Designation: E2520 – 21

Standard Practice for Measuring and Scoring Performance of Trace Explosive Chemical Detectors[1]

This standard is issued under the fixed designation E2520; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice may be used for measuring, scoring, and improving the overall performance of detectors that alarm on traces of explosives on swabs. These explosive trace detectors (ETDs) may be based on, but are not limited to, chemical detection technologies such as ion mobility spectrometry (IMS) and mass spectrometry (MS).

1.2 This practice considers instrumental (post-sampling) trace detection performance, involving specific chemical analytes across eight types of explosive formulations in the presence of a standard background challenge material. This practice adapts Test Method E2677 for the evaluation of limit of detection, a combined metric of measurement sensitivity and repeatability, which requires ETDs to have numerical responses.

1.3 This practice considers the effective detection throughput of an ETD by factoring in the sampling rate, interrogated swab area, and estimated maintenance requirements during a typical eight hour shift.

1.4 This practice does not require, but places extra value on, the specific identification of targeted compounds and explosive formulations.

1.5 The functionality of multi-mode instruments (those that may be switched between detection of trace explosives, drugs of interest, chemical warfare agents, and other target compounds) may also be tested. A multi-mode instrument under test shall be set to the mode that optimizes operational conditions for the detection of trace explosives. This practice requires the use of a single set of ETD operational settings for calculating a system test score based on the factors described in 1.2, 1.3, and 1.4. A minimum acceptable score is derived from criteria established in Practice E2520 – 07, and an example of such a test is presented in Appendix X1 (Example 2).

1.6 *Intended Users*—ETD developers and manufacturers, testing laboratories, and international agencies responsible for enabling effective deterrents to terrorism.

1.7 Actual explosives as test samples would be preferable, but standard explosive formulations are not widely available, nor are methods for depositing these quantitatively and realistically on swabs. This practice considers sixteen compounds that are available from commercial suppliers. This does not imply that only these sixteen are important to trace detection. Most ETDs are able to detect many other compounds, but these are either chemically similar (hence redundant) to the ones considered, or are unavailable from commercial suppliers for reasons of stability and safety. Under typical laboratory practices, the sixteen compounds considered are safe to handle in the quantities used.

1.8 This practice is not intended to replace any current standard procedure employed by agencies to test performance of ETDs for specific applications. Those procedures may be more rigorous, use different compounds or actual explosive formulations, employ different or more realistic background challenges, and consider environmental sampling procedures and other operational variables.

1.9 This practice recommends one method for preparation of test swabs, pipetting, because this method is simple, reproducible, quantitative, documented, and applicable to most current detection technologies. Other methods, such as inkjet printing and dry transfer, may generate more realistic analyte distributions and particle sizes, but these methods are not widely available and less familiar. They may be used if the procedures are validated and documented properly.

1.10 With any deposition method, some compounds are difficult to present to the ETD inlet quantitatively due to volatility and loss during the swab preparation process. Problematic issues pertinent to this practice are identified along with recommended instructions.

1.11 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.12 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the*

[1] This practice is under the jurisdiction of ASTM Committee E54 on Homeland Security Applications and is the direct responsibility of Subcommittee E54.01 on CBRNE Sensors and Detectors.

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responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.

1.13 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E1154 Specification for Piston or Plunger Operated Volumetric Apparatus

E2677 Test Method for Estimating Limits of Detection in Trace Detectors for Explosives and Drugs of Interest

E2771 Terminology for Homeland Security Applications

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *alarm, n*—visual or audible response, or both, from an ETD that signifies the detection of an explosive.

3.1.2 *ambient background, n*—particular mixture of environmental substances (dust, dirt, etc.) that is collected during swab sampling.

3.1.2.1 *Discussion*—The chemical background collected on swabs is expected to be highly variable, compositionally and temporally, and comprised of a nearly unlimited number of possible chemical species and formulations. Background challenge materials (BCMs) should mimic important types of chemical background found in ETD deployment areas.

3.1.3 *background challenge material, BCM, n*—a standard natural matrix material applied on a test swab to challenge the detection performance of an ETD.

