Designation: D7730 – 17

Standard Test Method for Determination of Dioctyl Sulfosuccinate in Sea Water by Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS)[1]

This standard is issued under the fixed designation D7730; 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 the determination of dioctyl sulfosuccinate (DOSS) in sea water by direct injection using liquid chromatography (LC) and detection with tandem mass spectrometry (MS/MS). This analyte is qualitatively and quantitatively determined by this test method. This test method adheres to selected reaction monitoring (SRM) mass spectrometry.

1.2 The detection verification level (DVL) and reporting range for DOSS are listed in Table 1.

1.2.1 The DVL is required to be at a concentration at least 3 times below the reporting limit (RL) and have a signal/noise ratio greater than 3:1. Fig. 1 and Fig. 2 display the signal/noise ratio of the selected reaction monitoring (SRM) transition.

1.2.2 The reporting limit is the concentration of the Level 1 calibration standard as shown in Table 5 for DOSS, taking into account the 50 % sample preparation dilution factor.

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 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:*[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

2.2 *Other Standards:*[3]

EPA Publication SW-846 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods

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 *detection verification level, DVL, n*—a concentration that has a signal/noise ratio greater than 3:1 and is at least 3 times below the reporting limit (RL).

3.2.2 *reporting limit, RL, n*—the concentration of the lowest-level calibration standard used for quantification.

3.3 *Abbreviations:*

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

3.3.2 *NA*—no addition

3.3.3 *ND*—non-detect

3.3.4 *ppb*—parts per billion, µg/L

4. Summary of Test Method

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

4.2 For DOSS analysis, samples are shipped to the lab between 0°C and 6°C and analyzed within 5 days. In the lab, the entire collected 20-mL sample is spiked with surrogate,

[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.

[3] Available from National Technical Information Service (NTIS), 5301 Shawnee Rd., Alexandria, VA 22312, http://www.ntis.gov.

TABLE 1 Detection Verification Level (DVL) and Reporting Range

Analyte	DVL (µg/L)	Reporting Range (µg/L)
DOSS	3	20–400

ammonium formate buffer solution and brought to a volume of 40 mL with acetonitrile. This prepared sample is then filtered using a syringe driven filter unit, and analyzed by LC/MS/MS. If visible oil is present, the prepared sample is allowed to settle resulting in an oil layer at the top of the 40-mL solution. A portion of the aqueous (bottom) layer is filtered, leaving the oil layer behind, through a syringe driven filter assembly and analyzed by LC/MS/MS.

4.3 DOSS and DOSS surrogate are quantitated by retention time and one SRM transition. The final report issued for each sample lists the concentration of DOSS and the surrogate recovery.

5. Significance and Use

5.1 DOSS is an anionic detergent that is approved by the United States Food and Drug Administration (U.S. FDA) and is used widely as a laxative, emulsifying, solubilizing, and dispersing agent, and is used in the cosmetic industry.[4] DOSS may also be used as a dispersing agent to treat oil. DOSS may be released into the environment at levels that may be harmful to aquatic life. The U.S. EPA aquatic life benchmark for DOSS is 40 ppb.[5]

5.2 This test method has been investigated for use with reagent and sea water.

6. Interferences

6.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other apparatus producing discrete artifacts or elevated baselines. All of these materials are demonstrated to be free from interferences by analyzing laboratory reagent blanks under the same conditions as samples.

6.2 All glassware is washed in hot water with detergent and rinsed in hot water followed by distilled water. The glassware is then dried and heated in an oven at 250°C for 15 to 30 minutes. All glassware is subsequently cleaned with methanol or 50 % acetonitrile/50 % water, or both.

6.3 System contamination and surface binding are problematic as DOSS is a surface active compound. It is important to thoroughly rinse sample containers with organic solvent to accurately measure DOSS concentrations. Thorough rinsing of all lab equipment is necessary to reduce contamination. Carefully analyze blanks to ensure that the method minimizes DOSS carryover.

6.4 All reagents and solvents should be pesticide residue purity or higher to minimize interference problems.

6.5 Matrix interferences may be caused by contaminants in the sample. The extent of matrix interferences can vary considerably from sample source depending on variations of the sample matrix.

6.6 Sulfonate filters contribute significantly to background interference and should be avoided for this standard. In addition to sample filtration, sulfonate filters may be present in water purification systems.

