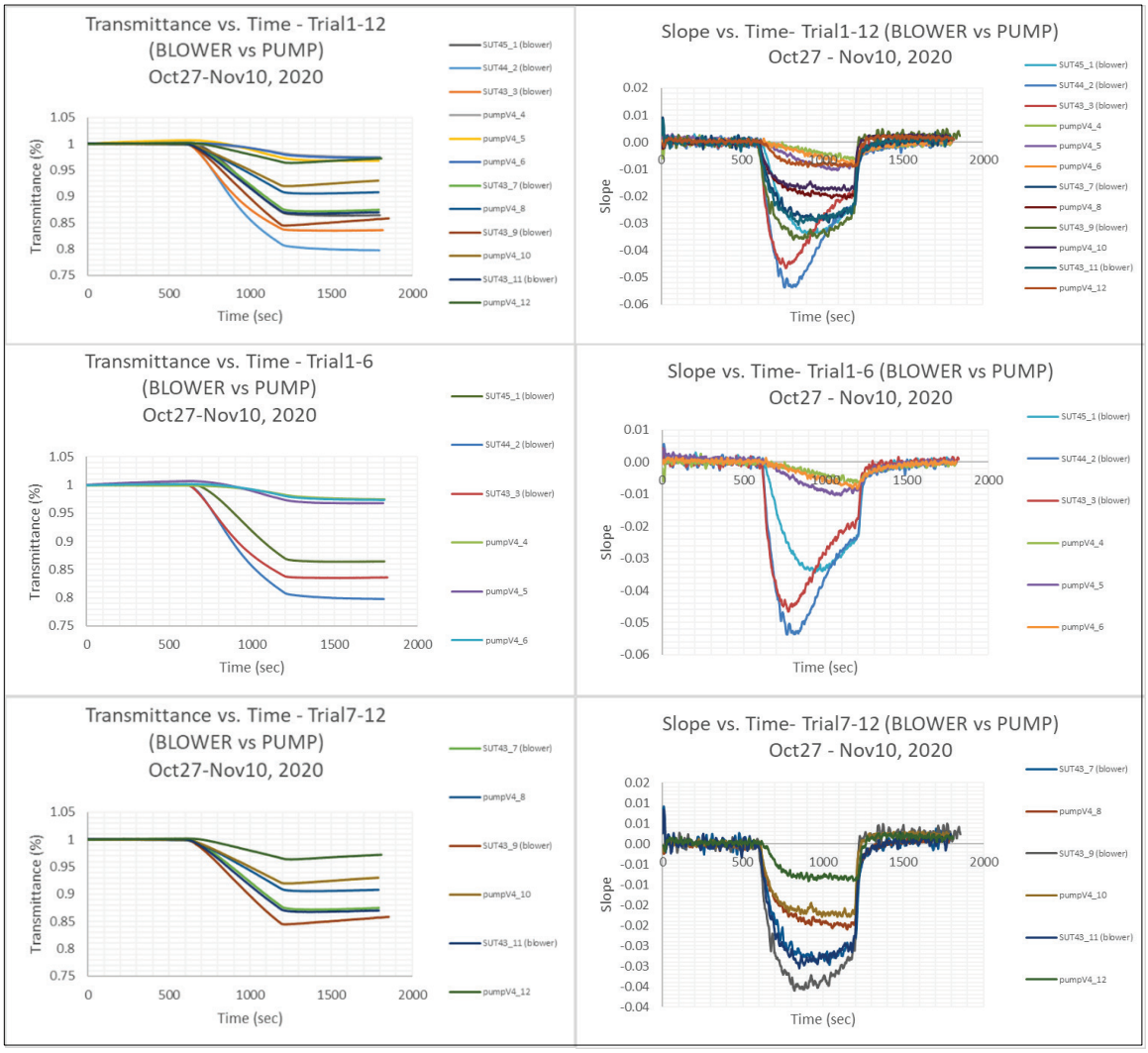
A.2.13 Trials 1–4: 16–17 November 2020; SUT43, 44, 43, 44 respectively (Table 8)

A couple more tests were performed to test dots from two batches: G155-20200924-4 and G115-20201111-2.

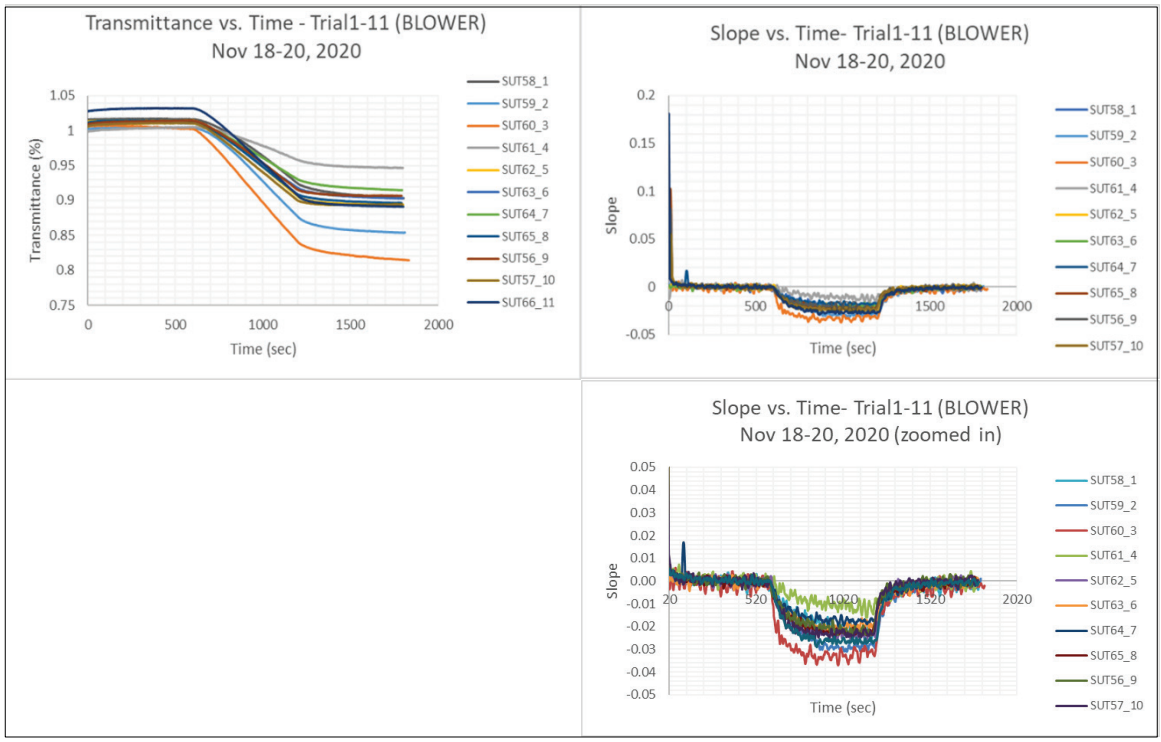
Trials 1–2: 16 November 2020; dots from batch G155-20200924-4.
Trials 3–4: 17–18 November 2020; dots from batch G115-20201111-2.



