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14. ABSTRACT This Test Operations Procedure (TOP) provides the current standard methods for chemical point detector testing and assessment with and without operational background materials for vapor hazards including toxic industrial chemicals (TIC), chemical warfare agents (CWA), emerging threats, and CWA simulants.				
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US ARMY TEST AND EVALUATION COMMAND
TEST OPERATIONS PROCEDURE

*Test Operations Procedure 08-2-188A

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CHEMICAL POINT DETECTOR VAPOR TESTING

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* This TOP supersedes TOP 08-2-188 CN1 Chemical Point Detector Vapor Testing, dated 10 May 2019.

1. SCOPE.

This Test Operations Procedure (TOP), which has been endorsed by the Test and Evaluation Capabilities and Methodologies Integrated Process Team (TECMIPT), will address chemical point detector vapor testing and assessment with and without operational background materials for vapors including toxic industrial chemicals (TIC), chemical warfare agents (CWA), emerging threats, and CWA simulants. Test procedures and operations have been provided in terms of a 'best practice' approach. Some detector-specific limitations may apply and should be addressed accordingly within test-specific documentation.

NOTE: From this point on, analyte will refer to the chemical challenge to be presented to the system under test (SUT) during test operations.

1.1 Purpose.

a. This TOP provides standardized procedures for test preparation, planning, conduct, and reporting test results that evaluate a point detector's capability to detect and/or identify chemical hazards.

b. Absolute humidity or water vapor content (WVC) will be used in this document instead of relative humidity. Absolute humidity is the measure of water vapor (moisture) in the air, regardless of temperature. It is expressed as grams of moisture per cubic meter of air (g/m^3). The maximum absolute humidity of warm air at 30 °Celsius ($^{\circ}\text{C}$) is approximately 30 g of water vapor per cubic meter of air or $30 \text{ g}/\text{m}^3$.

1.2 Limitations.

a. The procedures in this TOP alone are not sufficient to fully evaluate the effectiveness of a chemical point detector. These procedures are designed to be used as one component in an overall assessment program evaluating the materiel performance and manufacturing of chemical point detectors.

b. The results obtained by using these test procedures cannot be correlated to the full range of battlefield conditions; however, key documents, such as the system threat assessment, can help guide prioritization in establishing the range of battlefield conditions that should be tested.

c. The scope of this TOP will not cover emerging capabilities and will focus on the testing of chemical point detectors for vapor hazards. As new capabilities evolve, further TOPs will address their testing (e.g., aerosol hazards testing with operational background materials is addressed in TOP 08-2-054A^{1**}).

d. This TOP is limited to currently approved standards and procedures. Developments in practices, equipment, and analysis may necessitate new testing procedures. Additionally,

** Superscript numbers correspond to Appendix D, References

standards of performance must be adjusted as technologies advance. Test procedures and parameters listed in this TOP may require updating to accommodate new technologies in test items or in test instrumentation.

e. Instrumentation and detection methods may not exist for background substances in the operational environment. These materials are complex mixtures of variable components and significant efforts will be required to develop referee methodology.

f. Each program should concur on definitions for the terms “detection,” “identification,” and “classification” with stakeholders. The following definitions may be used as guidelines. Identification names a hazard uniquely and correctly, such as GB. Classification assigns the hazard to a class, such as G agent. Detection advises that some hazard is present.

2. FACILITIES, EQUIPMENT, AND INSTRUMENTATION.

2.1 Facilities.

<u>Item</u>	<u>Requirement</u>
Chemical surety laboratory and chemical agent storage facility.	Constructed to ensure safe and secure storage, handling, analysis, and decontamination of necessary quantities of chemical agents, other contaminants, or simulants.
Chemical agent test facility (chamber or laboratory) with environmental control system.	Constructed to house the detector fixture during agent or simulant challenge and sampling. The chamber should have sufficient volume to allow free air circulation around the SUT. Test areas in laboratories or chambers must be equipped with environmental controls that allow air temperatures and air-exchange rates to be maintained at prescribed levels throughout the testing period.
Detector fixture and exposure chamber.	Constructed to house the SUT during analyte dissemination. Includes environmentally controlled (temperature and humidity) test chamber, analyte disseminators, and all instrumentation necessary to perform testing (such as data recorders).

2.2 Equipment.

<u>Item</u>	<u>Requirement</u>
Analyte Vapor Dissemination System	Designed and built to provide a hazard challenge of the desired analyte to the SUT under required environmental conditions. This system is controlled by the use of mass flow controllers
Operational background materials Dissemination System	Designed and built to deliver the desired operational background material (as required) in the analyte airstream to the SUT under required environmental conditions.
Differential pressure (ΔP) measurement system	Measure ΔP upstream, next to, and downstream of the SUT for safety and for data quality. Dissemination regions are at positive pressure (ΔP at least 1 inch water gauge (iwg)) so that laboratory air does not leak in and dilute the challenge flow. The SUT is at near ambient pressure so that pressure changes do not affect its performance. The exhaust line is at negative pressure (ΔP less than -1 iwg) so that agent vapor does not leak out.
Referee sampling system (as close to real time as possible)	System to sample and quantify the challenge analyte. There will also be a referee system for the operational background materials challenge to the SUT if the methodology is available.
Distribution manifold.	Designed and built to equally distribute the analyte airstream to multiple SUTs and will be part of the vapor dissemination and operational background materials dissemination systems.
Humidity generation and control system.	System designed and built to provide humidity into the analyte airstream directed to the SUTs. WVC will be controlled and monitored. The system will also provide a humidity controlled environment within the detector fixture. WVC will be controlled and monitored.
Temperature control system.	System designed to provide temperature control and monitoring of the analyte airstream at the SUT, detector fixture, and exposure chamber.
Data acquisition system (DAS).	System designed to automate data collection from the detector fixture. All data will be time tagged and synchronized (as much as possible).
Video data acquisition (optional)	A system to collect visible detector responses on the screen. All video data will be time stamped. Adequate resolution and speed (frames/second) to document typical test procedures.

