

Designation: <del>E2520 - 15</del> E2520 - 21

# Standard Practice for Measuring and Scoring Performance of Trace Explosive Chemical Detectors<sup>1</sup>

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 ( $\varepsilon$ ) 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). Technologies that use thermodynamic or optical detection are not specifically addressed, but may be adapted into future versions of this practice.
- 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, standards, itch ai/catalog/standards/sist/fc949712-6ba8-40db-8fc3-f8979ce33ebd/astm-e2520-2
- 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. 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

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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. The user should be aware of the possibility that untested scenarios may lead to failure in the determination of reliable test scores.
- 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 responsibility of the user of this standard to establish appropriate safety safety, health, and health 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

**Document Preview** 

2.1 ASTM Standards:<sup>2</sup>

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, <u>and</u> 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

<sup>&</sup>lt;sup>2</sup> 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.



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 (90 % CL) in a particular ETD, while process blanks elicit alarms less than 10 % of the time.

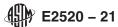
- 3.1.7.2 Discussion—
- LOD90A values will be dependent on the alarm rules and response thresholds set in an ETD for each analyte. By default, these rules and thresholds are normally established by the manufacturer, butand may be changed by the users.
  - 3.1.7.3 Discussion—
- LOD90A values are distinguished 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 higher greater in value than LOD90 values, sincevalues 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 <a href="http://pubapps.nist.gov/loda.">http://pubapps.nist.gov/loda.</a>
  - 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 <del>second</del>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 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 <u>is</u> 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 https://standards.iteh.ai
- 3.2.2 BCM, n—background challenge material (see 3.1.3).
- 3.2.3 CAN, n—calcium ammonium nitrate [5Ca(NO<sub>3</sub>)<sub>2</sub>+NH<sub>4</sub>NO<sub>3</sub>+10H<sub>2</sub>O]

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- 3.2.4 CIC, n—compound identity calibration lards/sist/fc949712-6ba8-40db-8fc3-f8979ce33ebd/astm-e2520-21
- 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 6.56.6 and 6.76.8)
- 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<sub>4</sub>, n—potassium perchlorate

- 3.2.15 KNO<sub>3</sub>, 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<sub>3</sub>, 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 6.76.8)
  - 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)

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- 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 Reference solutions are prepared, 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 thatset standard operating eonditions are set.conditions.
  - 4.4 Each Pretreat each test swab is pretreated 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 Twenty-five Analyze twenty-five process blank swabs are analyzed to determine the background response and the basic sampling rate.
  - 4.7 Determine the LOD90A for each target compound selected. Via pipette or syringe, <u>place</u> BCM and target compound <del>are placed anywhere within the EA of the swab as defined by the ETD manufacturer. Between analyses, <u>note</u> the time <del>is noted to recalibrate, retune, and troubleshoot the ETD system in order to maintain operational readiness.</del></del>
- 4.8 An Calculate an ETD score is calculated 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 The 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; to give developers tangible benchmarks designed to improve detection performance of next-generation 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.
- 5.3 It is not recommended to use scores exclusively to compare different ETD systems in order to make procurement or deployment decisions. The scores themselves signify ratings based on general detection performance, but do not necessarily reflect capabilities with specific analytes or BCMs, nor do scores consider many factors that users may also consider important: procurement and operating costs, robustness and dependability, training requirements, case of use, security features, size and weight constraints, network capabilities and interoperability, and radioactive material management.

# 6. Procedure

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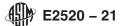
## 6.1 Selections:

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:

**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
Inorganic nitrates	AN, CAN, KNO₃
Nitrates	AN, CAN, KNO <sub>3</sub>
(Per)chlorates	NaClO <sub>3</sub> , KClO <sub>4</sub>
Smokeless powders	NG, EtC



## TABLE 2 Standard Materials Associated with Background Types

BCM Type	Standard Materials
Background Type of BCM	Standard Materials
Watershed sediment (integrated	SRM 2703 (Sediment for solid
large-area chemical background)	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	SRM 1648 (St. Louis air particulate)
(contain nitrates from combustion	SRM 1649 (Washington DC urban
processes)	dust)
	SRM 2975 (Diesel particulate matter, industrial forklift)

- 6.2.1 Swabs—A-Procure a sufficient quantity of clean swabs (that are designed for the ETD model under test) shall be procured from the OEM or second-party-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.
- 6.2.2 Swab Supports—Trays—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 X1As needed, the suspension may be transferred, 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). Dilutions are performed-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 automated pipette or syringe, 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 of 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.