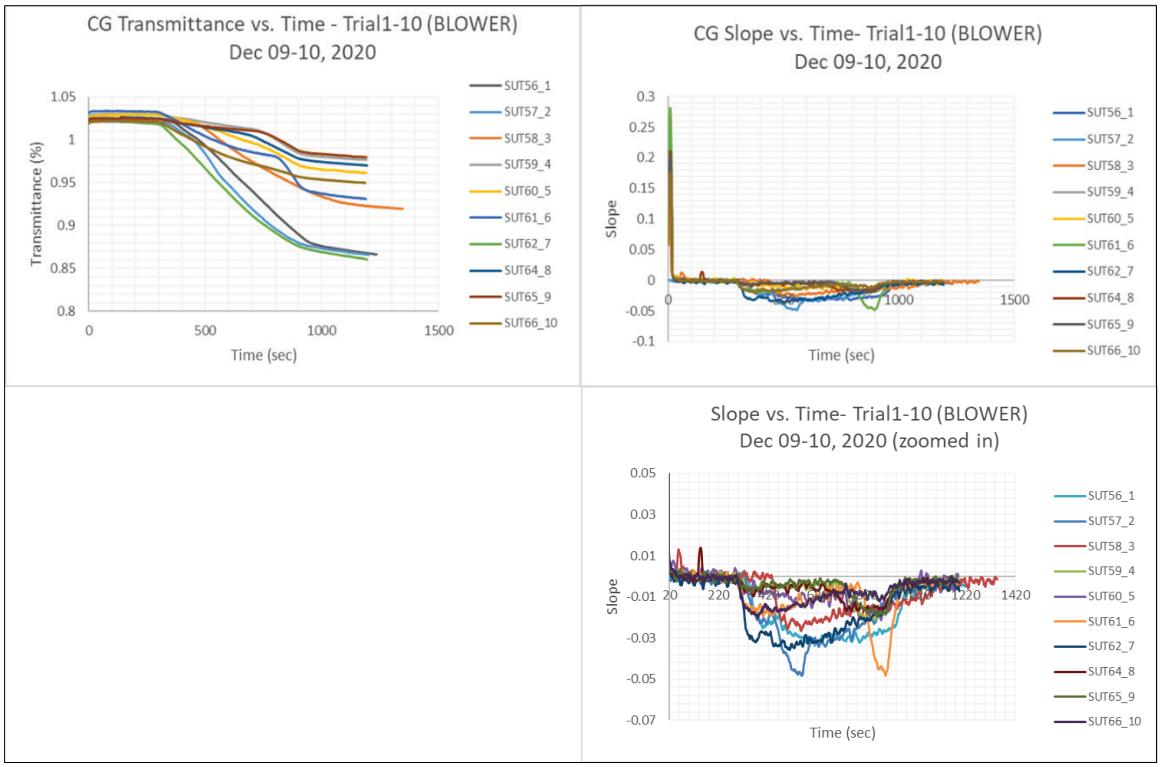
A.3 GB Test Matrix: Concentrations 0.044–0.250 mg/m^3 (Table 9)



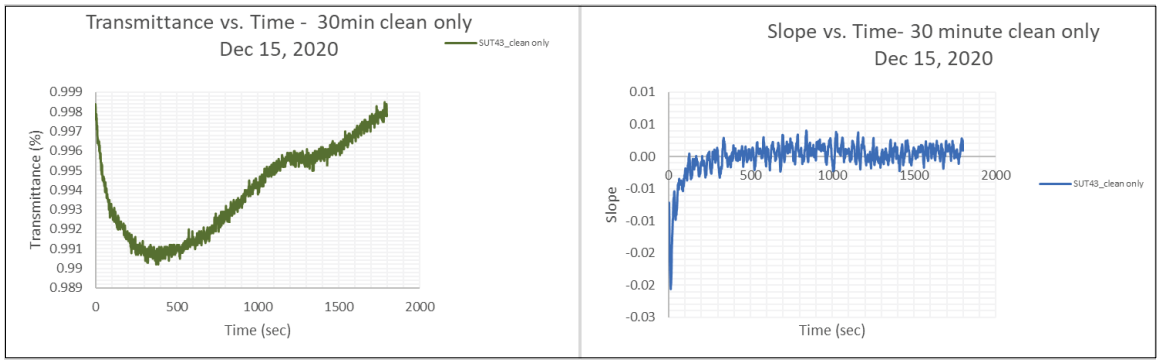
A.4 GB: 10 Trials at 0.147 ± 0.093 mg/m^3 (Table 10)



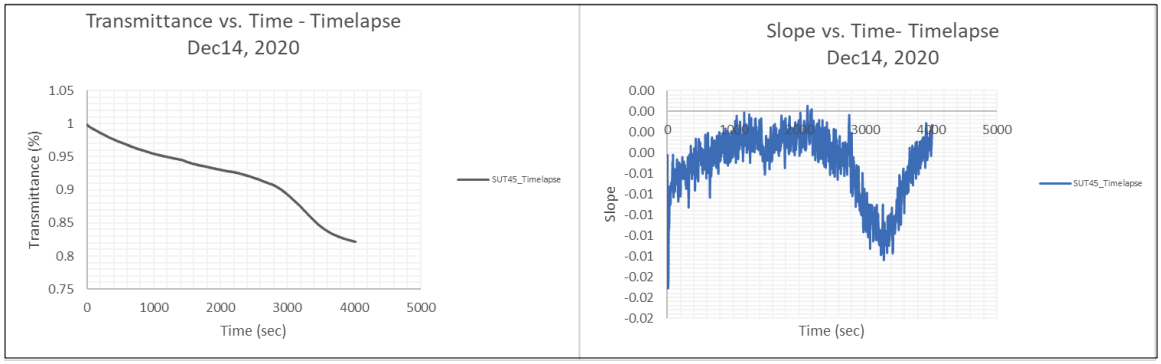
A.5 Phosgene (CG): 10 Trials at 0.0710 ppm (Table 11)