7. Apparatus

7.1 *LC/MS/MS System:*

7.1.1 *Liquid Chromatography System*—A complete LC system is needed in order to analyze samples.[6] Any system that is capable of performing at the flows, pressures, controlled temperatures, sample volumes, and requirements of the standard may be used.

7.1.2 *Analytical Column*—Waters Atlantis dC18,[7] 2.1 × 150 mm, 3-µm particle size was used to develop this test method. Any column that achieves baseline resolution of these analytes may be used. Baseline resolution simplifies data analysis and can reduce the chance of ion suppression, leading to higher limits of detection. The retention times and order of elution may change depending on the column used and need to be monitored.

7.1.3 *Tandem Mass Spectrometer System*—A MS/MS system capable of MRM analysis.[8] Any system that is capable of performing at the requirements in this standard may be used.

7.2 *Filtration Device:*

7.2.1 *Hypodermic syringe*—A Lock Tip Glass Syringe capable of holding a Millex HV Syringe Driven Filter Unit PVDF 0.22 µm,[9,10] or similar, may be used.

7.2.1.1 A Lock Tip Glass Syringe was used in this test method.

7.2.2 *Filter*—Millex HV Syringe Driven Filter Unit PVDF 0.22 µm, or similar, may be used.

8. Reagents and Materials

8.1 *Purity of Reagents*—High Performance Liquid Chromatography (HPLC) pesticide residue analysis and spectrophotometry grade chemicals shall be used in all tests. Unless indicated otherwise, it is intended that all reagents shall conform to the Committee on Analytical Reagents of the

[4] Code of Federal Regulations—Title 21: Food and Drugs, Part 172, Available from U.S. Government Printing Office, Superintendent of Documents, 732 N. Capitol St., NW, Washington, DC 20401-0001, http://www.access.gpo.gov.

[5] Additional information about DOSS is available at http://www.epa.gov/bpspill/dispersant-methods.html (2010)

[6] A Waters ACQUITY UltraPerformance Liquid Chromatography (UPLC) System, a trademark of the Waters Corporation, Milford, MA, was used to develop this test method. All parameters in this test method are based on this system and may vary depending on your instrument.

[7] The Waters Atlantis dC18 is a trademark of the Waters Corporation, Milford, MA.

[8] A Waters Quattro Premier XE tandem quadrupole mass spectrometer, a trademark of the Waters Corporation, Milford, MA, was used to develop this test method. All parameters in this test method are based on this system and may vary depending on your instrument.

[9] The sole source of supply of the Millex HV Syringe Driven Filter Unit PVDF 0.45 µm known to the committee at this time is Millipore Corporation, Catalog # SLHV033NS. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[10] Millex is a trademark of Merck KGAA, Darmstadt, Germany.