2.3 Instrumentation.

Instruments must be able to accurately measure the respective test parameters as described to meet the test program requirements.

<u>Parameter</u>	<u>Measuring Device</u>	<u>Permissible Measurement Uncertainty</u>
Analyte concentration (dissemination and detection) (milligram per cubic meter (mg/m ³))	Gas chromatograph, high-performance liquid chromatography, liquid chromatography, spectrophotometer, or equivalent.	± 15 percent; ± 25 percent at the bottom of the range being measured.
Temperature (-32° to 50 °C).	Temperature sensor with digital recording capability or equivalent.	± 0.5 °C.
Humidity	Hygrometer or similar measuring instrument with digital recording capability.	The WVC in the condition/ challenge airstream will be within ± 10 percent of the target value when the target WVC is ≥ 5 g/m ³ . When the target WVC is less than 5 g/m ³ , the WVC in the condition/ challenge airstream will be within ± 0.5 g/m ³ of the target.
Differential pressure (ΔP)	ΔP gauge oriented so that it reads positive when the fixture pressure exceeds ambient pressure. A gauge with a range of ± 1 iwg may be suitable.	± 0.1 iwg.

NOTE: The permissible measurement uncertainty is the two-standard deviation value for normally distributed instrumentation calibration data. Thus 95 percent of all instrumentation calibration data readings will fall within two standard deviations of the known calibration value.

2.4 Test Controls.

The following are suggested tolerance values for the test parameters identified. Specific program requirements may require tighter or allow less stringent tolerances. Many variables must be considered when determining the permissible error of measurement. The final outcome of the data analysis must be considered along with the criteria for the SUT that is being assessed. The statistical considerations that have gone into the test design must be included in the

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determination of tolerances as well as the propagation of uncertainty/error that will be a part of the final data output from testing. The table identifies tolerances that have been considered “best practice” for various test events, but each test event should adjust the tolerances to best fit the data needs for the analysis of the SUT. Actual instrumentation may have greater precision and accuracy; actual values will be reported.

NOTE: Tolerance values are the permissible limit or limits of variation in a measured value (temperature, humidity, etc.). These accuracy values are recommended, but will be further defined in the DTP to meet test needs.

<u>Parameter</u>	<u>Accuracy</u>
Analyte Challenge Concentration	± 15 to 25 percent
Temperature	± 2 °C
WVC	The WVC in the condition/ challenge airstream will be within ± 10 percent of the target value when the target WVC is ≥ 5 g/m ³ . When the target WVC is less than 5 g/m ³ , the WVC in the condition/ challenge airstream will be within ± 0.5 g/m ³ of the target.
Mass Flow	± 5 percent of full scale
ΔP	± 0.2 iwg

3. REQUIRED TEST CONDITIONS.

3.1 Test Planning.

a. This TOP provides guidance on test design issues and data requirements that should be accompanied by information from other documents, such as the system evaluation plan (SEP), system threat assessment, and the test and evaluation master plan (TEMP). For those testing programs in which a SEP is not available or applicable, the test facility should consult with the customer and use previous documents as a guide in addition to this TOP.

b. This TOP will be used as guidance when preparing detailed test plans (DTPs).

(1) The test procedures described in this document (Paragraph 4) must be referenced and incorporated into a DTP or similar document but may be modified in the DTP to accommodate unique items or materials, limitations of the SUT, or to satisfy testing requirements specified in the Operational Test Agency SEP or other acquisition documents. Alteration, however, will be made only after full consideration of how the changes may affect

the reliability and validity of the data. These alterations, justification for the alteration, and the anticipated impacts to the test data must be fully described in the DTP.

(2) At a minimum, coordination efforts will address the impact of the modifications to the following test areas:

- (a) Safety.
- (b) Test conditions.
- (c) Environmental effects.
- (d) Human use.
- (e) Data quality.
- (f) Test validity.
- (g) Manufacturer limitations for the SUTs.

c. Appendix A contains vapor testing issues that must be considered when planning to use emerging threats.

d. The following concerns should be considered when planning a test in a temperature conditioned environment:

(1) When an environmental chamber is at a significantly different temperature than the ambient laboratory, the air entering the chamber for the purpose of agent dilution or conditioning often does not have enough residence time inside the chamber to equilibrate to the environmental chamber. This can result in mismatches between the environment that the detector is in and the temperature of the agent and conditioning airstreams. When testing with fixtures or chambers, allow for the independent conditioning of the air sources prior to entering the environmental chambers.

(2) Referee line(s) inside the chamber may experience water condensation from a flow meter outside the chamber. Calibrated flow meters are generally kept outside of agent contaminated environmental chambers whenever possible. This causes condensation during flow measurements when outside air is being drawn through the meter into a cold environment. A dry air purge system (an inert gas may be used) should be developed to prevent this from happening.

(3) Any surface cooled below the dew point of ambient air is subject to condensation. Depending on the location, condensation may be prevented by flowing dry gas across the chilled surface.

e. The following concern should be considered when testing a SUT with wireless communication capability: A SUT may interfere with electronics outside the building, or electronics may interfere with the SUT. The test center may need to work with authorities to coordinate the allocation of radio spectrum frequencies.

f. Some detectors have integrated CWA, TIC, explosive level, and oxygen level sensors. Some TICs are corrosive or reactive. A sufficiently high concentration of TIC vapor may degrade other integrated sensors.

3.1.1 Experimental Design.

When performed correctly, design of experiments (DOE) is the most efficient way to test. Multiple factors are varied simultaneously in a specific systematic manner that is mathematically sound. This means that DOE techniques minimize the number of trials needed to obtain statistical validity. It is recommended that proper use of DOE be applied for all testing. When creating a DOE the following should be considered:

- a. The test objective(s).
- b. The response variable(s).
- c. The factors that affect the response variable(s).
- d. The levels (or ranges) of the factors.
- e. Any mathematical model assumptions.
- f. Statistical measures such as confidence, power, variability, and error structures, etc.
- g. Acceptable errors.
- h. The final analysis method (including, limits of detection and defined instrumental method of analysis).
- i. Any limitations of funding, SUT availability, and/or schedule.

