



Designation: D 3031 - 81 (Reapproved 1986)

Standard Test Method for Total Sulfur in Natural Gas by Hydrogenation¹

This standard is issued under the fixed designation D 3031; 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 determination of sulfur in natural gas containing not more than 1.0 grain of sulfur/100 ft³ (23 mg/m³) and not more than 0.5 % of C₅ and heavier hydrocarbons.

1.2 The values stated in inch-pound units are to be regarded as the standard.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems 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 2725 Test Method for Hydrogen Sulfide in Natural Gas (Methylene Blue Method)²

3. Summary of Method

3.1 A measured sample of gas is mixed with humidified hydrogen and passed over platinum gauze at 900°C in a tube furnace. Organic sulfur in the gas is converted to hydrogen sulfide, which is absorbed in a zinc acetate solution and determined by the methylene blue procedure (Test Method D 2725).

4. Apparatus

4.1 In addition to the apparatus specified in Test Method D 2725, the following items are required:

4.1.1 *Control Valves*—Valves 1, 2, and 5 of Fig. 2 shall be needle valves or other devices suitable for regulating the flow of hydrogen and sample gas. Valves 3 and 4 shall be either quick opening and closing valves or a 3-way metal or glass cock, for rapidly diverting the sample stream. Valves 6 and 7 shall be 3-way metal or glass cocks, for diverting the gas through the liquid sample injector according to 6.5.

4.1.2 *Flowmeters*—Variable area or capillary types, for measuring the rates of flow of sample gas at 0.2 ft³ (6 L)/h and of hydrogen at 0.8 ft³ (25 L)/h. The sensitivity of the sample flowmeter shall be such that a 1 % change in flow can be observed. The sensitivity of the hydrogen flowmeter shall be such that a 2 % change in flow can be observed.

4.1.3 *Pyrometer*—A pyrometer of suitable design, capable of indicating the temperature at 900°C within $\pm 10^\circ\text{C}$.

4.1.4 *Refractory Tube*, transparent fused silica tube $\frac{3}{4}$ -in. (20-mm) inside diameter, and 24 to 30 in. (60 to 75 cm) long, with reduced end for tubing connection.

4.1.5 *Timer*—A stopwatch or other device to register periods of 15 to 30 min with an accuracy 0.02 min or better.

4.1.6 *Tube Furnace*—The furnace shall be 9 to 12 in. (23 to 300 mm) long and capable of maintaining a temperature of $900 \pm 10^\circ\text{C}$ in its middle 3-in. (80 mm) section.

4.1.7 *Liquid Sample Injector*—A glass apparatus similar to that of Fig. 1 containing a rubber septum for introduction of a 1-mL liquid sample directly into the gas stream, and a liquid trap section which permits only vapor to leave the apparatus. Provision should be made to warm the trap area to speed evaporation of liquid.

4.1.8 *Syringe*—A hypodermic syringe for introduction of 1.0-mL liquid sample with 2 % accuracy through a rubber septum.

5. Reagents and Materials

5.1 The reagents specified in Test Method D 2725 are required, as well as the following:

5.1.1 *Hydrogen*—99.5 % purity or better. It shall meet the requirements of 7.3 and 7.4. A high-pressure cylinder shall be fitted with a pressure regulator to reduce the delivery pressure to 1 to 5 psi (6.9 to 34.5 kPa) gage.

5.1.2 *Methyl Alcohol*—Reagent grade.

5.1.3 *Platinum Catalyst*—A 3 by 12 in. piece of platinum-10 % rhodium gauze, 80 mesh, 0.003 in. diameter wire.

NOTE 1—Fused silica wool may be used at 1000°C in place of the platinum catalyst.

5.1.4 *Sulfur Absorbers*—Two activated charcoal packed absorption tubes, each approximately $\frac{3}{4}$ -in. (19 mm) diameter by 10 in. (250 mm) long with tubing connections on each end.

NOTE 2—One of these items not required if hydrogen purity meets absorbance specifications of 7.4.

5.1.5 *Sulfur Compound*—Any mercaptan, organic sulfide, or organic disulfide is satisfactory if the compound contains 3 to 5 carbon atoms per molecule. The purity must be such that the weight of sulfur contained in the liquid compound must be known to within 1 %.

6. Calibration

6.1 Calibrate the spectrophotometer or colorimeter as specified in Test Method D 2725.

7. Preparation of Apparatus

7.1 Wind the platinum-rhodium gauze into a roll 3 in. (8

¹ 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.

Current edition approved Dec. 28, 1981. Published April 1982. Originally published as D 3031 - 72. Last previous edition D 3031 - 72 (1977).

² *Annual Book of ASTM Standards*, Vol 05.05.