



## Standard Test Method for Hydrogen Sulfide and Mercaptan Sulfur in Natural Gas (Cadmium Sulfate Iodometric Titration Method)<sup>1</sup>

This standard is issued under the fixed designation D 2385; 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 determination of hydrogen sulfide and mercaptan sulfur in natural gas. It is applicable over the concentration range from 0 to 5 grains of hydrogen sulfide (about 11 mg/m<sup>3</sup>) and 0 to 1 grain of mercaptan sulfur/100 ft<sup>3</sup> (2.832 m<sup>3</sup>) of natural gas.

1.2 *This standard does not purport to address all of the safety problems, 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 Document

- 2.1 *ASTM Standard:*  
D 1193 Specification for Reagent Water<sup>2</sup>

### 3. Summary of Test Method

3.1 A measured volume of natural gas is bubbled through neutral cadmium sulfate solution to remove hydrogen sulfide, and then through basic cadmium sulfate to scrub out the mercaptans. The amounts of hydrogen sulfide and mercaptan sulfur in the absorbers are then determined iodometrically.

### 4. Significance and Use

4.1 Natural gas and its products of combustion must not be unduly corrosive to the materials with which they come in contact and the measure of hydrogen sulfide and other sulfur compounds is important. In addition, in some cases the odor of the gas must not be objectionable and from this standpoint the measurement of mercaptan sulfur is significant.

### 5. Apparatus

5.1 *Gas Washing Bottle*—A bottle with side inlet, coarse-porosity fritted disk (40 to 60  $\mu$ m maximum pore size), and 24/40 outer joint at the outlet. The diameter of the fritted disk is 60 mm, and the approximate outside diameter and height are 70 and 280 mm, respectively.<sup>3</sup>

5.2 *Spray Traps*, with 20/40 inner joint, for use on the

outlet side of the gas washing bottle.<sup>4</sup>

5.3 *Test Meter*, with dial divisions graduated to 0.01 ft<sup>3</sup> (28 cm<sup>3</sup>) and accurate to 0.5 percent when measuring gas volumes from 2 to 10 ft<sup>3</sup> (0.06 to 0.28 m<sup>3</sup>). The meter should be calibrated before use and should be a type that is usable in the field.

5.4 *Thermometer*, graduated in 1°F (0.5°C) divisions.

5.5 *Aspirator Bulb*, with a short piece of gum rubber tubing attached to the pressure end.

5.6 *Barometer*.

5.7 *Stop Watch*.

5.8 *Tubing and Connections*—Glass, aluminum, or stainless steel is satisfactory. Connections between tubing and apparatus should be made with gum rubber tubing, with a minimum amount of rubber exposed to the gas sample.

### 6. Reagents and Materials

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.<sup>5</sup> Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193.

6.3 *Cadmium Sulfate Solution A*—Dissolve 10 g of cadmium sulfate hydrate (3CdSO<sub>4</sub>·8H<sub>2</sub>O) (**Danger—Poison**). May be fatal if swallowed or inhaled) in water and dilute to 1 L.

6.4 *Cadmium Sulfate Solution B*—Dissolve 140 g of 3CdSO<sub>4</sub>·8H<sub>2</sub>O (**Danger—Poison**). May be fatal if swallowed or inhaled. See Annex A1.1) in water and dilute to 1 L.

6.5 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).

6.6 *Iodine Standard Solution (0.1 N)*—Weigh 13.0 g of resublimed iodine into a 250-mL beaker. Add 22 g of potassium iodide (KI) and 100 mL of water. Stir until solution is complete, dilute to 1 L, mix thoroughly, and store in an amber, glass-stoppered bottle. Standardize this solution

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D-3 on Gaseous Fuels and is the direct responsibility of Subcommittee D03.05 on Determination of Special Constituents of Gaseous Fuels.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>3</sup> The bottle available under Corning catalog No. 31750 has been found satisfactory for this purpose.

<sup>4</sup> The spray trap available under Corning Catalog No. 37723 (specify spray trap only) has been found satisfactory for this purpose.

<sup>5</sup> Reagent Chemicals, American Chemical Society Specifications, Am. Chemical Soc., Washington, D.C. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

