



Standard Test Method for Online Measurement of Sulfur Compounds in Natural Gas and Gaseous Fuels by Gas Chromatograph and Electrochemical Detection¹

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1. Scope

1.1 This test method is for on-line measurement of volatile sulfur-containing compounds in gaseous fuels by gas chromatography (GC) and electrochemical (EC) detection. This test method is applicable to hydrogen sulfide, C1 to C4 mercaptans, sulfides and tetrahydrothiophene (THT).

1.1.1 Carbonyl sulfide (COS) is not covered in this test method.

1.1.2 The detection range for sulfur compounds is approximately from 0.1 to 100 ppmv (mL/m^3) or 0.1 to 100 mg/m^3 . The detection range may vary depending on the sample injection volume, chromatographic peak separation and the sensitivity to the specific EC detector.

1.2 This test method describes a GC-EC method employing packed GC columns and a specific detector for natural gas and other gaseous fuel composed of mainly light (C4 and smaller) hydrocarbons. Alternative GC columns, detector designs and instrument parameters may be used, provided that chromatographic separation, quality control and measurement objectives needed to comply with user, or regulator needs or both, are achieved.

1.3 This test method does not intend to identify and measure all individual sulfur species, and is mainly employed for monitoring naturally occurring reduced sulfur compounds commonly found in natural gas and fuel gases or employed as an odorant in these gases.

1.4 The test method is typically employed in repetitive or continuous on-line monitoring of sulfur components in natural gas and fuel gases using a single sulfur calibration standard. Need for a multipoint calibration curve or quality control procedures can be satisfied by making use of procedures delineated in Test Methods D5504, D5623, D6228, D6968, ISO 19739, or GPA 2199.

¹ This test method is under the jurisdiction of ASTM Committee D03 on Gaseous Fuels and is the direct responsibility of Subcommittee D03.12 on On-Line/At-Line Analysis of Gaseous Fuels.

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1.5 The test method can be used for measurement of all sulfur compounds listed in Table 1 in air or other gaseous matrices, provided that no compounds that can interfere with the GC separation and electrochemical detection are present.

1.6 This test method is written as a companion to Practices D5287, D7165 and D7166.

1.7 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.8 *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:²

D3609 Practice for Calibration Techniques Using Permeation Tubes

D4150 Terminology Relating to Gaseous Fuels

D4626 Practice for Calculation of Gas Chromatographic Response Factors

D5287 Practice for Automatic Sampling of Gaseous Fuels

D5504 Test Method for Determination of Sulfur Compounds in Natural Gas and Gaseous Fuels by Gas Chromatography and Chemiluminescence

D5623 Test Method for Sulfur Compounds in Light Petroleum Liquids by Gas Chromatography and Sulfur Selective Detection

D6228 Test Method for Determination of Sulfur Compounds in Natural Gas and Gaseous Fuels by Gas Chromatography and Flame Photometric Detection

D6968 Test Method for Simultaneous Measurement of Sulfur Compounds and Minor Hydrocarbons in Natural Gas and Gaseous Fuels by Gas Chromatography and Atomic Emission Detection

² 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.

TABLE 1 Typical Retention Times of Sulfur Components of Different GC-ECD Runs

GC-ECD instrument	GC-ECD #1	GC-ECD #2	GC-ECD #3
GC-Column and parameters	1/8 in. IDx 70 cm L, N ₂ , 12 mL/min, 65 °C	1.6 mm IDx 1200 mm L, N ₂ , 100 mL/min, 20 °C	4 mm IDx 400 mm L, N ₂ , 100 mL/min, 20 °C
Detector Size	5x20 mm	5x20 mm	30x25 mm
Sulfur Compound	RT (sec.)	RT (sec.)	RT (sec.)
Hydrogen sulfide, H ₂ S	30	30	30
Methyl mercaptan (MeSH)	70	66	60
Ethyl mercaptan (EtSH)	105	150	80
Dimethyl sulfide (DMS)	120	200	80
i-Propyl mercaptan (IPM)	160	240	160
t-Butyl mercaptan (TBM)	220	342	240
n-Propyl mercaptan (NPM)	265	426	290
i-Butyl mercaptan (IBM)	440	...	560
n-Butyl mercaptan (NBM)	585
Thiophane (THT)	900 ^A	720 ^A	2100

^AThe shorter GC column is employed for simultaneous accelerated analysis of THT.