3.1.3.1 *Discussion*—A BCM should be a well-documented material that closely mimics the ambient background typically collected during swab sampling. Many certified reference materials, derived from a variety of natural matrices and processed to offer stable and reproducible characteristics, are internationally available from standards suppliers. The BCMs recommended here are Standard Reference Materials (SRMs). While these represent a limited number of natural matrices, they are compositionally complex and offer fair detection challenges to ETDs.

3.1.3.2 *Discussion*—For the purpose of verifying that an ETD meets minimum performance requirements, the presence of a BCM on the test swab is optional.

3.1.4 *clear-down, n*—the process of allowing an ETD to recover from an alarm through a repeated sequence of automated cleansing to clear out the residual sample from the instrument until the signal is reduced below a set threshold.

3.1.4.1 *Discussion*—May also be used as a verb, for example: "Enough time was allowed to clear-down the ETD."

3.1.5 *compound identity calibration (CIC), n*—act of providing the detector with a known substance so that the internal software parameters may be adjusted to identify explosive compounds correctly.

3.1.5.1 *Discussion*—Manufacturers of explosives detectors often provide so-called calibration media. In an IMS instrument, CIC allows the instrument to adjust the present values of the mobility (or drift) time of the calibrant to the most current conditions. For explosives detectors based on MS, CIC is often called tuning. Some IMS and MS explosives detectors may have built-in materials and software to perform CIC automatically.

3.1.6 *explosive trace detector (ETD), n*—a system designed to detect trace amounts (micrograms or less) of explosive compounds.

3.1.6.1 *Discussion*—In the context of this practice, an ETD under test will require the use of sample swabs. Some ETDs may sample vapors or particles directly from air or surfaces without swabs. This type of sample introduction involves environmental sampling procedures that this practice does not consider.

3.1.7 *limit of detection (LOD), n*—the lowest quantity of a substance that can be distinguished from the absence of that substance within a stated confidence limit.

3.1.7.1 *Discussion*—The LOD90A is the limit of detection for alarm, the mass of a particular analyte that elicits a detection alarm 90 % of the time in a particular ETD, while process blanks elicit alarms less than 10 % of the time.

3.1.7.2 *Discussion*—LOD90A values will be dependent on the alarm rules and response thresholds set in an ETD for each analyte. By default, these rules and thresholds are normally established by the manufacturer, and may be changed by the users.

3.1.7.3 *Discussion*—LOD90A values are distinguished from LOD90 values (the subject of Test Method E2677) in that the latter are 90 % limits of detection for channel signals, intrinsic to the ETD, and independent of alarm rules and alarm thresholds.

3.1.7.4 *Discussion*—LOD90A values are usually greater in value than LOD90 values because the alarm rules and thresholds in ETDs are normally set to avoid false alarms from a wide range of ambient background substances.

3.1.7.5 *Discussion*—LOD90A or LOD90 values may be calculated from appropriate measurement data through the website https://www-s.nist.gov/loda.

3.1.8 *process blank swab, n*—sample swab that has been dosed with the chosen BCM.

3.1.8.1 *Discussion*—For the sole purpose of verifying that an ETD meets minimum performance requirements, a process blank swab may be dosed with pure solvent.

3.1.9 *swabs, n*—sampling media that are made from various types of materials, including fabric and paper, that are supplied by the equipment manufacturer or other parties.

3.1.9.1 *Discussion*—Also referred to as sample traps, sample tickets, swipes, wipes, coupons, filters, tokens, and substrates by some manufacturers of ETDs.

3.1.9.2 *Discussion*—Swabs are used either manually (held

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

with gloved fingers) or placed in wands to collect sample residues for analysis in ETDs.

3.1.9.3 *Discussion*—With manual or wand use, swabs have an active area where sample is collected. Additionally, swabs have an interrogated area that is analyzed by the ETD, either through thermal desorption, scanning, or other means. These two areas are not always spatially congruent. The intersection of the active sampling area and the ETD interrogation detection area is called the effective area (EA).

