



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 procedure 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 method. This 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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards*:²

[D1193](#) Specification for Reagent Water

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.

[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 *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).

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

3.2 *Abbreviations*:

3.2.1 *ppb*—parts per billion, $\mu\text{g/L}$

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

3.2.3 *NA*—no addition

3.2.4 *ND*—non-detect

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, 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.

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>

TABLE 1 Detection Verification Level and Reporting Range

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

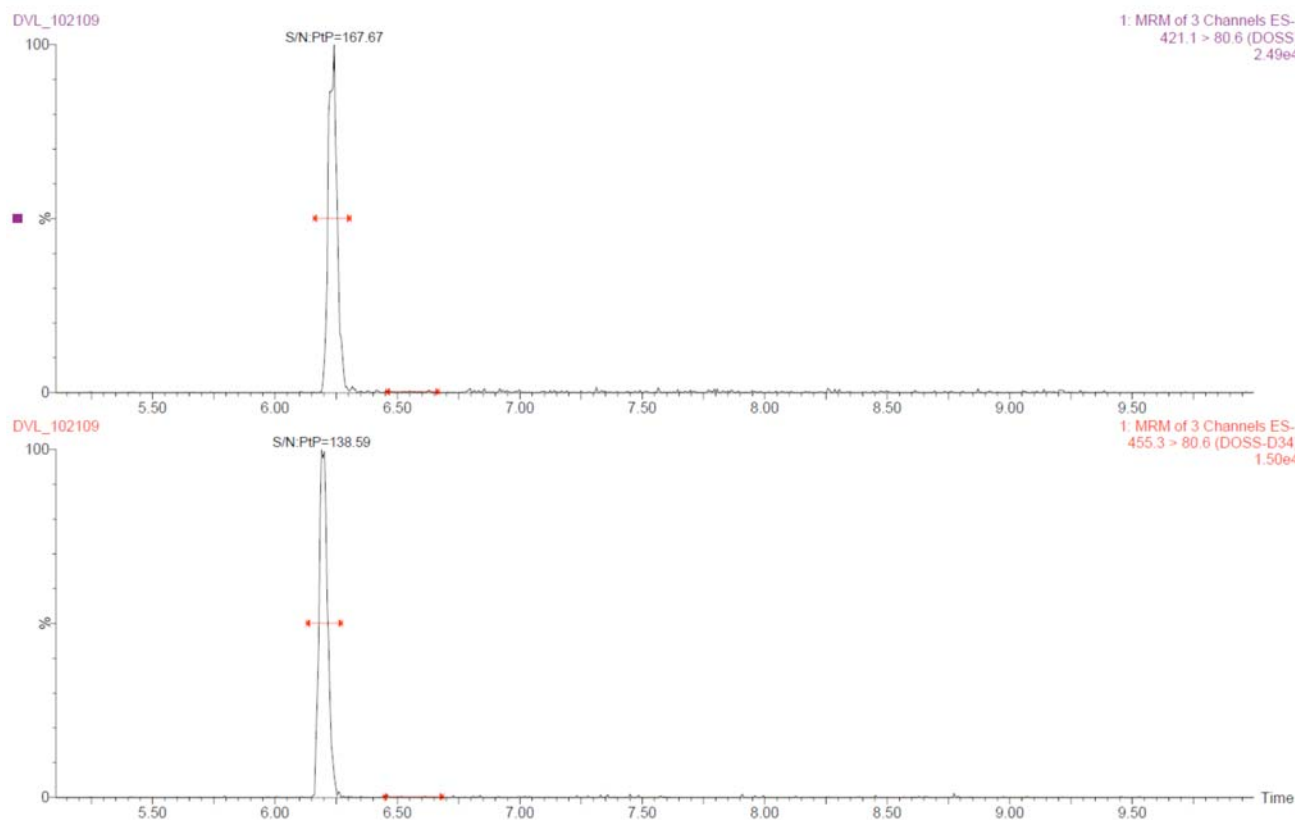


FIG. 1 Detection Verification Level Signal/Noise Ratio.