3.1.2 Simulant Selection.

a. The test and evaluation working integrated product team (T&E WIPT) will coordinate the selection and use of any simulants. Simulant selection (TOP 08-2-196A²) may be conducted under the acquisition program of record to identify and verify optimal simulant(s), based on the program's hazard and performance documents.

b. The simulant should produce a signature signal similar to the one from the hazard analyte. Additional considerations are listed.

(1) When the detector will be used for multiple chemicals, several different simulants may be needed to cover the range of signals produced by the various chemicals.

(2) Because the recognition algorithm is the major component of the detector that is being tested, the simulant must produce a signal similar in complexity to the chemicals being analyzed.

3.1.3 Documentation.

a. All pertinent test documentation that is required will be available before testing begins.

b. Familiarization.

(1) All pertinent current TOPs and standing operating procedures (SOPs) should be reviewed.

(2) Potential problem areas and test duplication must be identified by reviewing previous records and results of similar tests, if available.

(3) Development of DTPs requires familiarization with the applicable test planning and requirements documents such as the TEMP, SEP, capability development document (CDD), or capability production document. Test specifications such as selection of appropriate samples, methods, test sequences, facilities, and test equipment will be collected from review of requirement documents and background information such as references from preceding development, test phases, and similar studies.

(4) Safety and health issues must be given prime consideration in test planning. All applicable/available safety documents such as the safety assessment report and health hazard assessments should be reviewed to determine if any safety or health issues require special test protocols. For any tests involving military personnel not assigned as testers, safety release and human use committee approval are required.

3.2 Test Fixture.

3.2.1 Dissemination System.

a. The design and type of dissemination system (Facilities and Instrumentation, Paragraph 2) depends on the natural state of matter of the analyte being used.

b. The dissemination system must be able to maintain the concentration and other characteristics of the challenge for the time period specified in the program requirements (e.g., CDD, SEP, and TEMP).

NOTE: Because of potential agent loss in the tubing, all dissemination lines should be as short as possible and the tubing should be made of the most chemically resistant material possible. It was discovered that perfluoroalkoxy (PFA) tubing may swell. Stainless steel is the preferred tubing.

c. Vapor concentration adjustment may be achieved by introducing dilution gas between the vapor generator and the distribution manifold. Mass flow controllers placed upstream of the vapor in the system can be used to meet the vapor flow rate requirements.

d. When a dissemination system is placed in an environmental chamber that is at a significantly different temperature than the ambient laboratory, the air entering the chamber for

the purpose of agent dilution or conditioning often does not have enough residence time inside the chamber to equilibrate to the environmental conditions. This can result in mismatches between the environment that the detector is in and the temperature of the agent and conditioning airstreams. When testing with fixtures or chambers, allow for the independent conditioning of the air sources before entering the environmental chambers. Furthermore, ensure that the temperature measurement equipment is placed to properly characterize the challenge airstream and the detector's immediate environment.

e. Referee line(s) inside the chamber may experience water condensation from a flow meter outside the chamber. Calibrated flow meters are generally kept outside of agent-contaminated environmental chambers whenever possible. This causes condensation during flow measurements when outside air is being drawn through the meter into a cold environment. A dry air purge system (an inert gas may be used) should be developed to prevent this from happening.

3.2.2 Detector Test Fixture.

a. The transfer line is used to transport the conditioned airstream and the challenge airstream through the test fixture. It connects the dissemination system to the distribution manifold (DM). It is extremely important that during test planning the minimum amount of airflow to provide the detectors with a valid analyte challenge is established (typically from the detector tech package). The mass flow controllers in the system will be used to move the analyte airstream into the DM and to the detector inlet. The total airflow moving to the detectors must be greater than required to supply the SUTs, the referees, plus some excess to account for flow variability in order to eliminate the possibility of "starving" the detector and not presenting a valid analyte challenge. The amount of airflow is determined during the test planning phase with input from the customer and evaluators.

b. The DM is used to equally distribute the challenge stream to multiple SUTs and referee probes/sample lines. The DM should be made of the most chemically resistant material as possible.

c. The DM interface to the SUT (may vary depending on the design of the SUT) is designed to present an analyte challenge (with temperature, WVC, and operational background materials, as required, at appropriate conditions) to the SUT and must not affect the SUT's response. The fixture must be capable of allowing the SUT to sample clean (free of contaminant) conditioned air between analyte challenges.

3.2.3 Referee Systems.

a. Vapor Concentration. Vapor concentration probes or sampling lines should be connected to the DM. Sampling probes/lines should be installed as close as practically possible to the SUTs to avoid any line effects and accurately characterize the condition/challenge airstream. The referee instrumentation should be located outside the detector test fixture.

NOTE: Because of potential agent loss in the tubing, all referee sample lines should be as short as possible and the tubing should be made of the most chemically resistant material possible.

b. Operational Background Materials Concentration. Gases will be generated into a headspace and will be characterized as a percentage of the conditioned analyte airstream (e.g., 10 percent). Liquid operational background materials will have an airstream passed through the liquid saturated headspace before entering the analyte airstream. There is an on-going program to develop a worldwide operational background profile database. Future methods developed from the database for the introduction and measurement of operational background materials into the challenge airstream will be considered.

c. Temperature. Temperature probes will be installed in the detector test fixture and the DM. Locations for probe placement should be chosen such that the data recorded can be used to properly characterize the environment within the fixture and the DM.

d. Humidity (WVC). Humidity probes (or temperature/humidity probes) will be installed in the detector test fixture and the DM. Locations for probe placement should be chosen such that the data recorded can be used to properly characterize the environment within the fixture and the DM.

3.2.4 Control/Data Systems.

a. The control software is used to establish the required vapor concentration, temperature, and WVC until system stability is achieved for trial initiation.

b. DAS recording software will be used to digitally record the data.

