



Standard Test Method for Measurement of Total Hydrogen Sulfide in Residual Fuels by Multiple Headspace Extraction and Sulfur Specific Detection¹

This standard is issued under the fixed designation D 6021; 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.

^{ε1} NOTE—Warning notes were placed in the text editorially in May 2001.

1. Scope

1.1 This test method covers a method suitable for measuring the total amount of hydrogen sulfide (H₂S) in heavy distillates, heavy distillate/residual fuel blends, or residual fuels as defined in Specification D 396 Grade 4, 5 (Light), 5 (Heavy), and 6, when the H₂S concentration in the fuel is in the 0.01 to 100 µg/g (ppmw) range.

1.2 The values stated in SI units are to be regarded as the standard.

1.3 *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 5.1, 7.5, 8.2, 9.2, 10.1.4, and 11.1.

2. Referenced Documents

2.1 ASTM Standards:

D 396 Specification for Fuel Oils²

D 1193 Specification for Reagent Water³

D 2420 Test Method for Hydrogen Sulfide in Liquefied Petroleum (LP) Gases (Lead Acetate Method)²

D 3609 Practice for Calibration Techniques Using Permeation Tubes⁴

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products⁵

D 4084 Test Method for Analysis of Hydrogen Sulfide in Gaseous Fuels (Lead Acetate Reaction Rate Method)⁶

D 4323 Test Method for Hydrogen Sulfide in the Atmosphere by Rate of Change of Reflectance⁴

D 5504 Test Method for the Determination of Sulfur Compounds in Natural Gas and Gaseous Fuels by Gas Chromatography and Chemiluminescence Detection⁶

D 5705 Test Method for Measurement of Hydrogen Sulfide in the Vapor Phase Above Residual Fuel Oils⁷

3. Terminology

3.1 Definitions:

3.1.1 *heavy distillate, n*—a fuel produced from the distillation of crude oil which has a kinematic viscosity at 40°C between 5.5 and 24.0 mm²/s, inclusive.

3.1.2 *heavy distillate/residual fuel blend, n*—a blend of heavy distillate and residual fuel oil having a viscosity at 40°C between 5.5 and 24.0 mm²/s, inclusive.

3.1.3 *multiple headspace extraction, n*—a technique to determine the total concentration of a gas trapped in a liquid by analysis of successive gas extractions from the vapor space of a closed vessel containing a known amount of the sample.

3.1.4 *residual fuel oil, n*—any liquid or liquefiable petroleum product having a kinematic viscosity at 100°C between 5.0 and 50.0 mm²/s, inclusive, burned for the generation of heat in a furnace or firebox or for the generation of power in an engine.

4. Summary of Test Method

4.1 A representative sample of residual fuel oil is obtained in sufficient quantity to completely fill the sample container. The sample is taken to the laboratory preferably within one to 4 h, within 24 h maximum and placed in a refrigerator until the hydrogen sulfide analysis can be run. At that time, the sample is removed from the refrigerator and allowed to sit at ambient temperature until it flows freely.

4.2 A 0.05 to 5.0 g test specimen (aliquot) is placed in a headspace vial and heated in an oven at 60°C for more than five but less than 15 min. The headspace gas is sampled and injected into either of two types of apparatus capable of

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.E0.01 on Burner Fuels.

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² Annual Book of ASTM Standards, Vol 05.01.

³ Annual Book of ASTM Standards, Vol 11.01.

⁴ Annual Book of ASTM Standards, Vol 11.03.

⁵ Annual Book of ASTM Standards, Vol 05.02.

⁶ Annual Book of ASTM Standards, Vol 05.06.

⁷ Annual Book of ASTM Standards, Vol 05.03.