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Standard Test Method for Hydrogen Sulfide in Natural Gas (Methylene Blue Method)¹

This standard is issued under the fixed designation D 2725; 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—Editorial changes were made throughout in September 1992.

1. Scope

1.1 This test method covers the determination of hydrogen sulfide in natural gas containing not more than 1.0 grain of hydrogen sulfide per 100 ft³ (23 mg/m³ or 16 ppm volume).

1.2 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 *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.* For a specific hazard statement, see Note 1.

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

D 3031 Test Method for Total Sulfur in Natural Gas by Hydrogenation³

3. Summary of Test Method

3.1 A measured sample of the gas is bubbled through zinc acetate solution. Acid solutions of *N,N*-dimethyl-*p*-phenylenediamine, and ferric chloride are added to the zinc acetate solution to react with zinc sulfide to form the methylene blue dye. The methylene blue is determined by measurement of optical absorbance of the solution at a specified wavelength or wavelength range.

4. Apparatus

4.1 *Absorption Flask* (Fig. 1)—This flask is a 50-mL volumetric flask with an enlarged neck (Engler or Redwood type), and is fitted with an open-end bubbler tube.

4.2 *Photoelectric Photometer or Spectrophotometer*—Any instrument designed for the requirements of practical absorption photometry and suitable for measurements at approximately 745 nm may be used. A glass color filter⁴ of low wavelength cutoff type having less than 37 % transmis-

sion below 567 nm and more than 80 % transmission from 608 to 750 nm, or an interference filter with the peak of its band pass at any wavelength between 620 and 750 nm and having less than 0.5 % transmission below 570 nm, shall be used with instruments in which the spectral range is isolated by filters. A photometer equipped with absorption cells with at least 20 to 25-mm path lengths is recommended.

4.3 *Wet Test Meter*—0.05 or 0.1 ft³ (1.5 or 3 L) per revolution, or calibrated aspirator bottle of approximately 0.1-ft³ capacity. (A 1 L per revolution meter is satisfactory.)

5. Reagents

5.1 *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.⁵ 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.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type 2 of Specification D 1193.

5.3 *Ferric Chloride Solution* (27 g/L)—Dissolve 2.7 g of ferric chloride (FeCl₃·6H₂O) in 50 mL of hydrochloric acid (HCl) (sp gr 1.19) and dilute to 100 mL with water.

5.4 *Hydrogen Sulfide*⁶—Cylinder gas, 99.5 % minimum.

5.5 *Iodine, Standard Solution* (0.01 N)—Dissolve 1.27 g of iodine and 4 g of potassium iodide (KI) in water and dilute to 1 L. Store in a cool place in a dark-colored, glass-stoppered bottle. Standardize as follows: Pipet 25 mL of iodine solution into a 250-mL Erlenmeyer or iodine flask, add 25 to 50 mL of water and 5 mL of HCl. Titrate with standard thiosulfate solution until the iodine solution fades to a pale yellow. Add 5 mL of starch solution and titrate to the disappearance of the blue color. The end point will be sharper if the solution is cooled to 5°C or lower.

5.6 *N,N*-dimethyl-*p*-phenylenediamine sulfate, purified⁷ (*Diamine Solution*)—Dissolve 0.11 g in 100 mL of sulfuric

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.

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² *Annual Book of ASTM Standards*, Vol 11.01.

³ Discontinued; see 1991 *Annual Book of ASTM Standards*, Vol 05.05.

⁴ A glass color filter, C. S. No. 3-66, manufactured by Corning Glass Works, Corning, NY 14830, has been found satisfactory for this purpose.

⁵ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. 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."

⁶ Matheson C. P. grade, manufactured by Matheson Gas Products Inc., 30-T Seaview Drive, Secaucus, NJ 07094, has been found satisfactory for this purpose.

⁷ Eastman white label, manufactured by Eastman Chemical Products Inc., P.O. Box 431, Kingsport, TN 37662, has been found satisfactory for this purpose.