NOTE: The DAS should be capable of digitally storing the data and translating it into a comma-separated value format for export, which is compatible with commonly used statistical and data analysis software.

c. All clocks and time stamps for all data collection devices must be synchronized. Synchronized equipment must include, but not be limited to, all referee instruments, all sampling instruments, and remotely operated dissemination equipment (e.g., pressure, temperature, and humidity sensors, etc.).

d. Still photographs should be taken to document the test fixture setup. When possible, photographs of scales or rulers will be included to show relative dimensions and distances.

e. The SUT display must be recorded using digital video. This data will be used to ensure that DAS and SUT timing are synchronized and verify detector response performance. The video data may also be used with optical character recognition software to extract display data.

3.3 Pre-Test System Assessment.

3.3.1 Test Fixture Verification and Validation (V&V).

a. The detector test fixture must have a V&V before starting record testing. The V&V effort will determine and demonstrate the fixture capabilities. The V&V effort will also demonstrate repeatability and reproducibility of test methods and resulting data.

b. Pilot trials will be conducted to confirm test procedures, data collection, and analysis methods are all meeting test requirements before conducting record trials. The number and nature of the pilot trials will be negotiated between the test center and the program manager.

3.3.2 Pre-Test Systems Checks and Calibrations.

a. Ensure all equipment and instrumentation are functioning and/or recording properly.

b. Conduct a confidence check for each SUT as needed.

c. Verify that all calibrated items' certificates are current. If a calibrated item's certificate expires during testing for whatever reason, ensure that a replacement is calibrated and available for installation. Perform a pre-test instrument check to verify that drift has not occurred.

3.4 Safety.

3.4.1 General.

a. Operators should develop a risk management worksheet to quantify the risks involved in the operation based on the severity and probability of the hazards for the use of this test as well as the controls implemented to minimize the level of risk based on test site specific requirements. The composite risk management worksheet may be developed in accordance with (IAW) Army Regulation (AR) 385-10³, *The Army Safety Program*, and Department of The Army (DA) Pamphlet (PAM) 385-61⁴, *Toxic Chemical Agent Safety Standards*.

b. All test operators must read and indicate that they understand the SOP and test-specific procedures outlined in the DTP.

c. The required Safety Data Sheets (SDS), testing protocols, and safety procedures will be available at the test site.

d. When appropriate, the test personnel will wear required personal protective equipment (PPE).

e. Test personnel will be informed of potential safety and health hazards involved in test conduct and the precautions required to prevent accidents and limit exposure to the chemicals used in the test.

f. Safety checks and briefings will be conducted to ensure that all identified safety hazards have been addressed before testing proceeds.

g. For tests that involve carrying or lifting, test personnel and participants will be instructed in the proper lifting procedures.

h. Safety air monitoring should be used, whenever possible to ensure the safety of the test personnel during test conduct.

i. Training and Familiarization. Test personnel must be trained in the operation of the SUTs and test fixture to include the following:

(1) Description of the physical activities required during actual testing, to include applicable general operation. These will be provided in a written form, through audiovisual presentation, demonstration, or a combination of these methods.

(2) Any corrective maintenance and preventive maintenance that must be performed IAW the technical manuals.

(3) The types of data to be collected, quality control (QC) methods for data collection, and the relationship of the data to overall success of the test program [decision rules or data quality objectives (DQOs)].

(4) Chemicals being used in testing and any health hazards of the chemicals.

3.4.2 Chemical Handling.

a. Chemicals (TICs, CWAs, emerging threats, and simulants) must be safely handled with utmost care. Tests will only be conducted IAW the approved SOPs from the testing installation and the procedures specified in the DTP.

b. Test personnel must read and understand the SDSs associated with the chemical to be used. Also, the SDS for each chemical used in testing must be available in the test area along with the DTP, testing protocols, and safety procedures as required by the test site.

c. Appropriate PPE will be worn by personnel operating vapor generators whenever there is a potential hazard.

d. Should a chemical splash or exposure occur, means of rapid decontamination such as emergency deluge shower and/or emergency eyewash.

3.4.3 Hazards.

Identified safety hazards are those associated with using hazardous chemicals during testing. All test plans should contain a safety section identifying and addressing all safety concerns IAW the composite risk management guidelines of DA PAM 385-30⁵. The safety section of the test plan should be coordinated with the test site's safety office.

3.5 Quality Assurance (QA) and Quality Control (QC).

3.5.1 General.

a. Each test facility's QA program will be designed to ensure that data of the required quality are obtained from each test. The data quality requirements will be established by the customer as well as by the test facility's QA/QC SOPs.

b. The quality of instrument data produced depends on appropriate instrument maintenance, periodic calibration, QC measures, and careful documentation procedures. Calibration will be conducted IAW the validated calibration protocol of the test facility. In the absence of a validated protocol, calibration will be conducted as recommended by the instrument manufacturer.

c. Examples of QC measures associated with data reporting are sample collection documentation, tracking and evaluation of analytical results, and comparison of results. QC measures will be detailed in the DTP and will follow the test facility's QA/QC plan.

d. Sample collection QC measures will be IAW the test facility's sampling SOPs or as specified in the DTP. Any problems associated with a particular sample will be noted on the appropriate log sheet or data file. All data collected must be date and time stamped.

e. Data will be independently reviewed and authenticated as required by the test facility or the test program.

f. All analysis results and calculations will be peer reviewed to ensure that random errors in transcribing data or in performing analysis are eliminated, as required by the test facility or the test program.

g. For each trial, the analyte concentration at all required sample points will be measured and recorded. Analyte concentrations should be monitored as close to the SUT inlet as possible.

h. For each trial, the temperature and WVC will be monitored and recorded. If there are temperature and WVC changes between trials, exceeding the tolerances outlined in the DTP, these changes should be noted. The next trial will not proceed until the values are within prescribed tolerances.

i. Statistical analysis can be used to determine measurement errors and to process trial data.