[D7165 Practice for Gas Chromatograph Based On-line/At-line Analysis for Sulfur Content of Gaseous Fuels](#)

[D7166 Practice for Total Sulfur Analyzer Based On-line/At-line for Sulfur Content of Gaseous Fuels](#)

2.2 *ISO Standards*:³

[ISO 19739 Natural gas – Determination of sulfur compounds by gas chromatography](#)

2.3 *GPA Standard*⁴

[GPA 2199 Determination - Determination of Specific Sulfur Compounds by Capillary Gas Chromatography and Sulfur Chemiluminescence Detection](#)

3. Terminology

3.1 Common terminology used in this method are cited in Terminology [D4150](#). Sulfur compounds are commonly referred by their initials (chemical or formula), for example,

3.2 *Abbreviations*:

<i>hydrogen sulfide</i>	= H ₂ S
<i>methyl mercaptan</i>	= MeSH (MM)
<i>ethyl mercaptan</i>	= EtSH (EM)
<i>dimethyl sulfide</i>	= DMS
<i>i-Propyl mercaptan</i>	= IPM
<i>n-Propyl mercaptan</i>	= NPM
<i>t-Butyl mercaptan</i>	= TBM
<i>tetrahydrothiophene</i>	= THT or Thiophane

4. Summary of Test Method

4.1 Gaseous fuel is directly sampled on-line for analysis of sulfur compounds. Samples are introduced to the GC instrument through a sampling system. Sulfur compounds are separated by a GC column and measured by an EC detector. The method requires periodic calibration using certified standards. The test method conforms to the practices stated in Practice [D7165](#).

³ Available from International Organization for Standardization (ISO), 1, ch. de la Voie-Creuse, Case postale 56, CH-1211, Geneva 20, Switzerland, <http://www.iso.ch>.

⁴ Available from Gas Processors Association (GPA), 6526 E. 60th St., Tulsa, OK 74145, <http://www.gasprocessors.com>.

4.2 A fixed volume of the sample (normally 0.25 mL) is injected into a gas chromatograph operating isothermally where components are separated using two chromatographic columns.

4.3 GC-separated sulfur compounds are determined using an electrochemical detector utilizing a chromic acid electrolyte.

5. Significance and Use

5.1 Gaseous fuels, such as natural gas, petroleum gases and bio-gases, contain sulfur compounds that are naturally occurring or that are added as odorants for safety purposes. These sulfur compounds are odorous, corrosive to equipment, and can inhibit or destroy catalysts employed in gas processing and other end uses. Their accurate continuous measurement is important to gas processing, operation and utilization, and is frequently of regulatory interest.

5.2 Small amounts (typically, total of 4 to 6 ppmv) of sulfur odorants are added to natural gas and other fuel gases for safety purposes. Some sulfur odorants are reactive, and may be oxidized to form more stable sulfur compounds having lower odor thresholds which adversely impact the potential safety of the gas delivery systems and gas users. Gaseous fuels are analyzed for sulfur compounds and odorant levels to assist in pipeline integrity surveillance and to ensure appropriate odorant levels for public safety.

5.3 This method offers an on-line technique to continuously identify and quantify individual target sulfur species in gaseous fuel with automatic calibration and validation.

6. Apparatus

6.1 *Chromatograph*—Industrial gas chromatograph with an isothermal oven, automatic injection valve, and software necessary for interfacing to a chromic acid electrochemical detector and designed for the intended application. The GC system must be inert, well-conditioned and passivated with a gas containing the sulfur compounds of interest to ensure reliable results.

