



Designation: D5705 – 13

Standard Test Method for Measurement of Hydrogen Sulfide in the Vapor Phase Above Residual Fuel Oils¹

This standard is issued under the fixed designation D5705; 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 field determination of hydrogen sulfide (H_2S) in the vapor phase (equilibrium headspace) of a residual fuel oil sample.

1.2 The test method is applicable to liquids with a viscosity range of $5.5 \text{ mm}^2/\text{s}$ at 40°C to $50 \text{ mm}^2/\text{s}$ at 100°C . The test method is applicable to fuels conforming to Specification D396 Grade Nos. 4, 5 (Heavy), and 6.

1.3 The applicable range is from 5 to 4000 parts per million by volume (ppm v/v) (micro mole/mole).

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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.*

2. Referenced Documents

- 2.1 *ASTM Standards:*²
 - D396 Specification for Fuel Oils
 - D4057 Practice for Manual Sampling of Petroleum and Petroleum Products
 - D6021 Test Method for Measurement of Total Hydrogen Sulfide in Residual Fuels by Multiple Headspace Extraction and Sulfur Specific Detection
 - D7621 Test Method for Determination of Hydrogen Sulfide in Fuel Oils by Rapid Liquid Phase Extraction

3. Terminology

3.1 Definitions:

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.14 on Stability and Cleanliness of Liquid Fuels.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

3.1.1 *equilibrium headspace, n*—the vapor space above the liquid in which all vapor components are in equilibrium with the liquid components.

3.1.2 *residual fuel oil, n*—a fuel oil comprising a blend of viscous long, short, or cracked residue from a petroleum refining process and lighter distillates blended to a fuel oil viscosity specification.

3.1.2.1 *Discussion*—Under the conditions of this test (1:1 liquid/vapor ratio, temperature, and agitation) the H_2S in the vapor phase (sample's headspace) will be in equilibrium with the H_2S in the liquid phase.

4. Summary of Test Method

4.1 A 1-L H_2S -inert test container (glass test bottle) is filled to 50 volume % with fuel oil from a filled H_2S -inert container (glass sample bottle) just prior to testing. In the test container, the vapor space above the fuel oil sample is purged with nitrogen to displace air. The test container with sample is heated in an oven to 60°C , and agitated on an orbital shaker at 220 rpm for 3 min.

4.2 A length-of-stain detector tube and hand-operated pump are used to measure the H_2S concentration in the vapor phase of the test container. The length-of-stain detector tube should be close to but not in contact with the liquid surface.

5. Significance and Use

5.1 Residual fuel oils can contain H_2S in the liquid phase and this can result in hazardous vapor phase levels of H_2S in storage tank headspaces. The vapor phase levels can vary significantly according to the headspace volume, fuel temperature and agitation. Measurement of H_2S levels in the liquid phase provides a useful indication of the residual fuel oil's propensity to form high vapor phase levels, and lower levels in the residual fuel oil will directly reduce risk of H_2S exposure. It is critical, however, that anyone involved in handling fuel oil, such as vessel owners and operators, continue to maintain appropriate safety practices designed to protect the crew, tank farm operators and others who can be exposed to H_2S .

5.1.1 The measurement of H_2S in the liquid phase is appropriate for product quality control, while the measurement of H_2S in the vapor phase is appropriate for health and safety purposes.

*A Summary of Changes section appears at the end of this standard

5.2 This test method was developed to provide refineries, fuel terminals and independent testing laboratories, which do not have access to analytical instruments such as a gas chromatograph, with a simple and consistent field test method for the rapid determination of H₂S in the vapor phase above residual fuel oils.

NOTE 1—D5705 is one of three test methods for quantitatively measuring H₂S in residual fuels:

- 1) Test Method D6021 is an analytical test method to determine H₂S levels in the liquid phase.
- 2) Test Method D7621 is a rapid test method to determine H₂S levels in the liquid phase.

NOTE 2—Because of the reactivity, absorptivity and volatility of H₂S, any measurement method only provides an H₂S concentration at a given moment in time.

5.3 This test method does not necessarily simulate the vapor phase H₂S concentration in a fuel storage tank. It does, however, provide a level of consistency so that the test result is only a function of the residual fuel oil sample and not the test method, operator, or location. No general correlation can be established between this field test and actual vapor phase concentrations of H₂S in residual fuel oil storage or transports. However, a facility that produces fuel oil from the same crude source under essentially constant conditions might be able to develop a correlation for its individual case.