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 Food and Drug Administration 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 US EPA aquatic life benchmark for DOSS is 40 ppb.⁵

5.2 This 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.

⁴ Code of Federal Regulations-Title 21: Food and Drugs, Part 172 available at <http://www.gpoaccess.gov/cfr/index.html>.

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

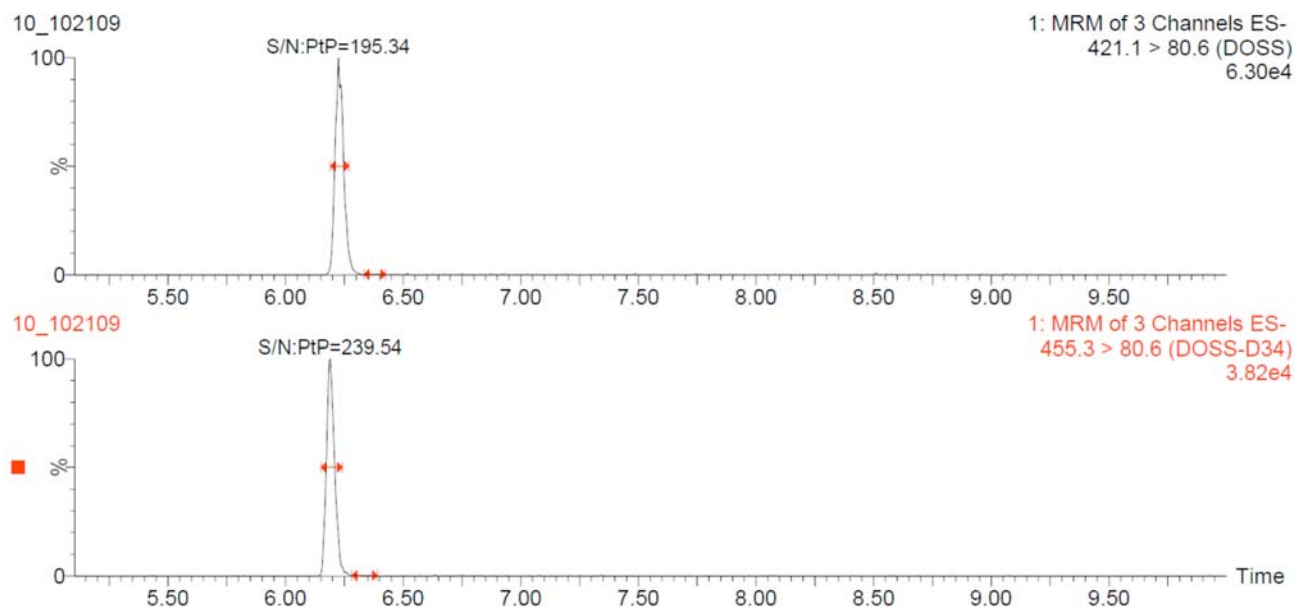


FIG. 2 Reporting Level Signal/Noise Ratio.

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, 2.1 x 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.⁷ 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 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 (Millipore Corporation, Catalog # SLGV033NS) 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 American Chemical Society.⁸ Other reagent grades may be used provided they are first determined to be of sufficiently high purity to permit their use without affecting the accuracy of the measurements.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type 1 of Specification D1193. It must be demonstrated that this water does not contain contaminants at concentrations sufficient to interfere with the analysis.

8.3 *Gases*—Ultrapure nitrogen and argon.

8.4 Acetonitrile (CH₃CN, CAS # 75-05-1).

8.5 Methanol (CAS # 67-56-1).

8.6 Ammonium formate (NH₄CO₂H, CAS # 540-69-2).

8.7 2-Propanol (CAS # 67-63-0).

⁶ A Waters ACQUITY UltraPerformance Liquid Chromatography (UPLC[®]) System 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 A Waters Quattro Premier[®] XE tandem quadrupole mass spectrometer 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.

⁸ Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, D.C. For Suggestions on the testing of reagents not listed by the American Chemical Society, see Annual Standards for Laboratory Chemicals, BDH Ltd., Poole, Dorset, U.K., and the United States Pharmacopeia and National Formulators, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.