Designation: D5143 – 06 (Reapproved 2015)$^{\varepsilon 1}$

Standard Test Method for
Analysis of Nitroaromatic and Nitramine Explosive in Soil by High Performance Liquid Chromatography[1]

This standard is issued under the fixed designation D5143; 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.

ε^1 NOTE—Reapproved with editorial changes in August 2015.

1. Scope*

1.1 This test method describes a procedure for the laboratory determination of the concentration of nitroaromatic and nitramine explosives in soil. The explosives involved in this test method are as follows: HMX (octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine), RDX (hexahydro-1,3,5-trinitrol-1,3,5-triazine), TNT (2,4,6-trinitrotoluene), TNB (1,3,5 trinitrobenzene), DNB (1,3 dinitrobenzene), tetryl (methyl-2, 4,6-trinitrophenylnitramine), and 2,4-DNT (2,4-dinitrotoluene).

1.2 All observed and calculated values shall conform to the guidelines for significant digits and rounding established in Practice D6026.

1.2.1 The procedures used to specify how data are collected/recorded or calculated, in this standard are regarded as the industry standard. In addition, they are representative of the significant digits that generally should be retained. The procedures used do not consider material variation, purpose for obtaining the data, special purpose studies, or any considerations for the user's objectives; and it is common practice to increase or reduce significant digits of reported data to be commensurate with these considerations. It is beyond the scope of this standard to consider significant digits used in analysis methods for engineering design.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *This standard does not purport to address the safety concerns associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials

D653 Terminology Relating to Soil, Rock, and Contained Fluids

D3740 Practice for Minimum Requirements for Agencies Engaged in Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction

D6026 Practice for Using Significant Digits in Geotechnical Data

E682 Practice for Liquid Chromatography Terms and Relationships

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of common technical terms used in this standard, refer to Terminology D653.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *HPLC*—high power liquid chromatography.

4. Significance and Use

4.1 This test method can be used to make reliable and reproducible measurements in soil in the range from the detection level to the percent levels of each of seven explosive compounds.

4.2 This test method does not attempt to quantify the reactivity or mobility of the explosive content, only the concentration of these compounds in the soil.

4.3 This test method can be used to determine the extent of contamination resulting from the use, misuse, or spillage of explosive compounds. It is useful to determine the effectiveness of clean-up actions at disposal sites, and to determine the environmental impact at explosives disposal, manufacturing, or storage sites.

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[1] This test method is under the jurisdiction of ASTM Committee D18 on Soil and Rock and is the direct responsibility of Subcommittee D18.06 on Physical-Chemical Interactions of Soil and Rock.

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[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.

***A Summary of Changes section appears at the end of this standard**

5. Apparatus

5.1 *Liquid Chromatograph,* conforming to the description and requirements of Practice E682 and equipped with two 25 cm by 4.6 mm reversed-phase HPLC columns (one LC-18, one LC-CN); a fixed 254 nm UV detector; an integrator and a 100 µL sample loop injector.

5.2 Solvent Delivery Module, should be reliable enough for isocratic analysis with flow range capability from 0.1 to 3.0 mL/min.

5.3 *Volumetric Pipets.*

5.4 *Scintillation Vials.*

5.5 *Plastic B-D Syringe,* fitted with a disposable 0.5 µm filter assembly.

5.6 *Vortex Mixer.*

5.7 *Amber Injector Vials.*

5.8 *Mortar and Pestle.*

5.9 *Rubber Tipped Pestle.*

5.10 *Standard no. 10 (2 mm) sieve.*

6. Reagents

6.1 Unless otherwise stated, it is intended that all reagents conform to the specification of the committee on Analytical Reagents of the American Chemical Society.[3]

6.2 *HPLC Grade Methanol.*

6.3 *HPLC Grade Acetonitrile.*

6.4 *ASTM Type I Water.*

6.5 Reference Standards of the following:
6.5.1 HMX,
6.5.2 RDX,
6.5.3 TNT,
6.5.4 DNB (1,3-dinitrobenzene),
6.5.5 TNB (1,3,5-trinitrobenzene),
6.5.6 Tetryl, and
6.5.7 2,4-DNT and 2,6-DNT.