6. Interferences

6.1 Typically, sulfur dioxide and mercaptans may cause positive interferences. In some cases, nitrogen dioxide can cause a negative interference. Most detector tubes will have a *precleanse* layer designed to remove certain interferences up to some maximum interferant level. Consult the manufacturer's instructions for specific interference information.

7. Apparatus

7.1 *Shaker*, a bench-top orbital shaker and platform equipped with a four-prong clamp to hold 1-L Boston round-bottom glass bottles and capable of operation at 220 rpm.

NOTE 3—An orbital shaker with an orbit of 19 mm has been found to be suitable.

7.2 *Timer*, capable of measuring from 1 s to 30 min at second intervals.

7.3 *Stopper with Temperature Measuring Device*, a No. 2 cork stopper with a temperature measuring devices inserted through it that is capable of accurately measuring the temperature of the sample at $60 \pm 1^\circ\text{C}$ as required in the procedure and extending at least 25 mm into the residual fuel but no closer than 25 mm from the bottom of a test bottle (see Fig. 1(a)). A dial thermometer having a range of -18 to 82°C and a 200-mm stem has been found suitable to use.

7.4 *Oven or Water Bath*, capable of heating the fuel oil samples to $60 \pm 1^\circ\text{C}$.

7.5 *Detector Tube Pump*, a hand-operated piston or bellows-type (Fig. 1(b)) pump with a capacity of $100 \text{ cm}^3 \pm 5 \text{ cm}^3$ per

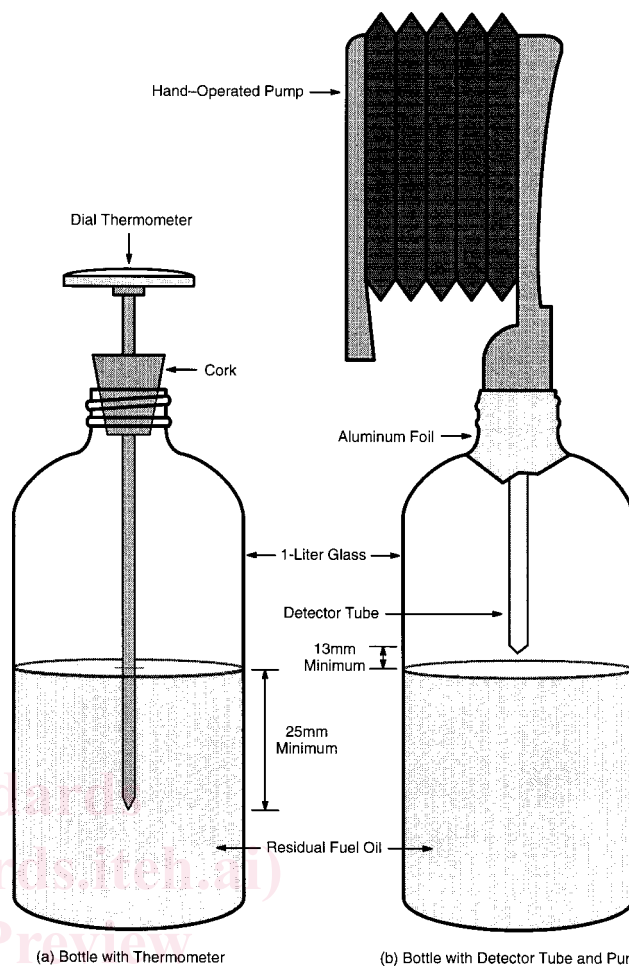


FIG. 1 Measurement of H₂S in the Vapor Phase of Residual Fuel Oil

stroke.³ It must be specifically designed for use with detector tubes. (**Warning**—A detector tube and pump together form a unit and must be used as such. Each manufacturer calibrates detector tubes to match the flow characteristics of its specific pump. Crossing brands of pumps and tubes is not permitted, as considerable loss of system accuracy is likely to occur.)

8. Reagents and Materials

8.1 *Containers*—Both sample and test containers are composed of H₂S-inert material such as 1-L size (clear Boston round-bottom) glass bottles with screw caps. The bottles are clean and dry. Mark test containers at the 50 % volume level by using a ruler. (**Warning**—Hydrogen sulfide reacts with metal surfaces and is easily oxidized, which depletes its concentration and gives false low test results. Containers such as epoxy-lacquered cans are suitable for sample collection. Alternative containers must give equivalent results to those obtained by using glass.)

³ Direct Reading Colorimetric Indicator Tubes Manual, First Edition, American Industrial Hygiene Association, Akron, OH 44311, 1976.