6.1.1 *Sample Inlet System*—The gas sample is introduced to the GC by sample loop injection. An automated non-reactive gas sampling valve is employed for a fixed sample loop

injection. The sample injection port must be heated continuously at a temperature significantly ($\sim 10^\circ\text{C}$) above the temperature at which the gas was sampled to avoid sample condensation and discrimination. Inert tubing made of non-permeable, non-sorbing and non-reactive materials, as short as possible and heat traced at the same temperature, should be employed for transferring the sample from a sample source to the gas sampling valve and to the GC inlet system. Silica-coated 316 stainless steel (s.s.) and non-permeable polytetrafluoroethylene (PTFE) type tubing are often employed. Different size fixed-volume sample loops (0.25 to 10.0 mL) may be used to target multiple concentration ranges for components in a gas, provided chromatographic separation and quality control objectives are obtained. The same non-reactive materials are used for the sample loop to avoid possible decomposition or absorption of reactive species. The sampling and GC inlet system must be well conditioned and evaluated frequently for compatibility with trace quantities of reactive sulfur compounds, such as tert-butyl mercaptan. A programmable and computer-controlled multi-stream sample selector can be used to sample fuel gases and calibration gases.

6.1.2 *Column Temperature*—The gas chromatograph must be capable of maintaining an isothermal temperature, normally at 65°C , with temperature variation not exceeding $\pm 0.5^\circ\text{C}$.

6.1.3 *Carrier and Detector Gas Control*—Constant flow control of carrier and detector gases is necessary for optimum and consistent analytical performance. Control is best provided by the use of pressure regulators and fixed flow restrictors. The gas flow rate is measured using a gas flow meter either volumetrically or based upon mass flow rates. Mass flow

controllers, capable of maintaining gas flow constant to $\pm 1\%$ at the required flow rates should be used. The supply pressure of the gas delivered to the gas chromatograph must be at least 69 kPa (10 psi) greater than the regulated gas at the instrument to compensate for system back pressure.

6.1.4 *Detector*—An EC detector, whose operation is based upon the reduction/oxidation reaction between reduced sulfur compounds and a solution of chromic acid (Fig. 1), is used in this method. The detector is set according to the manufacturer's specifications for this particular application. One EC detector is normally employed for measurement. A second column is employed for detection of late-eluting sulfur compounds, such as THT.

6.1.4.1 The detector consists of a glass or methyl polymethacrylate container. The electrodes, two pieces of platinum gauze grids, are arranged vertically in parallel, and are welded in a borosilicate glass tube. These grids are isolated from each other and other conductive materials and connected to an amplifier for data acquisition.

6.1.4.2 The electrolyte, a solution of chromium (VI) oxide in distilled or deionized water (100 g/L or 0.66 mole/L), is contained in an acid-resistant vessel. A tube fitted with the electrodes is dipped into the solution such that the liquid is retained by capillary action within the tube at a level approximately midway between two grids.

6.1.4.3 The gas flow from the GC column is discharged through a narrow glass or PTFE tube (2 mm ID) immediately above the upper grid center (normally 5 mm). Each sulfur compound sequentially elutes and reacts with chromic acid. Possible reaction mechanisms are illustrated as Eq 1 and Eq 2.

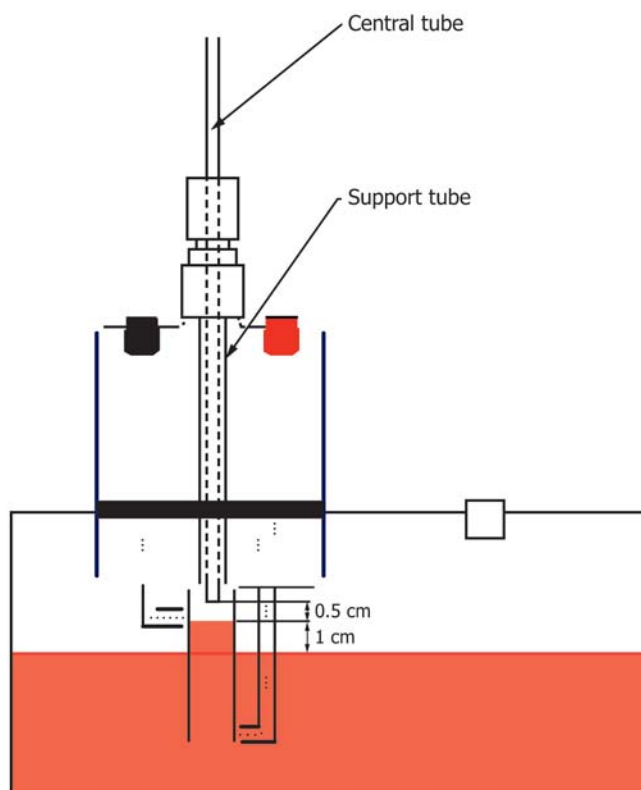
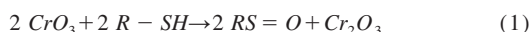


