Designation: D8025 – 23

Standard Test Method for Determination of Select Pesticides in Water by Multiple Reaction Monitoring Liquid Chromatography Tandem Mass Spectrometry[1]

This standard is issued under the fixed designation D8025; 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 test method covers a method for analysis of selected pesticides in a water matrix by filtration followed with liquid chromatography/electrospray ionization tandem mass spectrometry analysis. The samples are prepared in 20 % methanol, filtered, and analyzed by liquid chromatography/tandem mass spectrometry. This method was developed for an agricultural run-off study, not for low level analysis of pesticides in drinking water. This method may be modified for lower level analysis. The analytes are qualitatively and quantitatively determined by this method. This method adheres to multiple reaction monitoring (MRM) mass spectrometry.

1.2 A full collaborative study to meet the requirements of Practice D2777 has not been completed. This standard contains single-operator precision and bias based on single-operator data. Publication of standards that have not been fully validated is done to make the current technology accessible to users of standards, and to solicit additional input from the user community.

1.3 A reporting limit check sample (RLCS) is analyzed during every batch to ensure that if an analyte was present in a sample at or near the reporting limit it would be positively identified and accurately quantitated within set quality control limits. A method detection limit (MDL) study was not done for this method, the method detection limits would be much lower than the reporting limits in this method and would be irrelevant. A RLCS was determined to be more applicable for this standard. If this method is adapted to report much lower or near the MDL then a MDL study would be warranted.

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

1.5 The Reporting Range for the target analytes are listed in Table 1.

1.5.1 The reporting limit in this test method is the minimum value below which data are documented as non-detects. The reporting limit is calculated from the concentration of the Level 1 calibration standard as shown in Table 6 after taking into account an 8 mL water sample volume and a final diluted sample volume of 10 mL (80 % water/20 % methanol).

1.6 *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.*

1.7 *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]

D1129 Terminology Relating to Water
D1193 Specification for Reagent Water
D2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water
D3856 Guide for Management Systems in Laboratories Engaged in Analysis of Water
D4841 Practice for Estimation of Holding Time for Water Samples Containing Organic and Inorganic Constituents
D5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis
E2554 Practice for Estimating and Monitoring the Uncertainty of Test Results of a Test Method Using Control Chart Techniques

[1] This test method is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.06 on Methods for Analysis for Organic Substances in Water.

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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.

TABLE 1 Reporting Range

Analyte	Reporting Ranges, (ng/L)
2,4-D	250–10 000
Acetochlor	250–10 000
Alachlor	250–10 000
Aldicarb	250–10 000
Atrazine	62.5–2 500
Desethylatrazine	62.5–2 500
Desisopropylatrazine	125–5 000
Azoxystrobin	31.2–1 250
Bentazon	250–10 000
Carbaryl	250–10 000
Chlorpyrifos	250–10 000
Clopyralid	25 000–1 000 000
Clothianidin	62.5–2 500
Diazinon	62.5–2 500
Dicamba	12 500–500 000
Fipronil	250–10 000
Imidacloprid	62.5–2 500
Malathion	125–5 000
Methomyl	250–10 000
Metolachlor	62.5–2 500
Metribuzin	125–5 000
Picloram	6 250–250 000
Propiconazole	62.5–2 500
Simazine	62.5–2 500
Tebuconazole	62.5–2 500
Thiamethoxam	62.5–2 500
Triclopyr	1 250–5 000

2.2 *Other Document:*

EPA publication SW-846 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods[3]

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this standard, refer to Terminology D1129.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *batch QC, n*—all the quality control samples and standards included in an analytical procedure.

3.2.2 *independent reference material, IRM, n*—a material of known purity and concentration obtained either from the National Institute of Standards and Technology (NIST) or other reputable supplier.

3.2.2.1 *Discussion*—The IRM must be obtained from a different lot of material than is used for calibration.

3.2.3 *reporting limit, RL, n*—the minimum concentration below which data are documented as non-detects.

3.2.4 *reporting limit check sample, RLCS, n*—a sample used to ensure that if the analyte was present at the reporting limit, it would be confidently identified.

3.3 *Acronyms:*

3.3.1 *CCC, n*—Continuing Calibration Check

3.3.2 *CRW, n*—Chicago River Water

3.3.3 *IC, n*—Initial Calibration

3.3.4 *LC, n*—Liquid Chromatography

3.3.5 *LCS/LCSD, n*—Laboratory Control Sample/Laboratory Control Sample Duplicate

3.3.6 *MDL, n*—Method Detection Limit

3.3.7 *MeOH, n*—Methanol

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

3.3.9 *MRM, n*—Multiple Reaction Monitoring

3.3.10 *MS/MSD, n*—Matrix Spike/Matrix Spike Duplicate

3.3.11 *NA, adj*—Not Available

3.3.12 *ND, n*—non-detect

3.3.13 *P&A, n*—Precision and Accuracy

3.3.14 *ppt, n*—parts-per-trillion

3.3.15 *QA, adj*—Quality Assurance

3.3.16 *QC, adj*—Quality Control

3.3.17 *RL, n*—Reporting Limit

3.3.18 *RLCS, n*—Reporting Limit Check Sample

3.3.19 *RSD, n*—Relative Standard Deviation

3.3.20 *RT, n*—Retention Time

3.3.21 *SDS, n*—Safety Data Sheets

3.3.22 *SRM, n*—Single Reaction Monitoring

3.3.23 *SS, n*—Surrogate Standard

3.3.24 *TC, n*—Target Compound

3.3.25 *VOA, n*—Volatile Organic Analysis

4. Summary of Test Method

4.1 The operating conditions presented in this standard have been successfully used in the determination of the select pesticides in water; however, this standard is intended to be performance based and alternative operating conditions can be used to perform this method provided data quality objectives are attained.

4.2 For pesticide analysis, samples are shipped to the lab on ice and analyzed within 14 days of collection. A sample (8 mL) is transferred to an amber VOA vial, an isotopically labeled pesticide surrogate mix is added to all samples followed by a pesticide spike solution which is added only to the Reporting Limit Check Samples, Laboratory Control and Matrix Spike samples before the addition of methanol. Then 2 mL of methanol is added to each sample and hand shaken or vortexed for 1 min. The samples are then filtered through a PTFE membrane syringe driven filter unit and then analyzed by LC/MS/MS. All concentrations reported only to the reporting limit.

4.3 The analysis of the sample requires two separate analysis methods, one using the LC gradient conditions in Table 2 with the Methanol/Water/Ammonium Formate and the second using the LC gradient conditions in Table 3 with the Methanol/Water/Formic acid. Each analysis set is to be analyzed separately in two different complete sample sequences which includes the exact same samples and may even include the same calibration level standards. The only analytes reported from the formic acid run conditions are 2,4-D, Clopyralid,

[3] 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