3.5.2 Quality Objectives for Chemical Point Detector Testing.

In addition to the program-specific requirements, the following procedures will be followed:

a. SUTs, referee detectors, temperature sensors, humidity sensors, flow meters, photo and video streams, sampling locations, and raw data will be labeled in a manner precluding misidentification.

b. Data and analysis files will be reviewed and verified by qualified personnel knowledgeable and familiar with the test process, as determined by the test officer/director or the test facility's SOPs.

c. Each real-time monitor and/or near real-time monitor must be calibrated and checked IAW test site SOPs.

d. Details of data collection and handling (e.g., backups, data flow path) procedures are as follows: it is preferable to continuously record all test data with the DAS so that a complete analysis may be made of the test data. The DAS should record data from all instruments that have either a digital or analog output. Also, data should be time stamped and recorded in local time. Examples of these data streams are temperature and WVC statistics collected from an analog probe.

e. DQOs are designed to ensure scientifically valid and defensible data are obtained during testing. Both random and systematic errors in the measurements can occur because of shortcomings in test procedures, instrumentation, and in data collection systems. DQO principles are applied to measurements to determine how much error is acceptable before the data should be rejected.

f. Independent parameters most likely to vary during a single trial include: air flow through dissemination equipment, chemical vapor, or aerosol flow rate through dissemination equipment, analyte dissemination concentration, airstream temperature, and WVC. Lack of consistency in these parameters will affect performance measurements. If any DQOs are not met, subsequent trials should not continue until the source of the error is addressed or corrected.

g. Initial DQOs will be established based on the V&V process and recorded in the V&V report and configuration control documents. Program specific DQO needs that exceed the limits of a validated capability would require coordination with the program office and the T&E WIPT.

4. TEST PROCEDURES.

4.1 Receipt Inspection.

a. Upon receipt, all SUTs will be inspected IAW TOP 08-2-500A⁶.

b. As part of the receipt inspection, a SUT-specific functional check will be conducted to ensure that the SUT is undamaged, fully functional, and ready for testing.

c. Any problems or issues will be reported in test documentation.

4.2 Testing Procedures.

4.2.1 Pre-Test.

a. Examine the SUT for inlet type, inlet flow requirements, and any data connections. The SUT data stream also needs to be examined to see if any or all parts of the data stream will be used to determine SUT response or alarm to the challenge. At this time any proprietary software data downloading or data uploading onto networked computers can be addressed or mitigated. Information from this examination will help determine if any modifications to the DM inlet interface are required for a specific SUT being tested.

b. In coordination with the customer and the program T&E WIPT determine whether building electrical or battery power will be used during SUT testing in the fixture.

c. The customer and program T&E WIPT need to determine the frequency of performing confidence checks (e.g., at the start and end of every trial day) unless this information is provided by the manufacturer. The environmental conditioning time (the time the SUT will be at the desired environmental conditions before initiating the contaminant challenge) will also need to be established.

d. The challenge airstream (directed at the SUT inlet) tolerance limits and the SUT environment tolerance limits may be different. The challenge airstream may have more restrictive tolerance limits than the SUT environment.

e. The customer and program T&E WIPT will also need to establish the challenge time unless this information is provided by the manufacturer. The challenge time is the time the challenge airstream will be directed to the SUT for each detection opportunity.

f. The conditioning time, the challenge time, the time to achieve environmental conditions, and the time to achieve a specific contaminant concentration will be used to determine how many detection opportunities at a specific set of conditions and how many trials can be performed in a trial day and still allow end of day activities to be performed (e.g., data downloading).

g. It will be important in pre-planning to identify any classification issues with merged data streams or SUT performance that will impact data collection and allow mechanisms to be developed before testing is started to deal with those issues.

h. Relevant considerations listed above must be documented in the DTP.

4.2.2 Test Procedures.

a. The SUTs will be placed into the test fixture. The SUTs inlets will be aligned with the inlet interface. If an electrical connection is required to operate the SUT, then those connections will be made and verified that they will not come loose. The data connections will be made and data communication will be verified. Video cameras will be aligned with the SUT display screen.

b. Perform a SUT confidence check if required by the DTP.

c. Establish the initial environmental conditions as outlined in the DTP trial matrix.

d. At the end of the conditioning period, the challenge dissemination system will be started to achieve the contaminant concentration required by the trial matrix. Any time there is no challenge being directed to the SUT, the airstream to the inlet interface will only have clean conditioned air as outlined in the DTP trial matrix.

e. Once the challenge concentration is achieved, then the inlet airstream will be switched from the clean airstream to the challenge airstream for the required time. When the challenge time is reached, the clean, conditioned airstream will be switched to the SUT inlet.

f. The trial will continue until the required detection opportunities specified by the DTP or the predetermined trial duration limit is reached.

g. When a change in environmental conditions is required, the sequence for making the changes is: stop the water vapor injection system to drop the WVC, change the temperature as required. When the required temperature is achieved, then the water vapor generator can be initiated to achieve the desired WVC. Sufficient time should be allowed for the SUT to achieve equilibration as described in the DTP.

h. When the challenge concentration level must be changed, it may be necessary to drop the WVC until the new concentration level is achieved and then the WVC can be restored to the required level.

i. Execute the trial matrix outlined in the DTP.

j. A confidence check will be performed on each SUT if required at the end of each day's testing. At the end of each trial day, download all data for the trials conducted. Ensure that the SUTs are on clean, conditioned air. Stop all temperature conditioning and water vapor and contaminant challenge dissemination.

k. A test incident report will be generated whenever the SUT fails to function properly (e.g., fails to clear down after a detection opportunity).

4.2.3 Final Retrograde.

Upon completion of all testing, SUTs will be decontaminated IAW site specific regulations and procedures and the SUT technical manual. The test fixture will also be retrograded as required.

5. DATA REQUIRED.

The types of data collected should be well defined before testing starts and outlined in the DTP. The types and frequency of data that will be collected should be agreed upon by the evaluators and testers. The data format for presentation should be agreed upon by the evaluators and testers. Examples of data for consideration are included below.

NOTE: All referee data must be time stamped.

5.1 Receipt Inspection Data.

a. A photographic and text record [e.g., test incident report (TIRs)] of all inspected SUT equipment and accessories. The test item identification number assigned to any item.

b. Any test material deterioration or damage.

c. Record of repaired or replaced test material.

d. The operational status of the SUTs.

e. Any additional observations noted during the receipt inspection.

f. Results of SUT function checks.