3.1.9.4 *Discussion*—After swab sampling, the only collected sample that is effectively analyzed is in the EA, so a larger EA is beneficial to trace detection and is therefore factored into the scoring criteria. The location and size of the EA may vary considerably in different ETDs, and may be identified by the manufacturer of the ETD.

3.1.10 *swab support, n*—holder for a swab that prevents contact of the back side of the EA with any surface that might contaminate the swab or wick away solution.

3.1.11 *test score, n*—a metric of general detection performance for an ETD, which combines factors of scope, measurement sensitivity, selectivity, repeatability, and EA throughput.

3.1.11.1 *Discussion*—There is no maximum limit to a test score; improvements in scope, SSRs, and ESRs will result in higher scores.

3.1.12 *test solution, n*—dilute solution of a single explosive compound dissolved in a semivolatile solvent.

3.1.13 *test swab, n*—a sample swab that has been dosed with the BCM and target compound within the EA.

3.1.14 *wand, n*—a hand-held narrow rod that holds a removable swab, used for probing and sampling residues on surfaces.

3.1.14.1 *Discussion*—Some wands are designed by ETD manufacturers to fit into the sampling port of the ETD.

3.2 *Acronyms:*

3.2.1 *AN, n*—ammonium nitrate

3.2.2 *BCM, n*—background challenge material (see 3.1.3).

3.2.3 *CAN, n*—calcium ammonium nitrate [$5Ca(NO_3)_2+NH_4NO_3+10H_2O$]

3.2.4 *CIC, n*—compound identity calibration

3.2.5 *COTS, n*—commercial off-the-shelf

3.2.6 *EA, n*—effective area of the swab (see 3.1.9)

3.2.7 *ESR, n*—combined metric for effective sampling rate performance (see 6.6 and 6.8)

3.2.8 *EtC, n*—ethyl centralite (IUPAC: 1,3-diethyl-1,3-diphenylurea)

3.2.9 *ETD, n*—explosive trace detector (see 3.1.6)

3.2.10 *ETN, n*—erythritol tetranitrate (IUPAC: [(2R, 3R)-1, 3,4-Trinitrooxybutan-2-yl] nitrate)

3.2.11 *HMTD, n*—hexamethylene triperoxide diamine (IUPAC: 3,4,8,9,12,13-Hexaoxa-1,6-diazabicyclo[4.4.4] tetradecane)

3.2.12 *HMX, n*—high melting explosive (IUPAC: Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine)

3.2.13 *IMS, n*—ion mobility spectrometry

3.2.14 *$KClO_4$, n*—potassium perchlorate

3.2.15 *KNO_3, n*—potassium nitrate

3.2.16 *LOD90A, n*—limit of detection for 90 % alarm rate (see 3.1.7)

3.2.17 *MS, n*—mass spectrometry

3.2.18 *$NaClO_3$, n*—sodium chlorate

3.2.19 *NG, n*—nitroglycerin (IUPAC: 1,2,3-Trinitroxypropane)

3.2.20 *OEM, n*—original equipment manufacturer

3.2.21 *PETN, n*—pentaerythritol tetranitrate (IUPAC: [3-Nitrooxy-2,2-bis(nitrooxymethyl)propyl] nitrate)

3.2.22 *RDX, n*—research department explosive (IUPAC: 1,3,5-Trinitroperhydro-1,3,5-triazine)

3.2.23 *R-salt, n*—cyclotrimethylenetrinitrosamine (IUPAC: hexahydro-1,3,5-trinitroso-1,3,5-triazine)

3.2.24 *SRM, n*—Standard Reference Material, certified and distributed by the National Institute of Standards and Technology, Gaithersburg, MD, USA.

3.2.25 *SSR, n*—combined metric for sensitivity/selectivity/repeatability performance (see 6.8)

3.2.26 *TATP, n*—triacetone triperoxide (IUPAC: 3,3-Dimethyl-1,2-dioxacyclopropane)

3.2.27 *Tetryl, n*—2,4,6-trinitrophenylmethylnitramine (IUPAC: *N*-methyl-*N*,2,4,6-tetranitroaniline)

3.2.28 *TNT, n*—trinitrotoluene (IUPAC: 2-Methyl-1,3,5-trinitrobenzene)

3.3 *General Terms:*

3.3.1 Please refer to Terminology E2771.

4. Summary of Practice

4.1 Based on the capabilities of the ETD detection technology, select particular target compounds to be measured and the identity of BCM.

