



Standard Test Method for Determination of Sulfur Compounds in Natural Gas and Gaseous Fuels by Gas Chromatography and Chemiluminescence¹

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1. Scope

1.1 This test method is primarily for the determination of speciated volatile sulfur-containing compounds in high methane content gaseous fuels such as natural gas. It has been successfully applied to other types of gaseous samples including air, digester, landfill, and refinery fuel gas. The detection range for sulfur compounds, reported as picograms sulfur, is ten (10) to one million (1 000 000). This is equivalent to 0.01 to 1 000 mg/m³, based upon the analysis of a 1 cc sample.

1.2 This test method does not purport to identify all sulfur species in a sample. Only compounds that are eluted through the selected column under the chromatographic conditions chosen are determined. The detector response to sulfur is equimolar for all sulfur compounds within the scope (1.1) of this test method. Thus, unidentified compounds are determined with equal precision to that of identified substances. Total sulfur content is determined from the total of individually quantified components.

1.3 The values stated in SI units are standard. The values stated in inch-pound units are for information only.

1.4 *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:

D 1072 Test Method for Total Sulfur in Fuel Gases²

D 1145 Method of Sampling Natural Gas²

D 1945 Test Method for Analysis of Natural Gas by Gas Chromatography²

D 3609 Practice for Calibration Techniques Using Permeation Tubes³

D 4084 Test Method for Analysis of Hydrogen Sulfide in

Gaseous Fuels (Lead Acetate Reaction Rate Method)²

D 4468 Test Method for Total Sulfur in Gaseous Fuels by Hydrogenolysis and Rateometric Colorimetry²

D 4626 Practice for Calculation of Gas Chromatographic Response Factors⁴

D 4810 Test Method for Hydrogen Sulfide in Natural Gas Using Length-of-Stain Detector Tubes²

E 594 Practice for Testing Flame Ionization Detectors Used in Gas Chromatography⁵

3. Summary of Test Method

3.1 The analysis of gaseous sulfur compounds is challenging due to the reactivity of these substances. They are difficult to sample and analyze. Ideally, analysis is performed on-site to eliminate sample deterioration as a factor in analysis. Sampling must be performed using non-reactive containers, such as Silcosteel® lined vessels, Tedlar bags with polypropylene fittings or the equivalent. Tedlar bag samples require protection from light and heat. Laboratory equipment must be inert or passivated to ensure reliable results.

3.2 A one cc (mL) sample is injected into a gas chromatograph where it is eluted through a megabore, thick film, methyl silicone liquid phase, open tubular partitioning column or other suitable column, and separated into its individual constituents.

3.3 *Sulfur Chemiluminescence Detection*—As sulfur compounds elute from the gas chromatographic column, they are processed in a flame ionization detector (FID) or a heated combustion zone. The products are collected and transferred to a sulfur chemiluminescence detector (SCD). This technique provides a sensitive, selective, linear response to volatile sulfur compounds and may be used while collecting hydrocarbon and fixed gas data from a FID.

3.3.1 *Detectors in Series with a SCD*—A SCD can frequently be used in series with other fixed gas and hydrocarbon detectors. However, regulatory bodies may question detector compatibility and require demonstration of equivalence between a SCD in a multi-detector system and a SCD operated using a FID or combustion zone. The user is referred to USEPA

¹ This test method is under the jurisdiction of ASTM Committee D03 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 05.05.

³ *Annual Book of ASTM Standards*, Vol 11.03.

⁴ *Annual Book of ASTM Standards*, Vol 05.02.

⁵ *Annual Book of ASTM Standards*, Vol 14.01.