5.2 Pretest Data.

- a. Referee calibration.
- b. SUT confidence checks (as required).
- c. Type and purity of matrix gas.
- d. Chemical (including water) origin, product number, batch or lot number, and purity.

5.3 Performance Test.

- a. Analyte concentration (mg/m^3).
- b. Calculated operational background material concentration (percentage of total airstream).
- c. Airstream temperature ($^{\circ}\text{C}$).
- d. Airstream WVC (g/m^3).
- e. SUT chamber temperature ($^{\circ}\text{C}$).
- f. SUT chamber WVC (g/m^3).
- g. Operator level SUT settings.
- h. Time analyte and operational background challenge (if applicable) initiated.
- i. Time analyte and operational background challenge (if applicable) ended.
- j. SUT response to analyte challenge.
- k. Type of alarm (audible, visible).
- l. Time of alarm.
- m. Time alarm ceases.
- n. Calculated time to clear-down.
- o. Analyte concentration at time of alarm (as available).
- p. Analyte concentration when alarm ends (as available).
- q. Log of TIRs issued.

5.4 Data Analysis.

a. Data will be recorded, consolidated, and verified throughout testing and at the completion of test. Level III (quality checked by peer review versus raw) data will be released to the customer and evaluation community.

b. Any additional data analysis will be performed IAW the DTP.

c. Data will be archived for future use.

d. A data authentication group (DAG) will review all test data and TIRs for evaluation purposes. One of the main goals of the DAG is to determine if the test data meet the DQOs established in the DTP.

6. PRESENTATION OF DATA.

a. All receipt inspection data must be reported. Results will be summarized and presented in tabular form, including surface cleaning or maintenance performed, and emphasizing deviations from manufacturer specifications.

b. Data pertaining to SUT function checks will be reported in a form that will allow pretest and posttest functional performance data to be compared.

c. A graph showing temperature and WVC over time for each trial will be presented with alarms noted. Each graph will have the upper and lower control limit for temperature and WVC.

d. A graph showing the analyte concentration over time for each trial with the upper and lower control limits. The alarms for that trial will be noted on the graph.

e. A table for each trial will list each controlled parameter (temperature, WVC, and analyte concentration) and whether or not the parameter was maintained in control based on the tolerance limits.

f. A table for each trial will present the time to alarm, analyte concentration at the time of alarm, the time the alarm ended, the analyte concentration at the time the alarm ended, and clear down time (if required).

g. All calculated data will be labeled as such to allow the community to distinguish calculated data from directly measured data.

h. Comments/observations made during test conduct will be reported, if applicable.

i. Any additional desired information will be determined by the customer and specified in the DTP.

j. TIRs will be part of the final test report package.

APPENDIX A. EMERGING THREAT AGENT VAPOR TESTING CONSIDERATIONS.

This appendix is a coordinated compilation of information from the Detection Branch, Engineering Directorate, U.S. Army Combat Capabilities Development Command Chemical Biological Center, Aberdeen Proving Ground, Maryland and the Chemical Test Division, West Desert Test Center, Dugway Proving Ground, Dugway, Utah. These details are provided as lessons learned to provide recommendations and best information possible when testing emerging threats in a glove box fixture located in a laboratory fume hood, glove box, or engineering controlled chamber.

A.1 VAPOR GENERATION TEST FIXTURE DESIGN.

Solenoids and stream selection. Initially a stainless steel solenoid assembly was used to allow an operator to direct either agent contaminated air or clean conditioned air to the SUT. This process worked very well for high volatility agents. When the solenoids needed to be used with low volatility compounds they had to be heated to facilitate the movement of the low volatility vapor through the solenoid. Without a heated solenoid, there would be a delay in the rise of the concentration passing through the solenoid. This heating process makes the delivered agent air stream rise in temperature, which could adversely affect the test by impacting the agent delivery airstream temperature conditioning. Additionally, it was frequently seen that the solenoids would not be clean at the end of a test day. Due to the concerns with carryover contamination, a solenoid system should not be used to direct different airflows to the detector without considering these effects.

A.2 VAPOR GENERATION.

a. Compound delta tube to overcome condensation. The requirement for large volumes of “high” concentrations of low volatility agents means that the liquid agent needs to be heated to produce more vapor. When attempting to generate vapor using traditional glassware, the delta tube would be immersed in a temperature bath and meet a clean conditioned dilution air stream outside of the temperature bath. The change from hot agent air meeting room temperature clean dilution air and glassware created condensation, causing instability in the vapor generator. The vapor generation setup needed to be modified to allow for mixing of the agent air and clean conditioned air inside the water bath to mitigate this problem.

b. Low volatility compounds may have high volatility impurities from the synthesis process. If the testing requires a clean vapor sample, then a purge of the high volatility impurities would be required. Purging the impurities from the low volatility compounds requires trial and error to determine optimal settings for temperature, flow, time, and total agent mass loaded for each individual compound tested. Prior to using a low volatility compound in the vapor generation setup, it is recommended that a determination of suitable purge parameters be made; typical parameters include a delta tube in a water bath at 40-60 °C for 12 hours with 100 mL/min of nitrogen directed over the low volatility agent.

APPENDIX A. EMERGING THREAT AGENT VAPOR TESTING CONSIDERATIONS.

c. Vapor production with high surface area substrate. One method for producing a vapor is a new system which produces a saturated airstream by passing the air through a tube or a series of tubes filled with the test chemical and a high surface area substrate. Once the airstream is saturated, it is then mixed with additional clean air until the desired concentration is reached. The concentration produced is very consistent and quite reproducible. It was discovered that PFA tubing may swell. Stainless steel is the preferred tubing.

A.3 EMERGING THREAT DEGRADATION CONSIDERATIONS.

