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Standard Test Method for Determination of Diisopropyl Methylphosphonate, Ethyl Methylphosphonic Acid, Isopropyl Methylphosphonic Acid, Methylphosphonic Acid, and Pinacolyl Methylphosphonic Acid in Soil by Pressurized Fluid Extraction and Analyzed by Liquid Chromatography/Tandem Mass Spectrometry[1]

This standard is issued under the fixed designation E2866; 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 procedure covers the determination of Diisopropyl Methylphosphonate (DIMP), Ethyl Methylphosphonic Acid (EMPA), Isopropyl Methylphosphonic Acid (IMPA), Methylphosphonic Acid (MPA), and Pinacolyl Methylphosphonic Acid (PMPA), referred to collectively as organophosphonates (OPs) in this test method, in soil. This method is based upon solvent extraction of a soil by pressurized fluid extraction (PFE). The extract is filtered and analyzed by liquid chromatography/tandem mass spectrometry (LC/MS/MS). OPs are qualitatively and quantitatively determined by this method.

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

1.3 The method detection limit[2] (MDL), electrospray ionization (ESI) mode, and reporting range[3] for the OPs are listed in Table 1.

1.4 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *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:*[4]

D1193 Specification for Reagent Water

D2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water

D5681 Terminology for Waste and Waste Management

E2554 Practice for Estimating and Monitoring the Uncertainty of Test Results of a Test Method Using Control Chart Techniques

2.2 *Other Documents:*

EPA publication SW-846 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods[5]

40 CFR Part 136, Appendix B The Code of Federal Regulations[6]

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D5681.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *analytical column, n*—the particles of the solid stationary phase fill the whole inside volume of a tube (column)

[1] This test method is under the jurisdiction of ASTM Committee D34 on Waste Management and is the direct responsibility of Subcommittee D34.01.06 on Analytical Methods.

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[2] The MDL is determined following the Code of Federal Regulations, 40 CFR Part 136, Appendix B utilizing solvent extraction of soil by PFE. A detailed process determining the MDL is explained in the reference and is beyond the scope of this standard to be explained here.

[3] Reporting range concentrations are calculated from Table 4 concentrations assuming a 100 µL injection of the lowest and highest level calibration standards with a 40 mL final extract volume of a 10 g soil sample. Volume variations will change the reporting limit and ranges. The reporting limit (RL), lowest concentration of the reporting range, is calculated from the concentration of the Level 1 calibration standard as shown in Table 4.

[4] 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.

[5] Available from National Technical Information Service (NTIS), U.S. Department of Commerce, 5285 Port Royal Road, Springfield, VA, 22161 or at http://www.epa.gov/epawaste/hazard/testmethods/index.htm.

[6] Available from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401, http://www.access.gpo.gov.

TABLE 1 Method Detection Limit and Reporting Range

Analyte	ESI Mode	MDL (PPB)	Reporting Range (PPB)
Diisopropyl methylphosphonate	Positive	2.7	40–2000
Ethyl methylphosphonic acid	Negative	2.3	40–2000
Ethyl methylphosphonic acid	Positive	1.3	40–2000
Isopropyl methylphosphonic acid	Negative	5.7	40–2000
Isopropyl methylphosphonic acid	Positive	2.8	40–2000
Methylphosphonic acid	Positive	8.7	40–2000
Pinacolyl methylphosphonic acid	Negative	5.3	40–2000

that the mobile phase passes through using the pressure generated by the liquid chromatography system.

3.2.2 *filter unit, n*—in this standard, a filter that is supported with an inert housing to the solvents as described in Section 7 of this standard.

3.2.3 *filtration device, n*—a device used to remove particles from the extract that may clog the liquid chromatography system. Described in 7.3 of this standard.

3.2.4 *glass fiber filter, n*—a porous glass fiber material onto which solid particles present in the extraction fluid, which flows through it, are largely caught and retained, thus removing them from the extract.