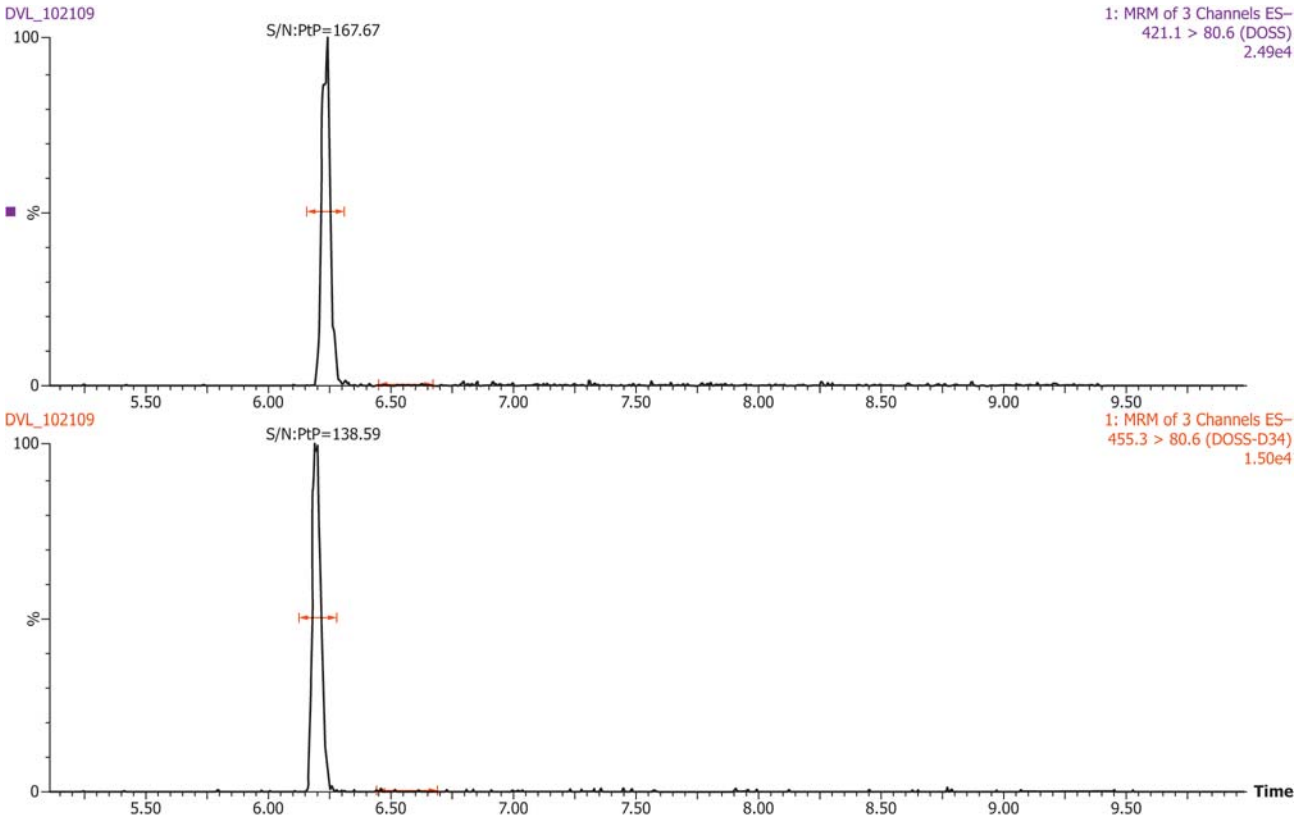

FIG. 1 Detection Verification Level Signal/Noise Ratio

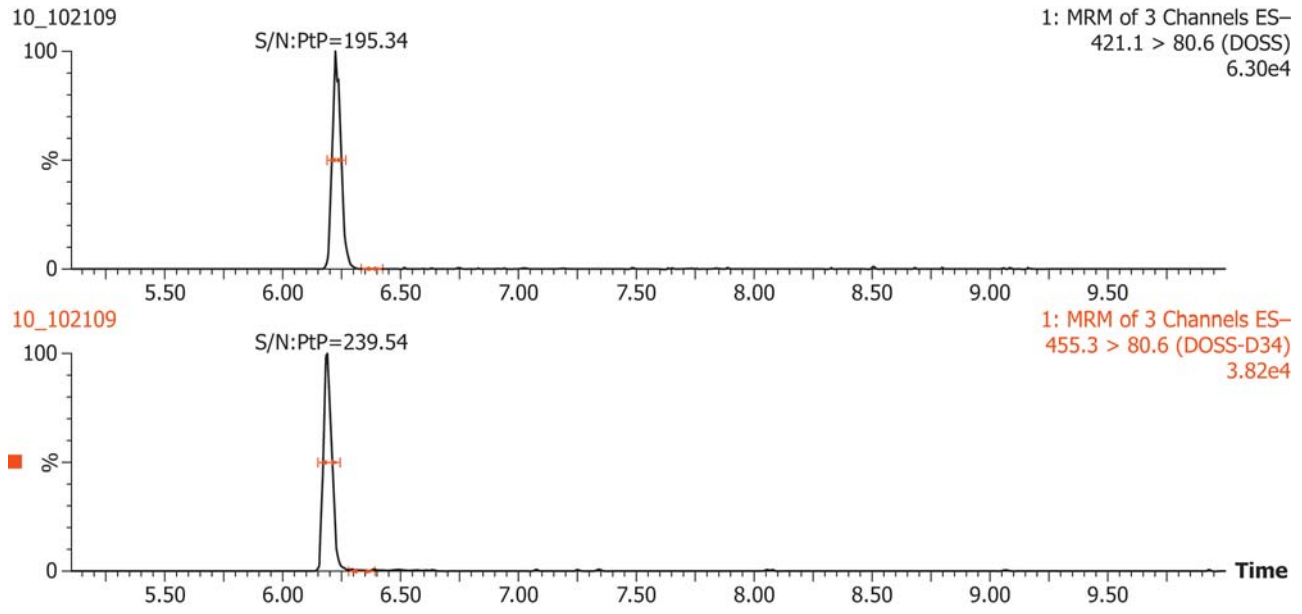

FIG. 2 Reporting Level Signal/Noise Ratio