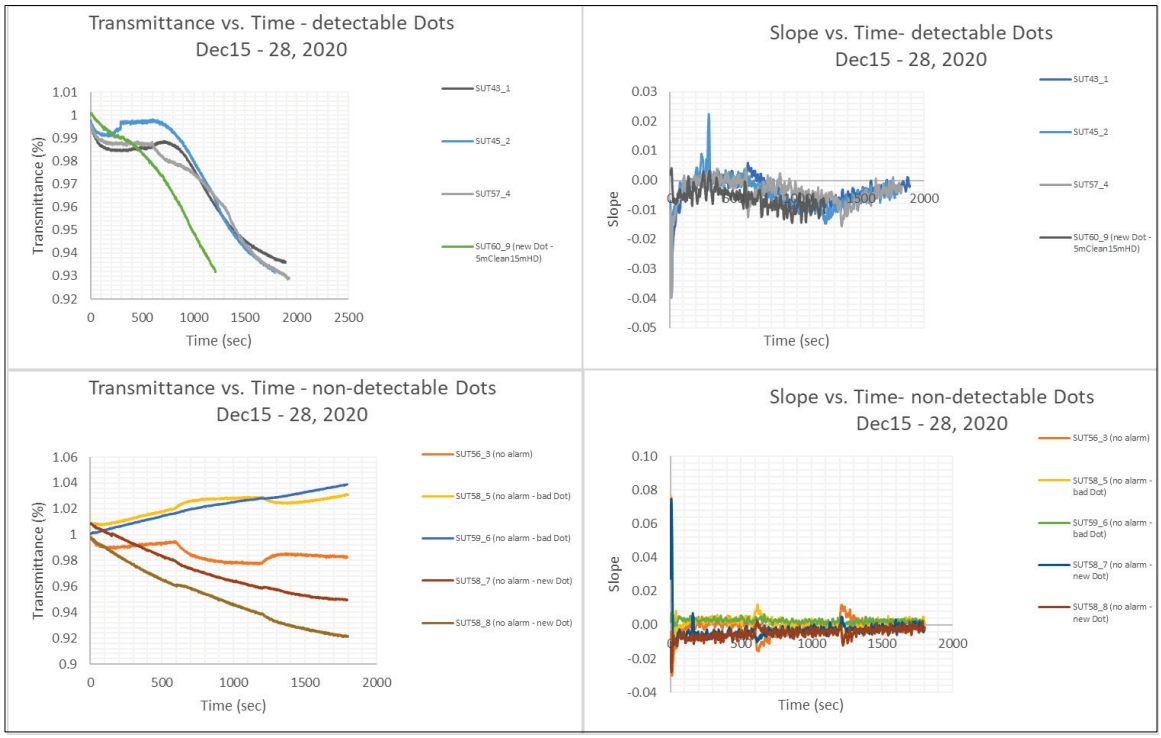
A.6 30 min Clean Stream over Mustard (HD) Dots (Section 4.3.3)



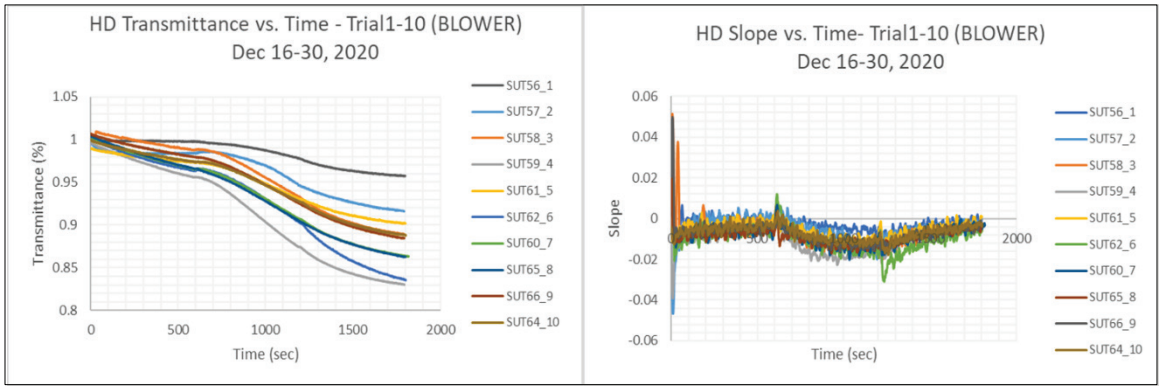
A.7 HD Time-Lapse Data for Various Concentrations: 0.0184, 0.1, 0.144, and ~1.72 mg/m^3; 10.18 min Clean/47 min HD/10 min Clean (Table 12)



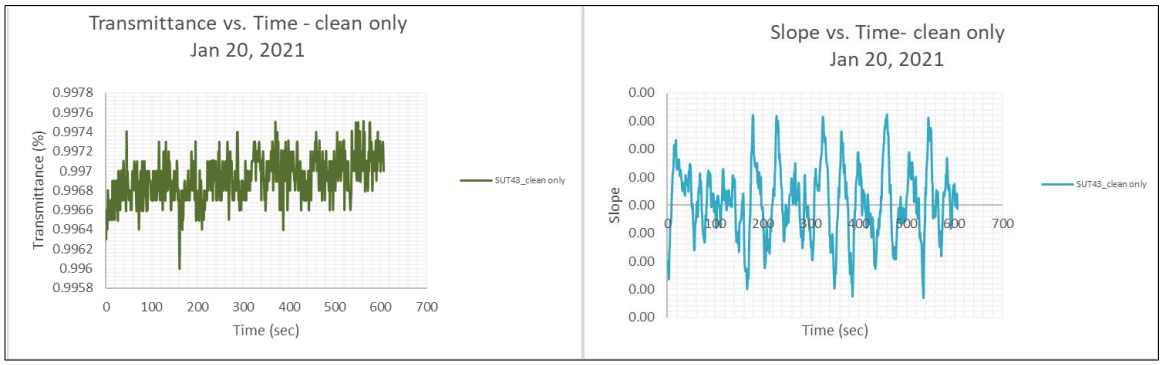
A.8 HD Test Matrix: Concentrations 0.03–1.3 mg/m^3 (0.13–1.3 mg/m^3 Detectable) (Table 13)