3.2.5 *hypodermic syringe, n*—in this standard, a luer-lock-tipped glass syringe capable of holding a syringe-driven filter unit as described in 7.3 of this standard.

3.2.6 *liquid chromatography (LC) system, n*—in this standard, a separation system using liquid as the mobile phase and a stationary phase packed into a column. The use of small particles packed inside a column and a high inlet pressure enables the separation of components in a mixture.

3.2.7 *organophosphonates (OPs), n*—in this test method, diisopropyl methylphosphonate (DIMP), ethyl methylphosphonic acid (EMPA), isopropyl methylphosphonic acid (IMPA), methylphosphonic acid (MPA), and pinacolyl methylphosphonic acid (PMPA), collectively.

3.2.8 *pressurized fluid extraction, n*—the process of transferring the analytes of interest from the solid matrix, a soil, into the extraction solvent using pressure and elevated temperature.

3.2.9 *reporting range, n*—the quantitative concentration range for an analyte in this standard.

3.2.10 *tandem mass spectrometer, n*—an arrangement in which ions are subjected to two sequential stages of analysis according to the quotient mass/charge.

3.3 *Abbreviations:*

3.3.1 *DIMP*—diisopropyl methylphosphonate

3.3.2 *EMPA*—ethyl methylphosphonic acid

3.3.3 *IMPA*—isopropyl methylphosphonic acid

3.3.4 *LC*—liquid chromatography

3.3.5 *LCS/LCSD*—laboratory control spike/laboratory control spike duplicate

3.3.6 *mM*—millimolar, 1×10^{-3} moles/L

3.3.7 *MPA*—methylphosphonic acid

3.3.8 *MRM*—multiple reaction monitoring

3.3.9 *MS*—matrix spike

3.3.10 *NA*—not applicable

3.3.11 *ND*—non-detect

3.3.12 *PFE*—pressurized fluid extraction

3.3.13 *PMPA*—pinacolyl methylphosphonic acid

3.3.14 *PPB*—parts per billion

3.3.15 *QC*—quality control

3.3.16 *SD*—standard deviation

3.3.17 *SRM*—single reaction monitoring

3.3.18 *VOA*—volatile organic analysis

4. Summary of Test Method

4.1 For OPs soil analysis, samples are shipped to the lab between 0 °C and 6 °C. The samples are to be extracted, filtered, and analyzed by LC/MS/MS within seven days of collection.

4.2 The OPs and the surrogates (diisopropyl methylphosphonate-D_{14}, pinacolyl methylphosphonic acid-$^{13}C_6$, and methylphosphonic acid-D_3) are identified by retention time and one SRM transition. The target analytes and surrogates are quantitated using the SRM transitions utilizing an external calibration. The final report issued for each sample lists the concentration of each organophosphonate target compound and each surrogate recovery.

5. Significance and Use

5.1 This is a performance-based method, and modifications are allowed to improve performance.

5.1.1 Due to the rapid development of newer instrumentation and column chemistries, changes to the analysis described in this standard are allowed as long as better or equivalent performance data result. Any modifications shall be documented and performance data generated. The user of the data generated by this standard shall be made aware of these changes and given the performance data demonstrating better or equivalent performance.

5.2 Organophosphate pesticides affect the nervous system by disrupting the enzyme that regulates acetylcholine, a neurotransmitter. They were developed during the early 19th century, but their effects on insects, which were similar to their effects on humans, were discovered in 1932. Some are poisonous and were used as chemical weapon agents. Organophosphate pesticides are usually not persistent in the environment.[7,8]

5.3 This test method is for the analysis of selected organophosphorous based pesticide degradation products.

5.4 This method has been investigated for use with various soils.

[7] Additional information about organophosphate pesticides is available at http://www.epa.gov (2011).

[8] Additional information about chemical weapon agents is available at http://www.opcw.org (2011).