FIG. 1 Typical Electrochemical Detection Cell

The redox reaction occurs on the electrode surface, creating a potential difference between the two electrodes, thus causing a current to be measured (using a low resistance measuring circuit). For example, t-butyl mercaptan is oxidized to t-butyl sulfoxide and chromium oxide (Eq 2).



where:

R = organic moieties, such as C_xH_y



6.2 Column—1200 mm of 1.6 mm ID glass or PTFE tubing packed with 150 to 180 μ m (80 to 100 mesh) Chromosorb W support has been successfully used in performance of this test method. However, other columns that provide adequate retention and resolution characteristics under the experimental conditions as described in 8.1 can be used. A second GC column of the same ID and phase, but of a shorter length, can be employed for faster measurement of late-eluting sulfur compounds such as THT. In this case, two columns are connected to the GC injection system using a 10-port valve to direct sample flow through the appropriate column and then onto the EC detector; thus, allowing measurement of low molecular weight sulfur gases and high molecular weight sulfur gases such as THT from a single sample injection. The elution of high molecular weight sulfur gases such as THT may also be accelerated by increased carrier gas flow rate after the elution of TBM. When samples may contain high boiling or instrument damaging substances, a backflush column may be employed to remove these materials before they reach the chromatographic column and EC detector. The performance of GC columns shall give adequate separation of target sulfur compounds for the particular application.

6.3 Data Acquisition

6.3.1 The device and software must have the following capabilities:

6.3.1.1 Graphic presentation of the chromatogram.

6.3.1.2 Digital display of chromatographic peak areas.

6.3.1.3 Identification of peaks by retention time or relative retention time, or both.

6.3.1.4 Calculation and use of response factors.

6.3.1.5 External standard calculation and data presentation.

6.3.1.6 Instrument control for electrochemical detector operation, such as gas pressure and flow control.

7. Reagents and Materials

7.1 High-pressure Cylinder Reference Gas Standards—Gas standards of high purity with certified stability and accuracy are used.

7.1.1 Sulfur Gas Standards—Single or multiple sulfur compounds in a compressed gas of high purity nitrogen, helium or methane base gas may be used. Care must be exercised in the use of compressed gas standards since they can introduce errors in measurement due to lack of uniformity in their manufacture or instability in their storage and use. The non-mandatory protocol for compressed gas standards cited in Appendix X1 of Test Method D5504 can be used to ensure the quality of standards and to establish traceability to a National

Institute of Standards and Technology (NIST) or other national metrology institute (NMI) standard reference material.

7.1.2 Multiple sulfur gas standard mixes should be used as recommended by a compressed gas standard manufacturer to assure the long term stability of sulfur components. The standard should be re-certified as per manufacturers' recommendations or as needed for regulatory compliance.

7.1.3 Compressed Gas Standard Delivery System—Pressure regulators, gas lines and fittings must be inert, appropriate for the delivery of sulfur gases and well passivated.

7.2 Sulfur Permeation Standards—Gaseous standards generated from individual or a combination of certified permeation tubes and devices at a constant temperature (± 0.1 °C) and a constant flow rate can be used for calibrations. The standard concentration is calculated by mass loss at a fixed temperature and dilution gas flow rate. Permeation devices should be calibrated gravimetrically to the nearest 0.01 mg. Impurities permeated from each device must be detected, measured and accounted for in the mass loss if they are present above a level of 0.1 % of the permeated sulfur species. Permeation devices shall be discarded when the liquid content is reduced to less than 10 % of the initial volume. See Practice D3609 for further information on the proper use of permeation devices.

NOTE 1—Warning: Sulfur compounds may be flammable and may be harmful if ingested or inhaled.

