



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

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1. Scope

1.1 This procedure 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 Igepal® 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 Standard 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 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
- D3856 Guide for Management Systems in Laboratories Engaged in Analysis of Water
- D3694 Practices for Preparation of Sample Containers and for Preservation of Organic 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 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 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.