



Standard Test Method for Total Trace Nitrogen and Its Derivatives in Liquid Aromatic Hydrocarbons by Oxidative Combustion and Electrochemical Detection¹

This standard is issued under the fixed designation D 6366; 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 test method covers the determination of the total trace nitrogen (organic and inorganic) naturally found in liquid aromatic hydrocarbons, its derivatives and related chemicals.

1.2 This test method is applicable for samples containing nitrogen from 0.05 to 100 mgN/kg. For higher concentrations refer to Test Method D 4629.

1.3 The detector response for the technique within the scope of this test method is linear with nitrogen concentration.

1.4 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.

1.5 *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 9 and Note 2, Note 3, Note 4, and Note 8.

2. Referenced Documents

2.1 ASTM Standards:

D 3437 Practice for Sampling and Handling Liquid Cyclic Products²

D 3852 Practice for Sampling and Handling Phenol and Cresylic Acid²

D 4629 Test Method for Trace Nitrogen in Liquid Petroleum Hydrocarbons by Syringe/Inlet Oxidative Combustion and Chemiluminescence Detection³

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications⁴

2.2 Other Document:

OSHA Regulations, 29 CFR, paragraphs 1910.1000 and 1910.1200⁵

3. Terminology

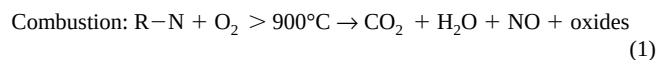
3.1 Definitions:

3.1.1 *oxidative combustion, n*—a process in which a sample undergoes combustion in an oxygen-rich environment at temperatures greater than 650°C and compounds decompose to form carbon dioxide, water and elemental oxides.

4. Summary of Test Method

4.1 A sample of liquid aromatic hydrocarbon is injected, at a controlled rate, into a stream of inert gas (helium or argon) or inert gas mixed with oxygen. The sample is vaporized and carried to a high temperature zone (> 900°C), where oxygen is introduced. Organic and inorganic nitrogen compounds, present in the specimen, are converted to nitric oxide (NO). Nitric oxide is reacted with the sensing electrode in a 3-electrode electrochemical cell. This reaction produces a measurable current that is directly proportional to the amount of nitrogen in the original sample material.

4.1.1 The reaction that occurs is as follows:



5. Significance and Use

5.1 Some process catalysts used in petroleum and chemical refining may be poisoned when even trace amounts of nitrogenous materials are contained in the feedstocks. This test method can be used to determine total nitrogen in process feeds and may also be used to control nitrogen compounds in finished products that fall within the scope of this test method.

NOTE 1—Virtually all organic and inorganic nitrogen compounds will be detected by this technique.

5.2 This technique will not detect diatomic nitrogen and it will produce an attenuated response when analyzing compounds (that is, 5-triazine and azo compounds, etc.) that form

¹ This test method is under the jurisdiction of ASTM Committee D16 on Aromatic Hydrocarbons and Related Materials, and is the direct responsibility of Subcommittee D16.04 on Instrumental Analysis.

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² Annual Book of ASTM Standards, Vol 06.04.

³ Annual Book of ASTM Standards, Vol 05.02.

⁴ Annual Book of ASTM Standards, Vol 14.02.

⁵ Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.