4.2 Prepare reference solutions, each containing a known concentration of a particular target compound.

4.3 Assure all target compounds are programmed into the ETD under test, and set standard operating conditions.

4.4 Pretreat each test swab with 100 µg of BCM.

NOTE 1—For the sole purpose of verifying that an ETD meets minimum performance requirements, the presence of a BCM is optional.

4.5 Using the manufacturer's instructions, perform steps to assure that the ETD is in operational readiness. This may involve compound identity calibration (CIC), verification, and minor tuning. Note the time needed to perform these tasks.

4.6 Analyze twenty-five process blank swabs to determine the background response and the basic sampling rate.

4.7 Determine the LOD90A for each target compound selected. Via pipette or syringe, place BCM and target compound within the EA of the swab as defined by the ETD manufacturer. Between analyses, note the time to recalibrate, retune, and troubleshoot the ETD system in order to maintain operational readiness.

4.8 Calculate an ETD score through a formula using the LOD90A values achieved for each target compound, the selectivity of each alarm, and the effective rate of sample throughput.

5. Significance and Use

5.1 This practice may be used to accomplish several ends: to establish a worldwide frame of reference for terminology, metrics, and procedures for reliably determining trace detection performance of ETDs; as a demonstration by the vendor that the equipment is operating properly to a specified performance score; for a periodic verification by the user of detector performance after purchase; and as a generally-acceptable template adaptable by international agencies to specify performance requirements, analytes and dosing levels, background challenges, and operations.

5.2 It is expected that current ETD systems will exhibit wide ranges of performance across the diverse explosive types and compounds considered. As in previous versions, this practice establishes the minimum performance that is required for a detector to be considered effective in the detection of trace explosives. An explosives detector is considered to have "minimum acceptable performance" when it has attained a test score of at least 80.

6. Procedure

6.1 *Selections:*

6.1.1 Given a particular ETD system running under a single set of operational conditions (or automated control of those conditions), choices must be made regarding the analytes and BCM to be used for the tests. This flexibility in the practice allows a significant increase in scope of the explosives considered without requiring an excessive test workload, and also allows avoidance of any particular BCM that causes difficulties with any particular detection technology. Eight types of explosives are identified in Table 1, along with sixteen chemical compounds that are associated with these types. No more than one compound from each type may be chosen for a maximum of eight compounds for testing. One BCM must also be selected from the list in Table 2 unless the sole purpose of the test is to verify minimum acceptable performance.

6.2 *Reagents and Materials:*

6.2.1 *Swabs*—Procure a sufficient quantity of clean swabs (that are designed for the ETD model under test) from the OEM or other party provider. At a minimum, expect to use 30 swabs per target compound plus 25 swabs to measure sampling rate and process blank response.

TABLE 1 Compounds Associated with Explosive Types

Chemical Class or Explosive Type	Target Compounds
Nitramines	RDX, HMX
Nitro-esters	PETN, ETN
Nitro-aromatics	TNT, Tetryl
Nitrosamines	R-salt
Peroxides	HMTD, TATP
Nitrates	AN, CAN, KNO_3
(Per)chlorates	$NaClO_3$, $KClO_4$
Smokeless powders	NG, EtC

TABLE 2 Standard Materials Associated with Background Types

Background Type of BCM	Standard Materials
Watershed sediment (integrated large-area chemical background)	SRM 2703 (Sediment for solid sampling) SRM 1646a (Chesapeake Bay sediment) SRM 1944 (NY-NJ waterway sediment)
Agricultural soil	SRM 2709a (San Joaquin soil) SRM 2586 (Garden soil)
Domestic dust	SRM 2585 (House and hotel dust)
Atmospheric particulate matter (contain nitrates from combustion processes)	SRM 1648 (St. Louis air particulate) SRM 1649 (Washington DC urban dust) SRM 2975 (Diesel particulate matter, industrial forklift)

6.2.2 *Swab Supports*—Obtain trays or other items designed to hold (and organize) the swabs so that BCM and target compound may be dispensed onto the EA and solvent evaporated quickly without risk of contamination.

