



Standard Test Method for Trace Nitrogen in Aromatic Hydrocarbons by Oxidative Combustion and Reduced Pressure Chemiluminescence Detection¹

This standard is issued under the fixed designation D 6069; 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 total nitrogen (organic and inorganic) in aromatic hydrocarbons, their derivatives and related chemicals.

1.2 This test method is applicable for samples containing nitrogen from 0.2 to 2 mgN/kg. For higher nitrogen concentrations refer to Test Method D 4629.

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

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 9.

2. Referenced Documents

2.1 ASTM Standards:

D 1555 Test Method for Calculation of Volume and Weight of Industrial Aromatic Hydrocarbons²

D 3437 Practice for Sampling and Handling Liquid Cyclic Products²

D 4629 Test Method for Organically Bound 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

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method⁴

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 D16.0E 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.

2.2 Other Documents:

OSHA Regulations, 29 CFR, paragraphs 1910.1000 and 1910.1200⁵

3. Terminology

3.1 Definitions:

3.1.1 *reduced pressure chemiluminescence*—a chemical reaction at pressure less than 760 mm mercury (Hg) in which light is emitted.

3.1.2 *oxidative pyrolysis*—a process in which a sample under goes combustion in an oxygen rich environment at temperatures greater than of 650°C.

3.1.2.1 *Discussion*—Organic compounds pyrolytically decompose to carbon dioxide, water and elemental oxides.

4. Summary of Test Method

4.1 A specimen is introduced into a gas stream, at a controlled rate, and carried into a high temperature furnace (>900°C) where an excess of oxygen is added. Pyrolysis converts organic material in the specimen to carbon dioxide and water. Organic nitrogen and inorganic nitrogen compounds, present in the specimen, are converted to nitric oxide (NO). Nitric oxide reacts with ozone in the detector producing nitrogen dioxide molecules in an excited state. As the excited nitrogen dioxide molecules relax to ground state, light is emitted. This light is detected by a photomultiplier tube with the resulting signal proportional to the concentration of nitrogen in the sample. Operating the detector at a reduced pressure, lowers the probability of the excited nitrogen dioxide molecules colliding with other molecules before it under goes chemiluminescence. Thus, reduced pressure provides improved sensitivity and lower noise.

5. Significance and Use

5.1 This method has been prepared to detect and quantitate nitrogen-containing compounds such as *N*-formylmorpholine (4-formylmorpholine, Chemical Abstract Service numbers (CAS) No. 250-37-6) or 1-methyl-2-pyrrolidinone (NMP) (CAS) No. 872-50-42 at a concentration of 1.0 mgN/kg or less in aromatic hydrocarbons used or produced in manufacturing

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