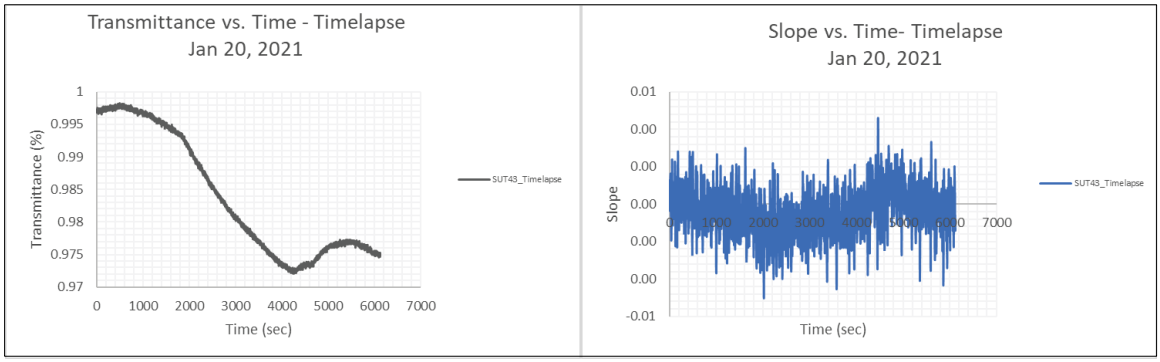
A.9 HD: 10 Trials at 0.854 ± 0.926 mg/m^3 (Table 14)



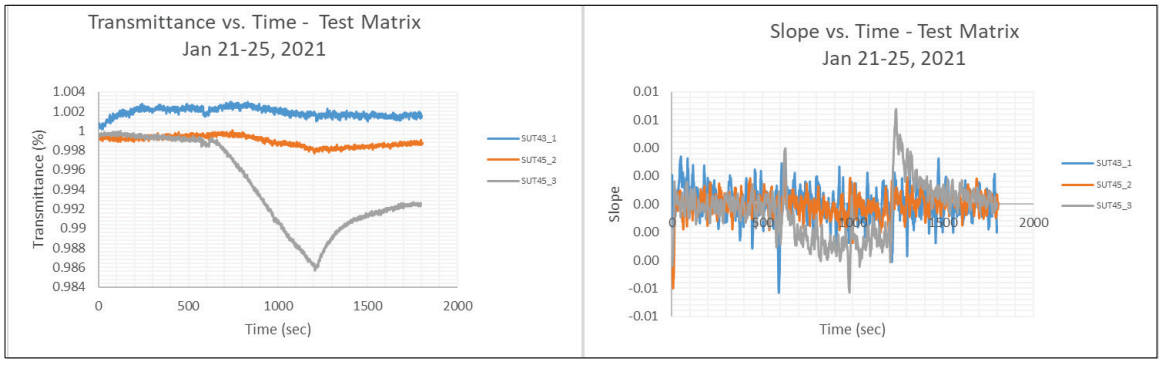
A.10 10 min Clean Stream over *O*-Ethyl-*S*-(2-diisopropylaminoethyl) Methyl Phosphonothiolate (VX) Dots (Section 4.4.2)



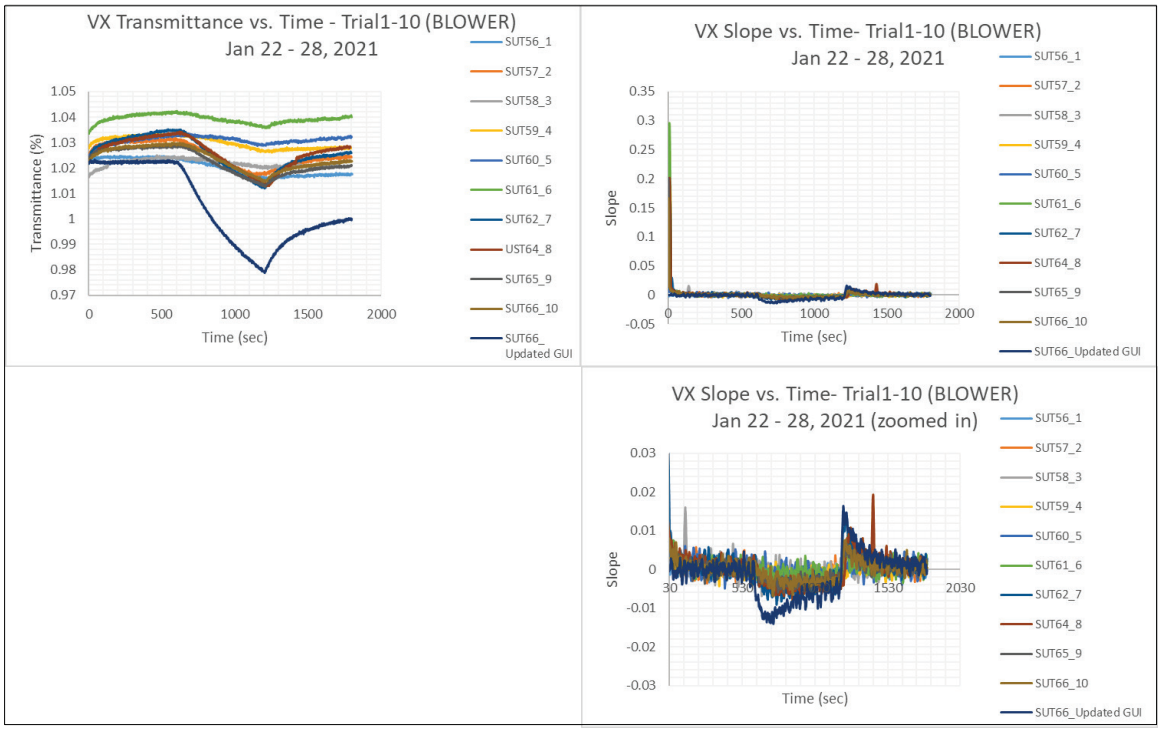
A.11 VX Time-Lapse Data for Various Concentrations: 0.00665-0.056 mg/m^3 (Table 15)



A.12 VX Test Matrix: Concentrations 0.005–0.028 mg/m^3 (Table 16)