6.2.3 *BCM Suspension*—Prepare the BCM suspension by weighing out 400 mg of the solid BCM and placing it into an appropriately sized squeeze bottle with conical lid, then adding 100 mL of analytical-grade isopropanol. Seal and shake well. Pure isopropanol may be used without BCM if the sole purpose of the test is to verify minimum acceptable performance (see Appendix X1, Example 2). In this case, subsequent references to "BCM" and "suspensions" shall refer to a process blank prepared with pure isopropanol. As needed, transfer the suspension quickly into a small plastic squeeze dropper bottle for dispensing. Properly shaken, a typical drop of 25 µL will contain about 100 µg of suspended (and partially dissolved) BCM. This amount is ten times higher than the highest trace analyte testing level, and represents a reasonable amount of ambient background collected during swab sampling. As isopropanol can form peroxides over time, only freshly opened bottles and freshly prepared suspensions should be used.

6.2.4 *Test Solutions*—Prepare test solutions of the selected target compounds in amber glass bottles, each solution made from progressive dilutions of commercially available single-component standard solutions as described in Test Method E2677 (and references therein). Perform dilutions with compatible analytical-grade solvents with vapor pressures appreciably higher than the solutes, resulting in test solutions with concentrations from 0.01 to 100 ng/µL. Store under refrigeration. The shelf-life of these solutions shall be no longer than the shelf-life specified on the commercial standard stock solutions from which they are made.

6.2.5 *Dispensing Device*—A precision dispensing device, such as an inkjet dispenser or a positive displacement volumetric delivery device of 10 µL or less, is needed to accurately deposit aliquots of analyte solution onto BCM-treated swabs (Specification E1154). For some swab materials (for example, meta-aramid fabrics), the aliquot amount should be 3 µL or less to control wicking and spread of solution within the EA.

6.3 *Determination of Background Response and Basic Sampling Rate*—The first procedure is to determine the ETD response to the BCM and the typical throughput rate for samples that elicit no alarms. If the ETD has a problem with a particular BCM (presence of alarms, high background signals, or clear-down issues), it is important to select another BCM. If all BCMs present excessive challenges to an ETD, the problem is likely with the ETD or its operational settings.

6.3.1 *Preparation of Work Space:*

6.3.1.1 Cover table or bench surface with clean, absorbent, disposable material.

6.3.1.2 Care should be taken not to contaminate the swabs. Handling with either unused gloves or clean tweezers is recommended. It is particularly important not to touch the EA of the swab.

6.3.1.3 Provide appropriate means of disposal of used test swabs and other consumables.

6.3.1.4 Swab supports are needed. Each support will provide a means for allowing the suspensions/solutions to evaporate completely from the swab without losses to the supporting surfaces. Label each of the four swab supports for the identity of the explosive or blank solution to be tested for exclusive use with that solution. This will prevent cross-contamination of the explosives on the holder.

6.3.1.5 In the case in which process blank swabs are not used immediately, have labeled containers that are of an appropriate size, are clean, and have covers. Glass or metal containers (including aluminum foil) are appropriate for this task.

6.3.2 *Preparation of Process Blank Swabs:*

6.3.2.1 Place 25 clean swabs on the swab supports.

6.3.2.2 Dose each swab with 100 µg of the selected BCM. This may be performed by shaking the plastic squeeze dropper bottle of suspended BCM, then opening the cap, holding the bottle at about 45° angle from horizontal, and squeezing the bottle slowly to deposit one or two drops on a disposable tissue. Then dispense one drop of the well-shaken BCM suspension directly on the EA of each swab. (If this results in excessive spread of solution outside of the EA, then the drop may be placed instead on a clean and smooth PTFE surface, allowed to dry, then the residue transferred to the swab by carefully rubbing the residue onto the EA.)

