



Designation: D7742 – 17 (Reapproved 2024)

Standard Practice for Determination of Nonylphenol Polyethoxylates (NPnEO, $3 \leq n \leq 18$) and Octylphenol Polyethoxylates (OPnEO, $2 \leq n \leq 12$) in Water by Single Reaction Monitoring (SRM) Liquid Chromatography/ Tandem Mass Spectrometry (LC/MS/MS)¹

This standard is issued under the fixed designation D7742; 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 practice covers the determination of nonylphenol polyethoxylates (NPnEO, $3 \leq n \leq 18$) and octylphenol polyethoxylates (OPnEO, $2 \leq n \leq 12$) in water by Single Reaction Monitoring (SRM) Liquid Chromatography/ Tandem Mass Spectrometry (LC/MS/MS) using direct injection liquid chromatography (LC) and detected with tandem mass spectrometry (MS/MS) detection. This is a screening practice with qualified quantitative data to check for the presence of longer chain ethoxylates in a water sample.

1.1.1 All data are qualified because neat standards of each alkylphenol ethoxylate (APEO) are not available and the synthesis and characterization of these neat standards would be very expensive. The Igepal² brand standards, which contain a mixture of various chain lengths of the alkylphenol ethoxylates (APEOs), were used. The mixture was characterized in-house assuming the instrument response at an optimum electrospray ionization cone and collision voltage for each APEO was the same. This assumption, which may not be accurate, is used to determine qualified amounts of each ethoxylate in the standards. The n-Nonylphenol diethoxylate (n-NP2EO) surrogate was available as a neat characterized standard, therefore, this concentration and recovery data was not estimated. APEOs are not regulated by the EPA, but nonylphenol, a breakdown product of NPnEOs, is regulated for fresh and saltwater dischargers. A request by a sewage treatment plant (STP) was made to make this practice available through ASTM in order to screen for the influent or effluent from sources of APEOs coming into the STP. The interest lies in stopping the source of the longer chain APEOs from entering the STP in order to meet effluent guidelines. Based upon the above, this is a practice rather than a test method. A comparison between samples is

possible using this practice to determine which has a higher concentration of APEOs.

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

1.3 The estimated screening range shown in [Table 1](#) was calculated from the concentration of the Level 1 and 7 calibration standards shown in [Table 4](#). These numbers are qualified, as explained in [Section 1](#), and must be reported as such. [Figs. 1-5](#) show the SRM chromatograms of each analyte at the Level 1 concentration with the signal to noise (S/N) ratio. This is a screening practice and method detection limits are not given. The S/N ratio for each analyte at the Level 1 concentration must be at least 5:1 for adequate sensitivity. If the instrument can not meet the criteria, the screening limit must be raised to an acceptable level.

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:³

[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](#)

¹ This practice 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 Igepal is a trademark of Rhodia Operations, Aubervilliers, CA.

³ 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 Estimated Screening Range

Analyte	Estimated Screening Range (µg/L)
Nonylphenol triethoxylate (NP3EO)	0.73–11.6
Nonylphenol tetraethoxylate (NP4EO)	1.1–18.3
Nonylphenol pentaethoxylate (NP5EO)	1.4–22.1
Nonylphenol hexaethoxylate (NP6EO)	1.8–28.2
Nonylphenol heptaethoxylate (NP7EO)	1.9–30.1
Nonylphenol octaethoxylate (NP8EO)	1.8–29.2
Nonylphenol nonaethoxylate (NP9EO)	1.6–26.3
Nonylphenol decaethoxylate (NP10EO)	1.5–24.1
Nonylphenol undecaethoxylate (NP11EO)	1.3–21.3
Nonylphenol dodecaethoxylate (NP12EO)	1.0–15.7
Nonylphenol tridecaethoxylate (NP13EO)	0.64–10.3
Nonylphenol tetradecaethoxylate (NP14EO)	0.41–6.5
Nonylphenol pentadecaethoxylate (NP15EO)	0.21–3.4
Nonylphenol hexadecaethoxylate (NP16EO)	0.11–1.7
Nonylphenol heptadecaethoxylate (NP17EO)	0.05–0.80
Nonylphenol octadecaethoxylate (NP18EO)	0.023–0.4
Total NPnEO	16–250
Octylphenol diethoxylate (OP2EO)	0.14–2.3
Octylphenol triethoxylate (OP3EO)	1.4–22.2
Octylphenol tetraethoxylate (OP4EO)	2.2–35.2
Octylphenol pentaethoxylate (OP5EO)	2.9–45.8
Octylphenol hexaethoxylate (OP6EO)	2.6–41.9
Octylphenol heptaethoxylate (OP7EO)	2.5–40.4
Octylphenol octaethoxylate (OP8EO)	1.8–28.8
Octylphenol nonaethoxylate (OP9EO)	1.1–17.6
Octylphenol decaethoxylate (OP10EO)	0.62–9.9
Octylphenol undecaethoxylate (OP11EO)	0.26–4.2
Octylphenol dodecaethoxylate (OP12EO)	0.11–1.8
Total OPnEO	16–250
n-Nonylphenol diethoxylate (n-NP2EO)	15.6–250 (Not Estimated)

D3694 Practices for Preparation of Sample Containers and for Preservation of Organic Constituents

D3856 Guide for Management Systems in Laboratories Engaged in Analysis of Water (Withdrawn 2024)⁴

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

2.2 Other Standard:⁵

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 *alkylphenol ethoxylates*, *n*—in this practice, nonylphenol polyethoxylates (NPnEO, $3 \leq n \leq 18$) and octylphenol polyethoxylates (OPnEO, $2 \leq n \leq 12$) collectively.

3.2.2 *screening limit*, *SL*, *n*—the estimated concentration of the lowest-level calibration standard used for quantification accounting for the sample dilution.

3.3 Abbreviations:

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

3.3.2 *ND*—non-detect

3.3.3 *ppt*—parts per trillion, ng/L

4. Summary of Practice

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

4.2 For APEOs analysis, samples are shipped to the lab between 0 °C and 6 °C containing 1 % formaldehyde and analyzed within 7 days of collection. In the lab, an aliquot of the sample is filtered, spiked with surrogate, and analyzed directly by LC/MS/MS.

4.2.1 Field samples from sewage systems propose a challenging analysis. Since this is a screening technique to determine if APEOs are present, a 10 mL to 25 mL aliquot of the sample is filtered through a PVDF syringe driven filter unit before spiking with surrogate. It was demonstrated that similar recoveries of the APEOs are achieved filtered and unfiltered using PVDF filters. Filtering using PTFE filters produced much lower recoveries. This practice does not account for the APEOs adhered to particulates or the sample bottle.

4.3 Nonylphenol polyethoxylates (NPnEO, $3 \leq n \leq 18$), octylphenol polyethoxylates (OPnEO, $2 \leq n \leq 12$), and n-nonylphenol diethoxylate (n-NP2EO, surrogate) are identified by retention time and one SRM transition. The target analytes and surrogates are quantitated using the SRM transition by external calibration. The final report issued for each sample lists their qualified concentration and the surrogate recovery.

5. Significance and Use

5.1 This practice has been developed in support of the U.S. EPA Office of Water, Office of Science and Technology by the Chicago Regional Laboratory (CRL).

⁴ The last approved version of this historical standard is referenced on www.astm.org.

⁵ Available from National Technical Information Service (NTIS), 5301 Shawnee Rd., Alexandria, VA 22312, <http://www.ntis.gov>.