



Standard Test Method for Oxides of Nitrogen in Gaseous Combustion Products (Phenol-Disulfonic Acid Procedures)¹

This standard is issued under the fixed designation D 1608; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method describes the phenol-disulfonic acid colorimetric procedure (1)² for the determination of total oxides of nitrogen [nitrous oxide (N₂O) excepted] in gaseous effluents from combustion and other nitrogen oxidation processes.

1.2 It is applicable to a concentration range of oxides of nitrogen as nitrogen dioxide (NO₂) of 5 ppm to several thousand parts per million by volume (four to several thousand milligrams per dry standard cubic metre).

1.3 Since the grab sampling technique used takes a relatively small sample over a very short period of time, the result obtained will be an instantaneous measure of the nitrogen oxides and, therefore, will be representative of the emissions only if the gas stream is well mixed and the concentration constant with time. Multiple samples are recommended.

1.4 The values stated in SI units are to be regarded as standard. The SI equivalents are in parentheses and may be approximate.

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 more specific safety precautionary information see 8.5 and Section 3.)*

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

D 1356 Terminology Relating to Sampling and Analysis of Atmospheres³

D 1357 Practice for Planning the Sampling of the Ambient Atmosphere³

¹ This test method is under the jurisdiction of ASTM Committee D22 on Sampling and Analysis of Atmospheres and is the direct responsibility of Subcommittee D22.03 on Ambient Atmospheres and Source Emissions.

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² Annual Book of ASTM Standards, Vol 11.01.

³ Annual Book of ASTM Standards, Vol 11.03.

D 1605 Practices for Sampling Atmospheres for Analysis of Gases and Vapors⁴

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D 1356.

4. Summary of Test Method

4.1 The gas sample is admitted into an evacuated flask containing an oxidizing absorbing solution consisting of hydrogen peroxide in dilute sulfuric acid. The oxides of nitrogen are converted to nitric acid by gas phase oxidation due to oxygen in the sample and by the absorbent solution. The resulting nitrate ion is reacted with phenol disulfonic acid to produce a yellow compound (the tri-ammonium salt of 6-nitro-1-phenol-2,4-disulfonic acid), which is measured colorimetrically. Calibration curves, prepared from samples of known nitrate content, are used to determine the amount of nitrate in the sample with results expressed as nitrogen dioxide.

5. Significance and Use

5.1 This test method provides a means to measure the total nitrogen oxides (NO_x) content of gaseous emissions for purposes such as determining compliance with regulations, studying the effect of various abatement procedures on NO_x emissions, and checking the validity of instrumental measurements.

6. Interferences (1, 2)

6.1 Inorganic nitrates, nitrites, or organic nitrogen compounds that are easily oxidized to nitrates interfere with the test method and give erroneously high results. The presence of certain reducing agents, for example, sulfur dioxide (SO₂), may interfere by consuming part of the hydrogen peroxide in the absorbing solution to leave an inadequate amount for reaction with the oxides of nitrogen. Halides lower the results, but interference from halide ion (and lead) are negligible in the concentration usually encountered in combustion sources.

⁴ Discontinued—see 1991 Annual Book of ASTM Standards, Vol 11.03.