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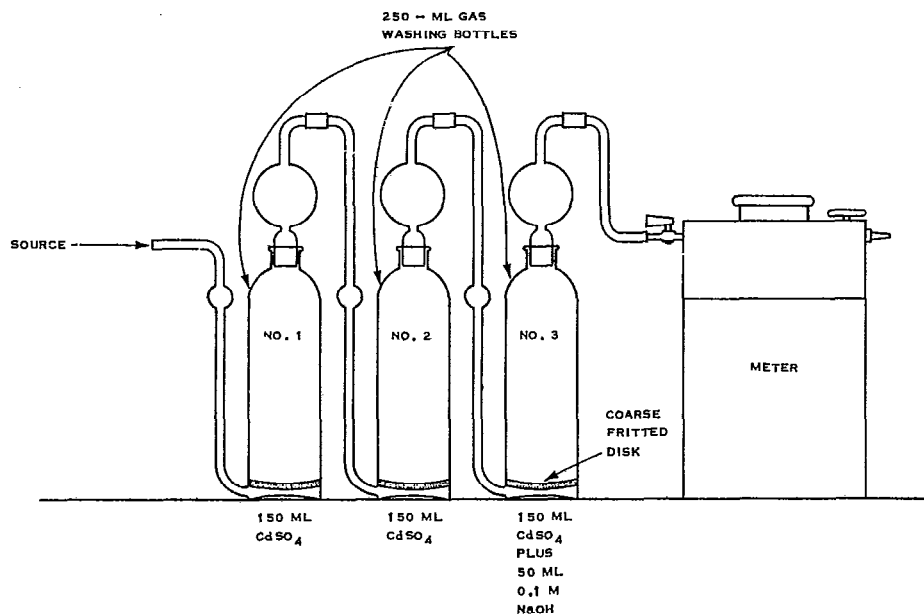


FIG. 1 Diagram of Apparatus Showing Absorption Train

against standard 0.1  $N$   $Na_2S_2O_3$  solution not more than one day from the time it is to be used.

6.7 *Iodine, Standard Solutions* (0.05 and 0.01  $N$ )—Prepare 0.05 and 0.01  $N$  iodine solutions by exact dilution of the 0.1  $N$  iodine solution.

6.8 *Sodium Hydroxide Solution* (4 g/L)—Dissolve 4 g of sodium hydroxide (NaOH) in water and dilute to 1 L.

6.9 *Sodium Thiosulfate, Standard Solution* (0.1  $N$ )—Dissolve 25 g of sodium thiosulfate ( $Na_2S_2O_3 \cdot 5H_2O$ ) in water and add 0.01 g of sodium carbonate ( $Na_2CO_3$ ) to stabilize the solution. Dilute to 1 L and mix thoroughly. Standardize by any accepted procedure by which the normality may be determined with an accuracy of  $\pm 0.0002$ .

6.10 *Sodium Thiosulfate, Standard Solutions* (0.05 and 0.01  $N$ )—Prepare 0.05 and 0.01  $N$   $Na_2S_2O_3$  solutions by exact dilution of the 0.1  $N$   $Na_2S_2O_3$  solution.

6.11 *Starch Solution*—A freshly prepared solution of any soluble starch suitable as an iodine indicator may be used. One suitable method of preparation is as follows: make a suspension of 2.5 g of arrowroot starch in a small amount of water. Add the suspension to 1 L of boiling water and boil

for 5 to 10 min, allow to cool, and decant the clear portion into glass-stoppered bottles.

## 7. Sampling

7.1 The gas to be analyzed *must* be sampled at the source. It is not permissible to transport the gas to the laboratory in a cylinder of any type. The sampling point should be located directly on the main flow line and the connections preferably should be a center line tap. Thorough purging of the tap line before sampling is necessary to remove any "dead" gas having a low  $H_2S$  content due to the reaction with the iron tap line. The connection from the sampling point to the absorption bottle should be as short as possible.

## 8. Procedure

8.1 Assemble the absorption train as shown in Fig. 1. Add 150 mL of  $CdSO_4$  Solution A or B (see Table 1) (**Danger—Poison.** May be fatal if swallowed or inhaled. See Annex A1.1) to each of the three absorbers. Replace the spray traps on the two hydrogen sulfide ( $H_2S$ ) absorbers. Add 50 mL of NaOH solution to the third absorber and replace the spray trap. Record the initial test meter reading. Start the flow of gas through the system by opening the control valve on the sampling line. Set the rate not to exceed  $6 \text{ ft}^3$  ( $0.169 \text{ m}^3$ )/h. Record the temperature of the test gas several times during the sampling period. The amount of sample required and the concentration of  $CdSO_4$  used depends upon the  $H_2S$  content of the gas. A close approximation of the  $H_2S$  content for use of Table 1 may be obtained quickly by means of commercial detectors.

8.2 After sufficient sample has been passed, stop the flow of gas through the system by shutting the control valve. Disconnect the apparatus. Rinse out the spray traps with water and add the rinse water to the absorber. Place a rubber policeman over inlet and outlet tubes of the absorber bottles. Record the final meter reading, the barometric pressure, and the average temperature of the gas that has passed through

TABLE 1 Sample and Cadmium Sulfate Concentration Requirements

Expected $H_2S$ Concentration, grains/100 $ft^3$ (2.832 $m^3$ ) (standard)	Expected Mercaptan Sulfur Concentration, grains/100 $ft^3$ (2.832 $m^3$ ) (standard)	Gas Volume, $ft^3$ (2.832 $m^3$ ) (standard)	Concentration of Cadmium Sulfate, g/L
0 to 0.25	0 to 0.25	14	10
0.25 to 0.50	0.25 to 0.50	10	10
0.50 to 0.75	0.50 to 0.75	8	10
0.75 to 1.0	0.75 to 1.0	6	10
1 to 2	...	10	140
2 to 3	...	10	140
3 to 4	...	8	140
4 to 5	...	6	140