6.3.2.3 Allow the solvent to evaporate. Wait until the solution is visibly dry on the test swab. Drying times vary from approximately 5 to 20 min or longer, depending on the test swab material and the room temperature. Do not dry the swabs with temperatures above 25°C. Care should be taken to avoid contaminating the prepared swabs with other foreign substances (such as fingerprints, cardboard fibers, and paper tissues).

6.3.2.4 Once the swabs are dry, they should be used quickly, or, if necessary, stored individually in a labeled container. Following preparation, process blank swabs may be stored at room temperature for no longer than 24 h. Longer storage may result in significant changes in the material.

6.3.3 *Background Response and Sampling Rate:*

6.3.3.1 Assure that the ETD is set in the configuration to be used for all tests, is programmed to collect all measurement data automatically, and is in operational readiness. Perform CIC, tuning, verification, and other steps as outlined by the ETD manufacturer. Note time (φ_1) needed for these steps, including clear-down and return to operational readiness, on Data Sheet (Fig. 1). An example of a completed Data Sheet is found in the Appendix X1 (Example 1).

6.3.3.2 As quickly as possible and timed under a stop watch, run the 25 process blank swabs. Run a clean swab between the process blank swabs if the ETD requires this. Note any alarms that occur on the Data Sheet. If more than 10 % (that is, >2) of the process swabs elicit any alarm, the ETD has failed the test.

6.3.3.3 If two or fewer process swabs elicit an alarm, then the swab sampling rate (R) may be calculated as $R = 25/t$, where t is the time interval needed to run 25 process blank samples in the normal manner, which includes analysis, clear-down, and return to operational readiness. Note the values of t and R (samples per minute) on the Data Sheet.

6.4 *Determination of Limit of Detection for Each Analyte*—The following procedure is based on Test Method E2677. Properly determined, the limit of detection is the basis of a performance metric that combines measurement sensitivity, selectivity, and repeatability (SSR).

6.4.1 If possible, look up the alarm threshold value for each target compound in the ETD system table. Note these values on the Data Sheet. If more than one pertinent threshold exists (via a multi-channel alarm rule), split the column and follow each channel during the LOD tests.

6.4.2 Prepare work space as before (see 6.3.1).

6.4.3 *Preparation of Test Swabs for LOD Test:*

6.4.3.1 Place 30 clean swabs on the swab supports.

6.4.3.2 As before, dose each swab with 100 µg of the selected BCM, and allow the solvent to evaporate (see 6.3.2.2 and 6.3.2.3).

6.4.3.3 When the swab is dry, an appropriate aliquot of test solution may be deposited on the EA. The deposit may be placed anywhere within the EA but avoid spreading outside the EA. Review Table 3 for special considerations regarding the target compound selected.

6.4.3.4 Prepare and analyze a test swab (with BCM) containing 10 ng of target compound. If the ETD alarms for that compound then prepare another test swab at 3 ng and repeat. Continue to explore dosing levels, moving up or down in dose until a level is found where the analyte elicits an alarm between 20 % to 80 % of the time for ten consecutive trials. That level will be the starting level for testing the target compound more rigorously.

6.4.3.5 Prepare and analyze twelve test swabs containing target compound (and BCM) at the starting level, running a clean swab as a blank between test swabs. If there is an alarm for the analyte, note the identity of the alarming compound and numerical response (signal) for the pertinent channel (or channels) on the Data Sheet. If there is no alarm, record a zero for that replicate. If there are ten or fewer alarms for that compound, that level will be called the LOW level. If there are eleven or twelve alarms, that level will be called the HIGH level.

6.4.4 Prepare and analyze twelve test swabs containing target compound at the next level above the LOW level (or next level below the HIGH level), running a clean swab as a blank between test swabs. Again, note the numerical responses from alarms on the Data Sheet. The measurements are finished when the LOW level contains ten or fewer pertinent alarms and

Data Sheet (ID__________) *Test Operator*______________________

*ETD Manufacturer and Model Number*______________________, *Serial Number*____________, *Location*______________________, *Test Date*______________

*ETD Configuration*____

*Swab manufacturer and type*______________________, *Area of EA (A)*______________________cm^2 *BCM*______________