7. Procedure

7.1 *Sample Preparation:*

7.1.1 Air dry the soil to a constant mass out of direct sunlight at normal ambient humidity and 20 to 25°C (room temperature).

7.1.2 Disaggregate the soil using a rubber tipped pestle and mortar, and sieve the soil through a No. 10 sieve to remove the coarser stones and pebbles. Discard only those particles that are not passable through the No. 10 sieve.

7.1.3 Grind the soil using a pestle and mortar.

7.1.4 Sieve the soil through a 30 mesh sieve. Ensure that all of the particles are ground to pass through the sieve openings and are collected prior to continuing.

7.1.5 Thoroughly mix the collected soil fraction and draw a 2.00 gm sample for each test replicate.

7.1.6 Thoroughly clean the sieves, pestles, and mortars with laboratory soap and water followed by an isopropanol rinse between samples.

7.2 *Extraction of Soil:*

7.2.1 Weigh out exactly 2.00 g of soil into a 11.1 mL (6 dram) screw top glass vial equipped with a TFE-fluorocarbon-lined cap.

7.2.2 With a volumetric pipette, introduce 10.0 mL of acetonitrile to the soil, and screw on the closures tightly.

7.2.3 Place the vials on a vortex mixer for 1 min followed by placing the sample in an ultrasonic bath for 18 h.

7.2.4 The ultrasonic bath should be maintained near ambient temperature to minimize loss of tetryl due to thermal degradation. Remove the samples from the bath and allow them to stand for a minimum of 15 min to allow the larger particles to settle.

7.2.5 With a volumetric pipet, remove a 5.00 mL aliquot of the suspension and mix it with a 5.00 mL of 5 g/L aqueous $CaCl_2$ in a glass scintillation vial. Shake the vials and allow to stand for 15 min.

7.2.6 Filter about 5 mL of the clarified sample into a clean scintillation vial by forcing the supernatant through a 0.5 µm filter using a 3 mL disposable syringe. The first millilitre is discarded and the remainder saved for analysis. Place the filtered sample in an amber injection vial for analysis.

7.3 *Liquid Chromatographic Analysis:*

7.3.1 Accomplish the liquid chromatograph separations isocratically by the use of a 5 µm, reversed-phase LC-18 and LC-CN cartridge column, with a 50/50 methanol/water mobile phase, at a flow of 1.5 mL/min. The LC-CN cartridge column is used for confirmation of the analytical results.

7.3.2 Make quantifications at the 254 nm wavelength.

7.3.3 Base quantitation on response factors established by replicate analysis of a single high range standard. Dilute standards, controls, and blanks 1:1 with aqueous $CaCl_2$ prior to analysis.

7.3.4 The elution time for the total assay is less than 15 min.

8. Calculation

8.1 Experience indicates that a linear calibration curve with zero intercept is appropriate for each compound as shown in the references from the Corps of Engineers. Therefore, calibration is accomplished by repeated analysis of a high range standard. The mean response (R) for each compound obtained in the peak height mode is calculated for each analyte. The response factors (RF) are then obtained by dividing each R by the known solution concentration (C) for that compound in units of µg/L.

$$RF = R/C \qquad (1)$$

8.2 The concentrations of analytes in the extracts are obtained by dividing the response of each analyte (R_a) by the appropriate response factor (RF_a).

$$C_a = R_a/RF_a \qquad (2)$$

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[3] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

8.3 The concentration in soil (X_a), on a µg/g basis, is then obtained by multiplying the solution concentration by the total volume of extract (0.010 L) and dividing by the actual mass of dried soil used (M).

$$X_a = (C_a \times 0.010)/M \tag{3}$$

9. Report

9.1 For each soil tested, report the following information:

9.1.1 The name of the person performing the test and the date of the test,

9.1.2 The sample identification,

9.1.3 The specific analyte,

9.1.4 The detection limit, and

9.1.5 The concentration of explosive found in µg/g of dry soil.

TABLE 1 Performance Data for Method for Determination of Nitroaromatics and Nitramines Residues in Soil

