



# Standard Test Method for Measurement of Hydrogen Sulfide in the Vapor Phase Above Residual Fuel Oils<sup>1</sup>

This standard is issued under the fixed designation D 5705; 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 ( $\epsilon$ ) 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  $40^\circ\text{C}$  to  $50 \text{ mm}^2/\text{s}$  at  $100^\circ\text{C}$ . The test method is applicable to fuels conforming to Specification D 396 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 the standard. The values given in parentheses are for information only.

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:*<sup>2</sup>

D 396 Specification for Fuel Oils

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products

## 3. Terminology

3.1 *Definitions:*

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  $60^\circ\text{C}$ , 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 Excessive levels of hydrogen sulfide in the vapor phase above residual fuel oils in storage tanks may result in a health hazard, OSHA limits violation, and public complaints about odors. Control measures to maintain safe levels of  $H_2S$  in the tank atmosphere for those working in the vicinity require a consistent method for the assessment of potentially hazardous levels of  $H_2S$  in fuel oils (**Warning**— $H_2S$  is a highly toxic substance. Use extreme care in the sampling and handling of samples that are suspected of containing high levels of  $H_2S$ ).

5.2 This test method has been 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  $H_2S$  in the vapor phase of residual fuel oils.

5.3 This test method does not necessarily simulate the vapor phase  $H_2S$  concentration of 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

<sup>1</sup> 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 on Burner, Diesel, Non-Aviation Gas Turbine, and Marine Fuels.

Current edition approved Nov. 1, 2003. Published December 2003. Originally approved in 1995. Last previous edition approved in 2000 as D 5705–95(2000)<sup>\epsilon</sup>1.

<sup>2</sup> 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.

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

concentrations of H<sub>2</sub>S 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.<sup>3</sup>

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  $82^\circ\text{C}$  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 stroke.<sup>4</sup> 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 H<sub>2</sub>S-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.)

8.2 *Length-of-Stain Detector Tube and Calibration Scale*, a sealed glass tube with breakoff tips sized to fit the tube holder of the detector tube pump. The reagent layer inside the tube, typically a silica gel substrate coated with the active chemicals,

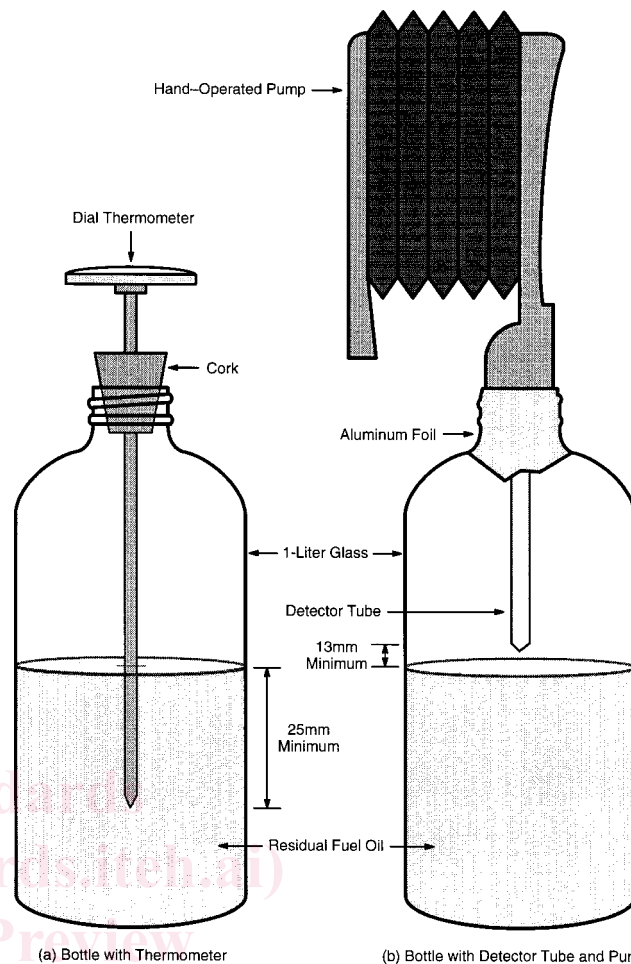


FIG. 1 Measurement of H<sub>2</sub>S in the Vapor Phase of Residual Fuel Oil

must be specific for hydrogen sulfide and must produce a distinct color change when exposed to a sample of gas containing hydrogen sulfide. Any substances known to interfere must be listed in the instructions accompanying the tubes. A calibration scale should be marked directly on the tube, or other markings that provide for easy interpretation (reading) of hydrogen sulfide content from a separate calibration scale should be supplied with the tubes. The calibration scale shall correlate H<sub>2</sub>S concentration to the length of the color stain. Annex A1 provides additional information. Shelf life of the detector tubes must be a minimum of two years from the date of manufacture, when stored according to the manufacturer's recommendations.

## 9. Sampling

9.1 The sampling of residual fuel oils is done according to Practice D 4057 for the sampling of storage tanks, ships, or barges. Composite sampling or running samples can be taken; spot samples should be taken from the midpoint or below midpoint of the fuel oil in a storage tank by first sufficiently purging sample transfer lines and then taking single samples where each sample comprises one and only one test.

<sup>3</sup> Baxter Scientific Product Models 3518 and 30100, or equivalent, are suitable for this test.

<sup>4</sup> Direct Reading Colorimetric Indicator Tubes Manual, First Edition, American Industrial Hygiene Association, Akron, OH 44311, 1976.