*At startup, time needed for CIC, tuning, verification, clear down, and return to readiness (φ1)*______________________ *seconds =*__________*minutes*

*Time needed to run 25 process blank samples and return to readiness (t)*________*seconds =* ____________*minutes R =* ____________*swabs/minute*

*During LOD tests, total time needed for CIC, tuning, verification, clear down, and return to readiness (φ2)*______________________ *seconds =*__________*minutes*

Explosive type	**Nitramine**	**Nitro-ester**	**Nitro-aromatic**	**Nitrosamine**	**Peroxide**	**Nitrate**	**(Per)chlorate**	**Smokeless Pwdr**
Target compound								
BCM alarm response values †								
Alarm selectivity (S)*								
Alarm response threshold‡								
LOW mass__________ng								
Signal -1								
Signal -2								
Signal -3								
Signal -4								
Signal -5								
Signal -6								
Signal -7								
Signal -8								
Signal -9								
Signal -10								
Signal -11								
Signal -12								
HIGH mass__________ng								
Signal -1								
Signal -2								
Signal -3								
Signal -4								
Signal -5								
Signal -6								
Signal -7								
Signal -8								
Signal -9								
Signal -10								
Signal -11								
Signal -12								
LOD90(A) in nanograms								

† None expected. If the BCM elicits more than two alarms (any alarms), the ETD has failed the background test. No LOD90A values may be calculated from such data.

* See 6.5.

‡ Typically found in ETD system tables. If more than one pertinent threshold exists (via multi-channel alarm rule), split column and follow each channel during LOD test.

FIG. 1 Data Sheet

TABLE 3 Technical Issues Affecting Target Compounds

Target Compound	Known Issues with Residue from Evaporation
RDX, HMX, R-salt, Ethyl centralite	A
TNT	B
NG, TATP	C
KNO_3, CAN, $NaClO_3$, $KClO_4$	A,D
AN	A,B,D,E
PETN, ETN, Tetryl	A,E
HMTD	A, F

[A] Residue characteristics such as spatial distribution, particle sizes, and crystal phases may influence the detection technology tested. Characteristics produced by solution evaporation are governed by several factors, including the physical and thermodynamic properties of the solvent and solute, the concentration and volume of solution deposited, the nature of the swab surface, and environmental factors such as temperature and relative humidity. Microscopic evaluation of the deposits on a suitable substrate would be advisable to ensure that the deposits are dry and crystalline and do not vary significantly in physical characteristics from one deposit to the next.

[B] This compound is difficult to crystallize by evaporation in trace amounts, and morphology of residues has been observed to change over time. This can affect detection if detection windows/filters are overly conservative. After evaporation to dryness, test swabs should be used as soon as possible to avoid changes in the target compound, or detection windows/filters should be widened to allow response to the changing target.

[C] These compounds are moderately volatile and are expected to evaporate or sublime quickly from test swabs. Therefore, compatible solvents utilized for dilutions and test solutions should have a vapor pressure significantly higher than the target compound. Test swabs should be used as soon as possible to avoid significant losses of compound. To decrease losses, methods are needed to stabilize deposits of these compounds without influencing detection. If a systematic decrease in signal is observed during replicate analyses, samples must be prepared individually for the tests described in 6.4.3.5 and 6.4.4.

[D] These compounds are fairly strong oxidizers and may react slowly with organic matter found in many BCMs. After evaporation to dryness, test swabs should be used as soon as possible to avoid losses from redox reactions. If possible, segregate the compound deposit from the BCM within the EA.

[E] These compounds are hygroscopic or have difficult drying to a crystalline state. If possible, prepare test materials in a dry environment.

[F] This compound is chemically unstable at temperatures above 50°C. Desorption temperatures and detection of parent/daughter products should be considered.

the HIGH level contains eleven or more pertinent alarms. If either condition is not met, another level must be tested until the criteria are acceptable.