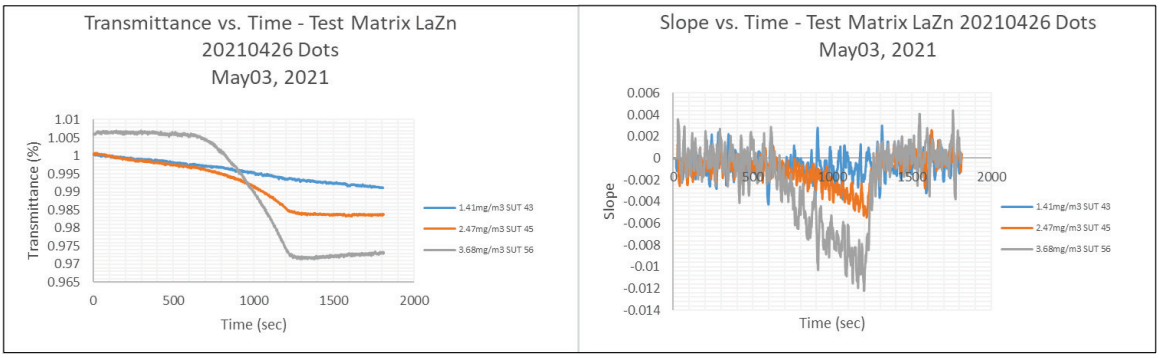
A.13 VX: 10 Trials at 0.0308 ± 0.0078 mg/m³ (Table 17)



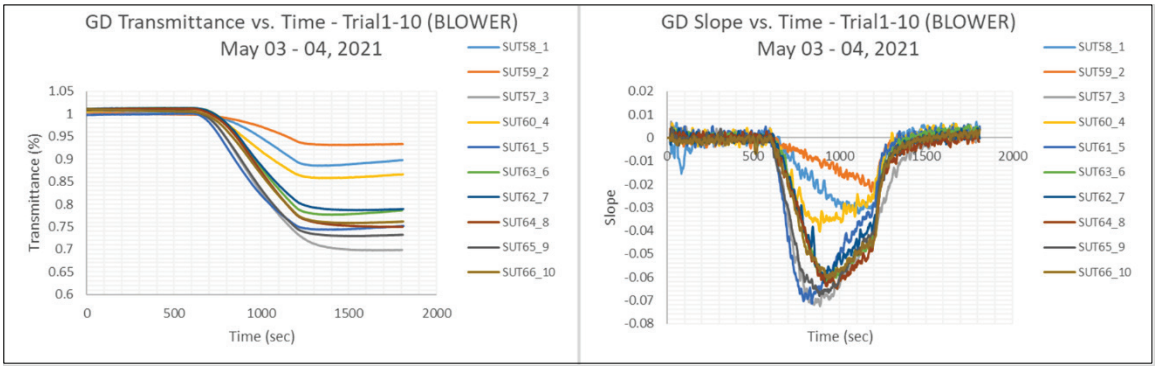
A.14 Soman (GD) Comparison of Three Dots: Average Concentration 12.18 mg/m³ (Table 19)

Dot 1 was 240La,150B; dot 2 was 240Eu,150B; and dot 3 was LaZn 20210426.

A.15 GD Test Matrix Concentrations: 1.41, 2.47, and 3.68 mg/m^3 (Table 20)



A.16 GD: 10 Trials at 4.17 ± 0.33 mg/m^3 (Table 21)



APPENDIX B:
TEST PLAN*

(202000519 MOSA GEN1 Work Plan Ver2.3.docx Appendix B)

MOSA Gen1 Test Plan
Prepared by: Ian Pardoe and Darren Emge

Introduction:

The purpose of this document is to outline the protocols for assessment of the MOSA Generation 1 (MOSA GEN1) prototypes constructed by Intelligent Optical Systems (IOS) to determine the limits and capabilities of the MOSA GEN1 to meet the needs of the CSIRP program. The intent of the CSIRP is to integrate small scale CBRN sensors onto/into robotics platforms with limited loss in performance. In this particular effort the goal is to assess the MOSA GEN1 chemical detector and to determine a baseline capability followed by a limited assessment of that capability while mounted to an unmanned aircraft system (UAS). Our guidelines for assessment will reflect these conditions.

- We are targeting an assessment reflecting a UAS flying through a cloud of disseminated chemical threat.
- We will start with an initial assumed exposure time of approximately 60 s to mimic the time for a UAS to travel through a disseminated cloud.
- The system under test (SUT) will have to sample from the ambient environment (i.e. there will be no active direction of the analyte flow directly onto the system).
- We assume continuous forward travel (parallel to the ground) of the UAS, so the air stream from which the SUT will sample will be actively flowing over the SUT so as to mimic this.
- Three SUTs are to be delivered and tested in tandem. All three SUTs will be placed into a sealed 2 L test chamber.
- The carrier air flow will be selected to ensure that given the test chamber volume and size of the opening through with the stream will pass out of the chamber that no backpressure of the stream will build up within the chamber (atmospheric pressure is maintained).
- The carrier air stream will be generated at room temperature (~20°C) and 50% relative humidity.
- Once both the baseline, non-moving, and on-the-move capabilities have been established it is recommended that more complex environmental factors be investigated.

Description of Test Articles: *Details of Sensor need to be incorporated once the prototype configuration is finalized.*

*This test plan is reproduced as it was originally written and has not been edited or changed for this report.

Description of Test Parameters:

Temperature and Humidity:

The initial "proof of operation" testing will be conducted under "ideal" or laboratory conditions to establish a baseline capability, vapor concentration. This baseline concentration can then be utilized as a starting point for more stressful testing in which the SUTs are subjected to variations in humidity, temperature, interferents, etc.

In addition due to the known dependence of the reaction of the IOS substrates when exposed to analytes once the "proof of operation" testing is completed, CBC with concurrence from the JPM will conduct a series of trials with three (3) selected analytes at two additional RH levels, low and high. These lower level will be with 20% RH at ambient temperature and the high at 80% RH at ambient temperature. All RH levels will be within 5% of the target level and automatically monitored and recorded during all test events.

Concentration:

Testing will begin using a high concentration of 5mg/m^3. We believe this concentration can produce a positive response from the MOSA SUTs based on previous work performed (we assume exposure times and carrier flows are held constant to start so in this case concentration correlates 1:1 with total mass flux). Addition concentrations will be determined using a logic diagram, using GB as an exemplar, shown below in Figure C_1.

