



Standard Test Method for Nitrobenzene in Aniline¹

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1. Scope

1.1 This test method covers a polarographic procedure for the determination of nitrobenzene in aniline.

1.2 This test method has been found applicable to the determination of nitrobenzene in aniline containing concentrations of not more than 25 mg/kg nitrobenzene.

1.3 The following applies to all specified limits in this test method: for purposes of determining conformance with this test method, an observed value or a calculated value shall be rounded off “to the nearest unit” in the last right-hand digit used in expressing the specification limit, in accordance with the rounding-off method of Practice E 29.

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.* For specific hazard statements, see Section 10 and Note 3.

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

D 1549 Test Method for Zinc in Lubricating Oils and Additives (Polarographic Method)³

D 3436 Practice for Sampling and Handling Aniline⁴

D 4790 Terminology of Aromatic Hydrocarbons and Related Chemicals⁴

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications⁵

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals⁶

2.2 Other Document:

OSHA Regulations, 29 CFR, paragraphs 1910.1000 and 1910.1200⁷

3. Terminology

3.1 See Terminology D 4790 for definition of terms used in this test method.

4. Summary of Test Method

4.1 Polarography is an electrochemical method of analysis. A quantitative evaluation of an electro-reducible material such as nitrobenzene can be determined by measuring the characteristic current flow, as a function of an increasingly applied voltage, resulting from the solution undergoing electrolysis.

4.2 The current flowing through the specimen during the analysis is in the microampere range so several polarograms can be obtained on the same specimen solution without significant differences.

4.3 The nitrobenzene content is determined by mixing four volumes of aniline with one volume of concentrated hydrochloric acid. The diffusion current of nitrobenzene is then measured by polarographic technique. A standard calibration graph is developed with various concentrations of nitrobenzene in aniline versus their diffusion currents. A reading of the diffusion current level of the test specimen from the calibration graph determines its nitrobenzene content.

5. Significance and Use

5.1 This test method gives a quantitative means of detection of nitrobenzene in industrial grade aniline both for quality control and quality assurance of the product.

6. Interferences

6.1 Since the diffusion coefficient for solutions of organic molecules can possibly increase up to 2 % per degree rise in temperature, the polarographic cell temperature should be relatively the same when comparing specimen analyses with standard calibration analyses.

6.2 The sensitivity of an analysis is affected by the dimensions of the dropping mercury electrode (DME) capillary and also by the height of the mercury column above the electrode. It is advisable to use the same DME capillary with a constant head of mercury that will yield a uniform dropping rate during a series of analyses. Recalibrate when the DME is changed.

6.3 Sudden or severe vibrations of the DME system can cause interferences in the resulting polarogram.

6.4 Residual dissolved oxygen in the specimen solution will interfere with the analysis. This test method includes a nitrogen purge to remove dissolved oxygen.

¹ This test method is under the jurisdiction of ASTM Committee D-16 on Aromatic Hydrocarbons and Related Chemicals and is the direct responsibility of Subcommittee D16.0D on Organic Nitrogen Compounds.

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² Annual Book of ASTM Standards, Vol 11.01.

³ Discontinued 1984; see 1984 Annual Book of ASTM Standards, Part 23.

⁴ Annual Book of ASTM Standards, Vol 06.04.

⁵ Annual Book of ASTM Standards, Vol 14.02.

⁶ Annual Book of ASTM Standards, Vol 03.05.

⁷ Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.