6.4.5 Calculate the LOD90A for the particular target compound using the website https://www-s.nist.gov/loda. Copy and paste from a spreadsheet containing two columns of data: one column of the dosing levels (25 zeroes for process blanks, plus 12 LOW dosing replicates and 12 HIGH dosing replicates) and the other column containing the corresponding channel signals when there was an alarm (and zeroes for non-alarms). If there are intermediate dosing levels they may be entered as well. If multiple channels were involved in the alarm rule, multiple LOD90A values (one for each channel) may be calculated and the lowest LOD90A used for subsequent scoring, although differences are expected to be insignificant.

6.4.6 Repeat these steps (6.4.1 to 6.4.5) for each target compound selected and note the resulting LOD90A values on the Data Sheet. If the ETD needs re-CIC, re-tuning, excessive clear-down time, or other procedures to maintain operational readiness between the LOD determinations, note total time (φ_2) needed for these steps on Data Sheet.

6.5 *Determination of Selectivity Coefficients*—On the Data Sheet, record the value of *S*, the selectivity coefficient for the alarm for each analyte. If more than half of the alarms for an analyte identify the analyte among no more than two choices of compounds, $S = 2$; if more than half of the alarms do not identify the analyte or give more than two choices, $S = 1$. Instead of the specific compound, the alarm may identify the specific explosive formulation or type (such as SEMTEX or Smokeless Powder). If over half the alarms identify the formulation among no more than two choices of formulations, $S = 2$; if more than half of the alarms do not identify the formulation or give more than two choices, $S = 1$. An example of the determination for *S* is found in the Appendix X1 (Example 1).

6.6 *Determination of Effective Sampling Rate*—Another factor critical to security-driven trace detection is the sample surface throughput. The number of swabs that can be run in an 8 h shift and the EA are combined to determine the effective sampling rate (ESR) of an ETD system. $ESR = R \cdot A[1-(\varphi_1 + \varphi_2)/480]$, where *R* is the basic sampling rate of "non-alarm" process blanks (in samples per minute), and *A* is the area of the EA (in cm^2), which is the congruency of the active sampling area and the ETD interrogation detection area. $\varphi_1 + \varphi_2$ are the estimated times (in minutes) needed to calibrate, tune, verify, clear-down, and otherwise maintain the ETD system in operational readiness at the start (φ_1) and during (φ_2) an 8 h shift. It is intended that $\varphi_1 + \varphi_2$ represent the total time during an 8-h shift when an ETD is unavailable for sample analysis. Routine periodic (daily, weekly, monthly) preventive maintenance need not be included. An example of the determination of *A* is found in the Appendix X1 (Example 2).

6.7 *Documentation*—Document all selections, observations, measurements, and LOD results listed on the attached Data Sheet. Assign a unique identification code to this Data Sheet so it may be linked with the separate Scoring Sheet (Fig. 2).

6.8 *Scoring*—Scores are derived from the information on the Data Sheet and calculated on the Scoring Sheet. Table 4 shows the LOD scoring criteria and meanings of the values. Note that LOD scores increase as the magnitude of LOD90A values decrease. The SSR score for each target compound is equal to $\log[10^5/\text{LOD90A}] \cdot S$, which may range from 0 to 10 or more. The ESR score, $R \cdot A[1 - (\varphi_1 + \varphi_2)/480]$ may also range from less than 1 to 10 or more, so the two performance factors are weighted fairly equally. The SSR and ESR scores for each target compound are multiplied together and the SSR·ESR product scores are added across the compounds tested. While there is no maximum score, a test score of 800 would be extraordinary—perhaps beyond the capabilities of current ETDs. Modern COTS ETD systems may be able to achieve 300, whereas a system that receives a test score of 80 would be able to state that it conforms to a standard of minimum performance.

NOTE 2—If the sole purpose of the test is to verify minimum acceptable performance of an ETD, the procedures may be simplified. Instead of determining the limit of detection for alarm (6.4), each target compound is tested against amounts originally specified in Practice E2520 – 07: 3 ng for RDX; 40 ng for PETN; 23 ng for TNT. These amounts are likely larger than the LODs so will result in reduced scores, but if the compounds are detected at these levels the scores will be sufficient and preparation of test samples will be greatly reduced. An example of a Data Sheet and Scoring Sheet is shown in the Appendix X1 (Example 2).