Material degradation due to heating. The amount of temperature and time that various compounds were stable had to be determined through trial and error and is not fully understood. In the vapor generation setup, the recommended maximum temperature found through trial and error when working with a low volatility compound over a long duration is 55 °C with nitrogen over the headspace of the agent. Additional materials tested in very early work demonstrated different thermal stability, indicating that this type of study needs to be performed on any new compounds generated.

A.4 VAPOR REFEREE.

a. Referee sorbent sampling methodology. Custom inlets were developed and manufactured to fit Agilent 5890/6890 gas chromatographs (GCs). These inlets are designed to completely remove any need to transfer low volatility compounds through a heated transfer line – the compounds are thermally desorbed from a 3 millimeter (mm) sorbent tube directly onto the column. Once designed, each agent was tested against a specific method on the instrument. Low volatility compounds were initially tested using the method for VX. All agents used Tenax sorbent material. The methods developed for analyzing these compounds were designed to take as little time as possible to allow collection and analysis of as many samples as possible. Studies were conducted to determine if there is breakthrough using the Tenax material and multiple GC cycles were run using a single tube. The determination was made that there is little risk of breakthrough or carryover on the sorbent tube.

b. Additional solid sorbent tube referee methods. A method using solid sorbent tubes (SST) for sample collection was developed as a secondary method that could be used with or in place of a near real-time method. The solid sorbent tube method was developed in parallel to the near real-time methods. Glass SSTs (6 mm outside diameter) filled with Chromosorb 106 sorbent were used. The SSTs are solvent extracted in acetonitrile. The extractant is analyzed by an Agilent Liquid Chromatograph with a Triple Quadrupole Mass Selective Detector (LC-3Q).

c. Near Real-time Referee Methodology. Three MINICAMS were modified to use 7-m columns (with 0.32-mm column diameter). The DB-1701 column provided the best resolution for the compounds. It is a mid-polarity column with a unique cyanopropylphenyl group making up 14% of the stationary phase. It was discovered that the MINICAMS was hardcoded for a maximum purge mode time of 10 minutes. The software allowed an operator to set a purge

APPENDIX A. EMERGING THREAT AGENT VAPOR TESTING CONSIDERATIONS.

mode to any length of time but when the setpoint was transferred to the MINICAMS it defaulted to 10 minutes. A second limitation was the maximum column temperature. The best results on a benchtop GC were observed when the column temperature was over 225 °C. The MINICAMS have an independent protection circuit incorporated into all of the heated zones. This prevents a heated zone from “running away” and causing thermal damage to the MINICAMS. This circuit would trip and shut off power to all of the heated zones if the column temperature exceeded 205 °C. Instead of deactivating the circuit the maximum column temperature was set to 200 °C.

d. Fast GC/mass spectrometer (MS) Referee Methodology. This method used an Agilent 5975T fast GC/MS with a CDS ACEM9350 continuous sampling system. This system was configured with a 30 m × 0.25 mm DB-624 resistively-heated column. Resistive heating is performed by wrapping a standard fused silica column with a resistive heating coil. This heating technique does not require a large oven and can provide very fast heating rates of 300-400 °C/min. Because there is not a large oven, the cool down time of the system is much shorter than a regular convective GC. All of these features help shorten runtimes without sacrificing resolution. The Fast GC/MS system may be operated in selective ion monitoring (SIM) or full scan modes. The best quantitation is obtained using the SIM mode. Each of the four compounds tested were completely resolved using the Fast GC/MS. The overall sensitivity is equivalent to the sensitivity of the MINICAMS. The system’s performance was equivalent with the MINICAMS performance. The main drawback compared to the MINICAMS is the data collection system. The GC/MS can interface with either Chemstation or MassHunter. Like a traditional Agilent GC/MS the software expects discrete samples so sequence files containing general sampling information must be created prior to sample collection and analysis. The sequence file must contain enough samples to cover the anticipated sampling duration. There is also no standard automated data analysis process although there are many macro programming options and custom reports available to aid in the processing of monitoring samples. A lot of time is required to create an automated sample processing system to handle the sampling data.

A.5 SAMPLE LINE VERIFICATION.

A near real-time method requires that a sample be transported through a sample line to the instrument for collection and analysis. Several different types of sampling lines were evaluated. Each line was 5 m long, heat traced along the entire length of the line, and covered in insulation. Each of the lines were heated to about 70 °C. After the lines were heated QC samples were injected into the distal end. This process was repeated several times. The average recoveries for each near real-time system were in the range of 80-85 percent. One key factor is the sampling time must be long enough to allow the challenge to evaporate before the sampling period has ended. This requirement can be facilitated by heating a small section (10 centimeters) of the distal end with extra heat trace to increase the temperature to around 140 °C. This aids in evaporating the challenge within the first sampling cycle. This double heated end is not required when monitoring vapor concentrations because the chemical is already in a vapor state.

APPENDIX B. GLOSSARY.

<u>Term</u>	<u>Definition</u>
Analyte	A substance or chemical constituent that is undergoing analysis.
Calibration	A comparison between measurements, one of which is a measurement standard of known accuracy, to detect, correlate, adjust, and report any variation in the accuracy of the item(s).
Chamber	A natural or artificial enclosed space or cavity.
Confidence check	A means to check the SUT to ensure correct functionality and performance during operation through the use of a simulant.
Data quality objectives	A systematic, scientific method to establish data quality criteria and performance specifications for decision making.
Distribution Manifold (DM)	A piece of equipment that is used to equally distribute the airstream containing the trial analyte and background material (if present) to multiple SUTs and referee probes/sample lines.
False alarm	In the event that the referee system indicates no analyte is present and the SUT signals the presence of an analyte.
Time to alarm	The time it takes the SUT to respond when exposed to a constant concentration of an analyte.
Test Fixture (Apparatus)	A group or combination of instruments, machinery, tools, materials, etc., having a particular function or intended for a specific use.
Time to clear down	The time it takes the SUT to stop alarming once the challenge concentration drops below detectable levels (as determined by the referee system).

APPENDIX C. ABBREVIATIONS.