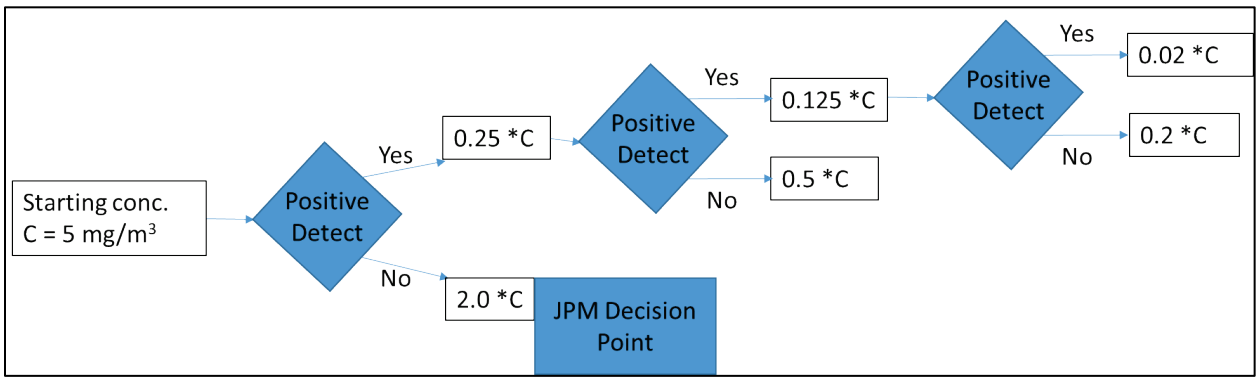


Figure C_1 Detection Logic Diagram. NOTE: Starting concentration is based upon work under Cooperative Research and Development Agreement (CRADA) 1935C.

Exposure Time:

The initial exposure time for all analytes will be 60 s. This exposure will be preceded by a 5 min clean air stream at the designated RH and a 5 min clean air stream after the dilute analyte stream is switched off.

Timed Exposure:

Once the baseline concentration is established a timed exposure will be conducted and twice the established concentration – can be adjusted at the discretion of the JPM. The

purpose of this test is to determine the response time of the system to a short exposure, e.g., passing through a small vapor cloud. The logic to be used in this test configuration is shown in Figure C_2.

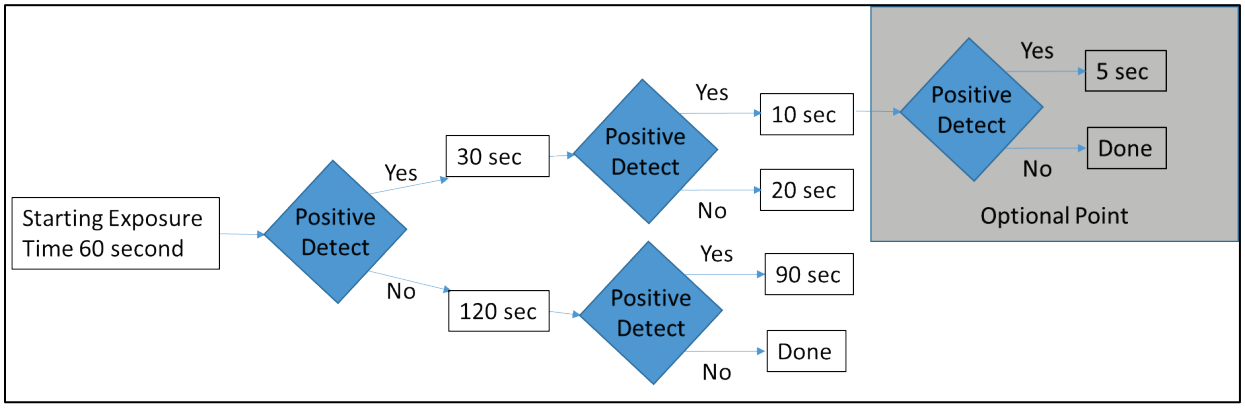


Figure C_2 Time Exposure Logic Diagram.

Air Flow Rate:

A constant flow of humidified carrier air will flow into the sealed chamber at a rate of 2 L/min. This flow rate ensures that during the 60-second exposure time, there should be at least one complete exchange of the air within the test chamber ensuring that all three MOSA units should receive sufficient exposure to the target analyte.

When the 60-second exposure period begins, an additional 0-20 mL/min of dry carrier air from the analyte vapor-generating saturator cell will be introduced into the carrier stream. The cell continuously generates a saturated headspace of analyte vapor evaporating off of a porous ceramic wick inside of a glass tube. The analyte vapor stream is kept dry in order to prolong the lifetime of any analytes which may be susceptible to hydrolysis. By maintaining a constant carrier stream flow rate of 2 L/min, the addition of 0-20 mL/min of analyte carrier air would affect the humidity and flow rates by no more than 1%, keeping those parameters within allowable variance tolerances for testing and should not negatively impact the SUT performance.

Description of Test Events:

NOTE: All three (3) SUTs (during standalone testing) will be sealed in the test chamber with a clean stream of carrier air actively flowing through the chamber.

Baseline Testing:

A baseline capability is required to establish an acceptable operational concentration of the system under “ideal” conditions. This concentration will be used as a starting point for on the move testing and any follow on system stress testing (e.g. variation in temperature, humidity, etc.)To establish this baseline a series of tests with the standalone MOSA GEN1 configuration will need to be conducted. The analyte list can be found in Table C-1 where concentration C is the initial concentration, for GB this will be 5 mg/m^3.

Table C-1: Test Matrix

Chemical	RH	Conc. C	Conc. #1	Conc. #2	Conc. #2
GB	50 %	5mg/m^3			
HD	50 %	5mg/m^3			
GD	50 %	5mg/m^3			
CG	50 %	5mg/m^3			
DFP	50 %	5mg/m^3			

Details of Sensor Operations need to be incorporated once the prototype configuration is finalized.

The test event will begin once the operator hits start on an Excel spreadsheet using a macro to record the time to the second. This simultaneous timestamping will correct for time differences between all computers used for data collection.

1) The clean carrier stream will flow over the SUTs for 5 min. During this time the lab operators will make note of any false alarms which may occur.
2) At the end of the 5 min period, the clean and dilute analyte streams will be switched (both are continuously flowing at the same rate).
3) The analyte stream will flow over the SUTs for 60 s. Lab operators will locally record the times that the analyte stream is both turned on and off using the macro enabled Excel sheet.
4) At the end of the 60 second period, the dilute analyte stream will be turned off. Lab operators will continue to record data for an additional 5 min after the analyte flow is turned off. Additional alarms during this period will be recorded by lab personnel.
5) The test event will conclude when the operator hits stop button on the Excel spreadsheet.

All test events will be performed in triplicate, therefore 9 independent trials will be recorded per challenge.

A MINICAMS pre-concentrating GC unit will be used to monitor the dilute analyte stream. Concentrations for each set of three test events will be determined as follows:

1) Before running the 3 test events, the dilute analyte stream will be directed into the chamber for 10 min, during this time the MINICAMS will collect two data points establishing a concentration level.
2) After running the 3 test events, the analyte stream will again be directed into the chamber for 10 min allowing the MINICAMS system to collect two data points. These data points taken before and after each triplicate set of test events will be accepted as the range of concentrations present for the 3 test events that they bookend.