Analyte	Retention Time, Min	Detection Limits,[A] µg/g	Bias (Recovery)[B], %	Precision[C], µg/g Within-Lab	Precision[C], µg/g Between-Lab
				1s (2ds)	1s (2ds)
HMX	2.4	1.27	95.4	3.7 (10.5)	5.7 (16.1)
RDX	3.7	0.74	96.8	2.3 (6.5)	4.3 (12.2)
TNB	5.1	0.29	92.0	4.4 (12.4)	6.8 (19.2)
DNB	6.2	0.11	93.0	4.0 (11.3)	6.9 (19.5)
Tetryl	6.9	0.12	74.7	17.9 (50.6)	30.7 (86.8)
TNT	8.4	0.08	96.8	3.5 (9.9)	4.7 (13.3)
2,4-DNT	10.1	0.03	96.0	3.4 (9.6)	4.4 (12.4)

[A] Obtained using EPA MDL procedure at the 99 % confidence level.
[B] Obtained from the slope of the regression line versus known concentrations collaborative tests.
[C] Obtained from collaborative test results.

10. Precision and Bias

10.1 *Precision:*

10.1.1 *Within-Laboratory Precision*—The within-laboratory standard deviations (also called repeatability) for the seven analytes were obtained by means of a collaborative test. The test results were obtained from seven laboratories conducting analysis of eight soils in duplicate. Four of the test soils were field contaminated soils and four were spiked soils. The within-laboratory standard deviation was obtained from the agreement of duplicates[4].Within-laboratory precision estimates are presented in Table 1. Therefore, the results of two properly conducted tests by the same operator with the same equipment on duplicate samples should not be considered suspect unless they differ by more than the values presented in the within lab, 2ds column of Table 1 (See Footnote 7).[4] The data in Table 1 satisfy the 1s and d2s requirements outlined in Practice C670.

10.1.2 *Between-Laboratory Precision*—The between-laboratory precision (also called reproducibility) was obtained from the results of a seven-laboratory collaborative test. The between-laboratory precision estimates (see Table 1) were obtained by the method described in Footnote 8.[5] Therefore, the results of two properly conducted tests by different operators with the different equipment on duplicate samples should not be considered suspect unless they differ by more than the values presented in the between lab, 2ds column of Table 1.

10.2 *Bias*—The procedure in this test method for measuring the explosive content of soils has no bias because the value of the explosive residue content is only defined in terms of the test method. However, to aid the user of this standard in determining bias relative to other methods, the percent recovery was obtained from regression analysis of the four spiked soils analyzed in duplicate in the collaborative test described above. Results are presented in Table 1.

11. Keywords

11.1 army; explosives; liquid chromatography; military; soil

REFERENCES

(1) U.S. Army Corps of Engineers, Cold Regions Research and Engineering Laboratory Report 88-8, Development of an analytical method for the determination of explosive residues in soil, Part II; Additional development and ruggedness testing. July, 1988.

(2) U.S. Army Corps of Engineers, Cold Regions Research and Engineering Laboratory Report 89-9, Development of an analytical method for the determination of explosive residues in soil, Part III; Collaborative test results and final performance evaluation. 1989.

(3) U.S. Army Corps of Engineers, Cold Regions Research and Engineering Laboratory Report 85-15, TNT, RDX, and HMX explosives in soils and sediments, analysis techniques and drying losses. October 1985.

(4) U.S. Army Corps of Engineers, Cold Regions Research and Engineering Laboratory Report 85-22, Comparison of extraction techniques and solvents for explosive residues in soil. November, 1985.

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[4] U.S. Army Corps of Engineers, Cold Regions Research and Engineering Laboratory Report 87-7, Development of an Analytical Method for Explosive Residues in Soil, June 1987.

[5] Youden, W. J. and Steiner, E. H. Statistical Manual of the AOAC, 1978.

SUMMARY OF CHANGES

In accordance with Committee D18 policy, this section identifies the location of changes to this standard since the last edition (D5143 – 06(2010)$^{\varepsilon 1}$) that may impact the use of this standard.

(1) Editorial changes were made throughout as part of the five-year review for this standard.

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