AD No.	accession number
AR	Army Regulation
ATEC	U.S.Army Test and Evaluation Command
°C	degrees Celsius
CB	chemical-biological
CDD	capability development document
CWA	chemical warfare agent
DA	Department of the Army
DAG	data authentication group
DAS	data acquisition system
DM	distribution manifold
DOE	design of experiments
DQO	data quality objective
DTIC	Defense Technical Information Center
DTP	detailed test plan
g/m ³	grams per cubic meter
GC	gas chromatograph
IAW	in accordance with
iwg	inch water gauge
LC-3Q	liquid chromatograph with triple quadrupole mass selective detector
mg/m ³	milligram per cubic meter
mm	millimeter
MS	mass spectrometer
PAM	pamphlet
PFA	perfluoroalkoxy
PPE	personal protective equipment
QA	quality assurance
QC	quality control
SDS	safety data sheet
SEP	system evaluation plan
SIM	selective ion monitoring
SOP	standing operating procedure
SST	solid sorbent tube
SUT	system under test

APPENDIX C. ABBREVIATIONS

T&E WIPT	test and evaluation working integrated product team
TECMIPT	Test and Evaluation Capabilities and Methodologies Integrated Process Team
TEMP	test and evaluation master plan
TIC	toxic industrial chemical
TIR	test incident report
TOP	Test Operations Procedure
V&V	verification and validation
WVC	water vapor content
ΔP	differential pressure

APPENDIX D. REFERENCES.

1. US Army Test and Evaluation Command (ATEC), Aberdeen Proving Ground, Maryland, Test Operations Procedure (TOP) 08-2-054A Chemical Aerosol Point Detector Testing, 11 October 2022.
2. US Army Test and Evaluation Command (ATEC), Aberdeen Proving Ground, Maryland, Test Operations Procedure (TOP) 08-2-196A Simulant Selection for Laboratory, Chamber, and Field Testing, 3 May 2022.
3. Headquarters, US Army, Washington DC, Army Regulation (AR) 385-10, The Army Safety Program, 24 February 2017.
4. Headquarters, US Army, Washington DC, Department of the Army (DA) Pamphlet (PAM) 385-61, Toxic Chemical Agent Safety Standards, 1 November 2018.
5. Headquarters, US Army, Washington DC, Department of the Army (DA) Pamphlet (PAM) 385-30, Risk Management, 2 December 2014.
6. TOP 08-2-500A, Receipt and Inspection of Chemical-Biological (CB) Materiel, 31 August 2017.

APPENDIX E. APPROVAL AUTHORITY.

CSTE-CIP (73-1jj)

10 March 2023

MEMORANDUM FOR

Commander, U.S. Army White Sands Missile Range
Executive Director, U.S. Army Evaluation Center
Commander, U.S. Army Operational Test Command
Commander, U.S. Army Yuma Proving Ground
Commander, U.S. Army Dugway Proving Ground
Commanders, U.S. ATEC Test Centers
Director, U.S. ATEC Tropic Regions Test Center
Director, U.S. ATEC West Desert Test Center

SUBJECT: Test Operations Procedure 08-2-188A

1. Test Operations Procedure (TOP) 08-2-188A Chemical Point Detector Vapor Testing, has been reviewed by the U.S. Army Test and Evaluation Command (ATEC) Test Centers, the U.S. Army Operational Test Command, and the U.S. Army Evaluation Center. All comments received during the formal coordination period have been adjudicated by the preparing agency.
2. Scope of the document. This TOP provides the current standard methods for chemical point detector testing and assessment with and without operational background materials for vapor hazards including toxic industrial chemicals (TIC), chemical warfare agents (CWA), emerging threats, and CWA simulants.
3. This document is approved for publication and has been posted to the Reference Library of the ATEC Vision Digital Library System (VDLS). The VDLS website can be accessed at <https://vlds.atec.army.mil/>.
4. Comments, suggestions, or questions on this document should be addressed to U.S. Army Test and Evaluation Command (CSTE-CI), 6617 Aberdeen Boulevard-Third Floor, Aberdeen Proving Ground, MD 21005-5001; or e-mailed to usarmy.apg.atec.mbx.atec-standards@army.mil.

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MICHAEL K. DICKERSON
Director, Capabilities Integration

APPENDIX E. APPROVAL AUTHORITY.

**TECMIPT Test Operations Procedure (TTOP)
08-2-188A Chemical Point Detector Vapor Testing**

The Chemical Detection (CD) Capability Area Process Action Team (CAPAT) recommends approval of the TECMIPT Test Operations Procedure (TTOP) 08-2-188A. If a representative non-concurs, a dissenting position paper will be attached.

Organization	Signature	Date
Secretary of the Army (Secretariat) Under Secretary for Test and Evaluation (SAUS-TE)	FRIESE,AMY.JEAN.112 7559216 Amy Friese	_____
Joint Program Executive Office of Chemical Biological Defense (JPEO-CBD) Test & Evaluation	RYBAK,JOSEPH.1 364953735 Joseph Rybak	_____
Joint Requirements Office for Chemical, Biological, Radiological and Nuclear Defense (JRO-CBRND)	BULSON,CHRISTOPHER.D.12363254 HER.D.1236325462 Lt Col Christopher D. Bulson	3 June 2022
Joint Science and Technology Office (JSTO)	ODELL,BRETT.KYLE .1274940553 Brett Odell	8 June 22
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Forward comments, recommended changes, or any pertinent data which may be of use in improving this publication to the following address: Policy and Standardization Division (CSTE-CI-P), U.S. Army Test and Evaluation Command, 6617 Aberdeen Boulevard, Aberdeen Proving Ground, Maryland 21005-5001. Technical information may be obtained from the preparing activity: Commander, West Desert Test Center, U.S. Army Dugway Proving Ground, ATTN: TEDT-DPW, Dugway, UT 84022-5000. Additional copies can be requested through the following website: <https://www.atec.army.mil/publications/documents.html>, or through the Defense Technical Information Center, 8725 John J. Kingman Rd., STE 0944, Fort Belvoir, Virginia 22060-6218. This document is identified by the accession number (AD No.) printed on the first page.