In addition, sorbent tubes will be placed within or used to draw samples during the test event. The sorbent tubes will be analyzed to determine the mass of anlayte to which the MOSA GEN1 was exposed during the duration of each trial, providing a secondary confirmation of average exposure levels.

On-the-Move (Flight) Testing:

CCDC CBC has established the capability to perform small scale on-the-move (flight mimicking) testing. The Modular Test Chamber (MTC) is an open ended modular tunnel with a 30" cross section, allowing for systems up to 24 inches in overall width (24" rotor tip to rotor tip for Quad UAS) to be moved through a semi-contained cloud at speed, maximally 10 mph. The MTC consists of three sections with the center section having automated dual direction sliding doors allowing for a small "cloud" of vapor to be generated and characterized for a short period of time. Once the cloud is established in the center section of the MTC the UAS can be translated down the center line of the MTC on a linear stage (max speed 10mph, max weight 10 lbs.). Just before the UAS enters the center section its automated doors will open and the UAS can traverse the cloud with or without rotors in operation. This is a new piece of infrastructure and full details are being documented currently and will be validated prior to use.

The initial test with a MOSA GEN1 sensor can be conducted on a mock UAS (shell) in order to determine the efficacy of the sensor in the chosen mounting location, with rotors off - no rotor wash. The initial testing concentration will be driven by the results of the baseline testing described above, likely twice (x2) the lowest successful detection level. Concentration and speed can be adjusted in order to establish a baseline on-the-move detection capability. With the established on-the-move baseline the rotors can be started and test repeated.

Can you provide "step by step" information on how this test will be conducted?

1) The UAS will be mounted on a pole which is attached to a 3m linear stage located along the top central axis of the MTC. The linear stage will allow the SUT to traverse the length of the MTC under controlled motion, position, and speed.
2) The central section of the MTC will begin with the doors closed, and the sample vapor will introduced to the desired concentration. The concentration will be confirmed.
3) If required the SUT sensor can be activated and allowed to run for a short duration, 5 min is suggested, to ensure the baseline signal is reached.
4) The UAV rotors will be either dormant (off) or active (operating) as defined in the test matrix
5) Once the SUT has begun to traverse the length of the MTC, both central section doors will open and the SUT will move through the vapor cloud at speed, as dictated by the test matrix
6) A sample of the central section of the MTC will be taken to determine the vapor concentration during test event, bounding the concentration range
7) Clean air will be flowed through the chamber to clear the sample vapor and monitored vapor presence until the MTC is cleared prior to the next trial.

As during baseline testing, MINICAMS will be used to monitor the dilute analyte stream. See baseline testing for specific information on how concentration will be determined. All test events will be performed in triplicate, therefore 9 independent trials will be recorded per challenge.

Reporting:

At the end of testing CCDC CBC will provide a summary test report within 30 days for review by the JPM CBRN Sensors. This summary report will provide a list of all test events to include start and stop times, challenge levels, and responses from ground truth instrumentation. In addition CBC will photo-document exemplar trials and generate Test Incident Reports (TIRs) for any event that causes a cessation of testing. All TIRs will be summarized in the final report.
It is requested the JPM review the summary report within 30 days, and CBC will address all comments and required changes within 30 days of receipt. A final report will then be generated in CBC tech-report format. At the concurrence of the JPM the final report will be submitted as an official tech-report for addition to the DTIC library.

Notes from CRADA 1935C Development work For Reference Only:

By establishing the concentration of analyte, knowing the total volume flowing through the test chamber per unit time and the exposure times for each test event, the total mass flux of GB to which the SUTs were exposed can be calculated. This total mass flux will be the independent variable against which the SUTs are challenged because it was determined that concentration alone is not a relevant independent challenge variable (i.e. if 2,000 ng of GB are added to a 2 L/min carrier stream for 5 min in Trial A and 2,000 ng of GB are added to a 4 L/min carrier stream for 5 min in Trial B, although the concentration is only half the value in Trial B, both trials expose the SUTs to the same mass of GB). Since the MOSA technology in general is a polymer dye, bit of a misnomer in that the polymer itself is not color-imparting; rather, the polymer is merely the translucent material out of which the dots are made. It's porous in order to increase surface area and increase the odds that a vapor molecule impacting the dot will actually interact with the dye molecules incorporated into the polymer. If a vapor molecule contact the the dot, it doesn't just automatically stick to the surface and react; it can merely bounce back off unaltered. It needs to impact the surface with the right energy and the correct angle such that the reactive parts of the vapor molecule and dye molecule actually come into contact and have a chance to react. The polymer dye response is a function of anlayte molecules interacting with IOS media, hence the total mass interacting with the polymer is the driving force behind a response.

We don't know the exact formulation, but the dye is likely either integrated onto the surface of the polymer or trapped in the pores, but the polymer's role is to simply increase the odds that a vapor analyte molecule will stick around long enough and interact with a dye molecule in the proper conformation in order to cause a chemical reaction and color change.

APPENDIX C:
OFF-GASSING OBSERVATIONS

Darren K. Emge and Kathy Crouse
19 October 2020

After observing inconsistent results with the multi-analyte optical sensor array (MOSA) units after our restart, it was decided to track back the source of the inconsistencies. Of particular interest, and a point of discussion over the summer of 2020, was the concern over the housing and off-gassing during subsequent trials. To this end, U.S. Army Combat Capabilities Development Command Chemical Biological Center (Aberdeen Proving Ground, MD) designed a simple experiment that was conducted on Monday, 19 October 2020. The Monday date was chosen after a three-day down period where all components (e.g., chamber, boards, housings, etc.) were allowed to "air" for three days within engineering controls where approximately 3500 ft^3/min of air was flowing. On Monday morning, a singular MOSA, was wired up, and the fan was turned on. The unit was then placed in the chamber and exposed to a vapor stream of sarin (GB) for 10 min followed by a 10 min clear-down period with clean air. At approximately 5 min into the 10 min GB exposure, long enough for equilibration, a tube was pulled for 1 min at 200 mL/min and analyzed using a calibrated gas chromatography system (GC) with a flame photometric detector (FPD). At the end of the 10 min clear down (with clean air), the MOSA unit was pulled from the chamber and placed in a sealed jar for 15 min. At the end of the 15 min, an absorption tube was pulled for 1 min at 200 mL/min and analyzed using a calibrated GC with FPD. The results are shown herein.

