



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](#)

[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, µg/L

3.2.2 *mM*—millimolar, 1 x 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.

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.

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), U.S. Department of Commerce, 5285 Port Royal Road, Springfield, VA, 22161 or at <http://www.epa.gov/epawaste/hazard/testmethods/index.htm>