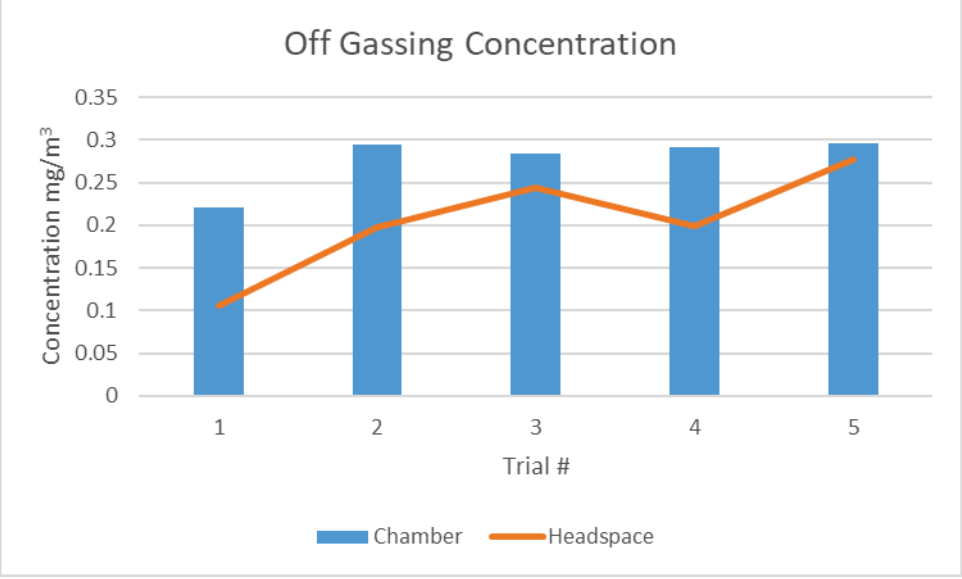


Figure C-1. Off-gassing concentration.

In the first trial, the housing and chamber were both clean after 72+ h of "airing" in the fume hood. Off-gassing was observed due to the presence of approximately half of the GB concentration in the headspace of the jar containing the MOSA unit. The MOSA unit was pulled from the jar and placed back in the chamber, and the experiment was repeated five times. In Figure C-1, the blue bars show that the off-gassing from the MOSA unit increased the subsequent chamber trial concentration by around 30%.

The concentration in the headspace shows an increase as a function of trial with the exception of trial 4. It was noted that the cap nut on the Swage lock bulkhead connector was left off during this trial, which is the most likely cause of this loss in signal. This observation raises the concern that the performance (i.e., detection capability) of the MOSA unit has been and will continue to be biased, based upon where the sequence of test events for a trial occurs.

Additionally, looking back at the blower housing data from 10, 13, and 14 October, this bias was most likely present, as shown in Figure C-2.

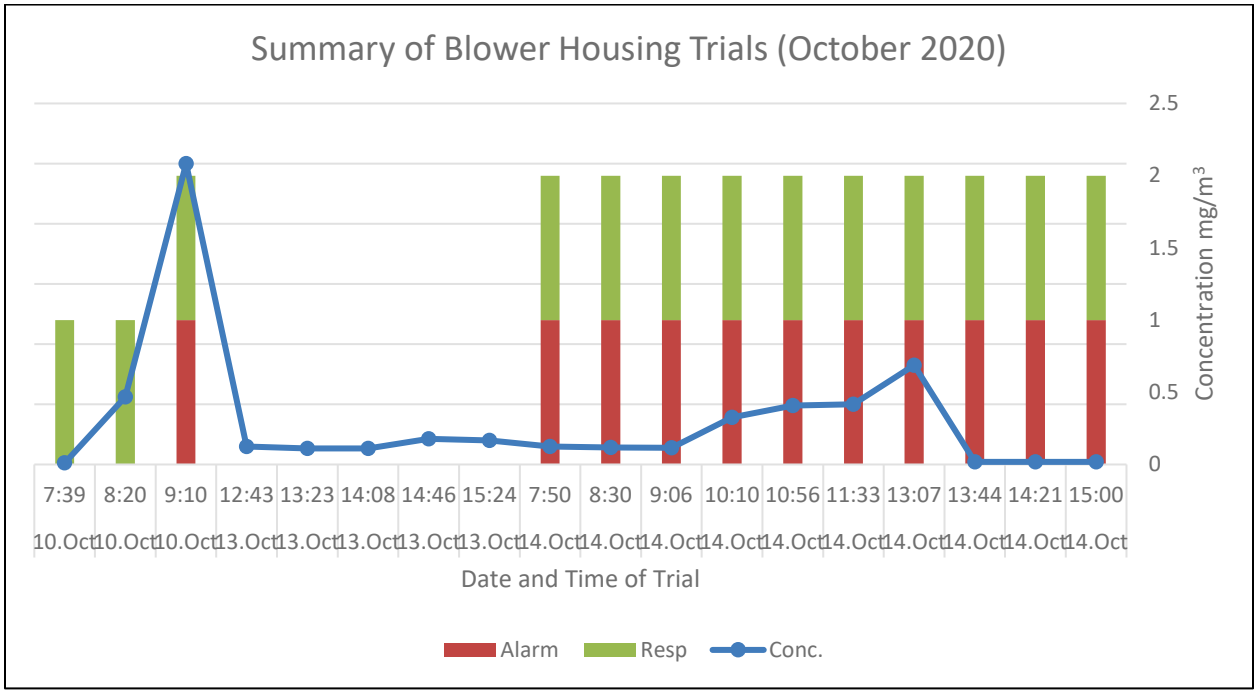


Figure C-2. Blower housing trial summary.

As can be seen, on the first day of testing (10 October), the trend in the data was quite positive and logical, showing low-value responses (gray bar) with an alarm at high concentrations (orange bar). A long weekend followed (Columbus Day), and testing was started in triplicate at low levels, ~0.05 mg/m^3; increased to ~0.08 mg/m^3; then increased to ~0.15 mg/m^3. Conformational trials at ~0.01 mg/m^3 also resulted in unexpected alarms and trends in the data. This and the apparent lack of stability in the concentration on 14 October in the period from 10:10 to 11:33 prompted us to perform the experiments conducted on 19 October and described above.

From what is shown, if the MOSA devices are to be reused during testing, there will need to be a sufficient number to ensure that they can be allowed to off-gas overnight, or an established resting period should be allowed between trials to alleviate any bias in the results. A study would need to be performed and would most like be dependent on both concentration and exposure time.

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