7.3 Carrier Gas—Helium, air or nitrogen of high purity (99.999 % minimum purity). Use of air as a carrier gas is not recommended since many sulfur compounds react with air. Additional purification of carrier gas is recommended using molecular sieves or other suitable agents to remove hydrocarbons, oxygen, or both in helium and nitrogen. A dual stage regulator and a carrier gas pressure sufficiently high to ensure a constant carrier gas flow rate are needed (see 6.1.4).

NOTE 2—Warning: Carrier gas employed may be from a source of compressed gases under high pressure.

7.4 Chromium Oxide—Reagent grade (99.9 % minimum purity).

NOTE 3—Warning: Toxic chemical, handling with rubber gloves and caution. Waste reagent should be chemically reduced and properly disposed.

8. Preparation of Apparatus and Calibration

8.1 Chromatograph—Place in service according to the manufacturer's instructions. Typical operating conditions are shown in Table 2.

8.2 Electrochemical detector—Place the detector in service according to the manufacturer's instructions. In general, carefully remove the glass detector assembly from the empty chromic acid reservoir. Fill the reservoir with chromic acid solution to the mark, approximately 3 cm high. Slowly insert the detector into the reservoir until chromic acid solution reaches and passes the upper grid. Very slowly lift the detector until the level of the solution is at 1 cm below the upper grid. The solution should suspend in the detector tube by capillary action. Repeat the insertion of glass detector if air bubbles are present or the solution level in the detector tube is incorrect, or both.

TABLE 2 Typical Gas Chromatographic Operating Parameters

Gap Sample Loop:	0.25 mL at 10 °C above GC column oven temperature
Injection Type:	Sampling Loop
Carrier Gas:	Helium, air or nitrogen at 12 mL/min with micro EC detector
Column Oven:	Isotherm, 65 °C
Detector:	Reagent and instrument operating parameters as recommended by the GC-EC manufacturer

8.3 *Sample Injection*—A fixed size sample loop is used to conduct a performance check. A linear calibration curve may be determined by using standards of varying concentrations or different sample loop sizes.

8.4 *Chromatography*—A typical chromatogram for sulfur compounds present at ppmv levels is illustrated in Fig. 2. Retention times for compounds in this chromatogram are listed in Column 1 of Table 1. The retention times of selected sulfur compounds using alternative instrument settings are listed for reference in Table 1. The elution sequence and distribution of sulfur peaks should remain approximately the same. Acceptable resolution defined as baseline separation of adjacent peaks is the goal for chromatographic separation, although GC peak broadening is a characteristic of EC detectors with peak overlap occasionally observed. Baseline separation of two peaks using EC detection is normally defined as the EC detector signal of the lead compound returning to a point at least 5 % below the top of the smallest peak. Less than baseline separation may lead to poorer precision and larger bias than

baseline separated peaks. Each GC-EC chromatogram should be examined thoroughly. High concentrations of some hydrocarbons may interfere with measurements; although, typical natural gas hydrocarbon concentrations have not been reported to show interference with sulfur gas measurement. Matrix studies can be conducted to ascertain whether a particular gas composition results in chromatographic or detector interferences, or both.

8.5 *Detector Response Calibration*—Analyze calibration gases to obtain the chromatograms and peak areas for response factor determination as per Eq 3 (Practice D4626). Response factors of different sulfur compounds vary considerably as shown in Table 3. Therefore, response factors for each component of interest are necessary for the accurate determination of specific species. Determine the linear range of detector response toward each sulfur compound. A linear standard curve is constructed and the linear correlation factor is calculated. Detector linearity based upon a multi-point calibration curve is essential for on-line measurement accuracy and should be verified at a frequency recommended by the instrument manufacturer, as required by regulation or as required for process control.

$$RF = C_n/A_n \tag{3}$$

where:

- RF = response factor of sulfur compound
- C_n = concentration of the compound in the sampled gas on a common measurement unit
- A_n = peak area of the compound measured

8.5.1 Example: Consider a 1.0 ppmv of dimethyl sulfide (DMS) in natural gas injected onto a GC with a 0.25 mL fixed sample loop producing a sulfur response of 2,000 counts.

$$1 \text{ PPMv DMS} = 1 \text{ PPMv Sulfur}$$

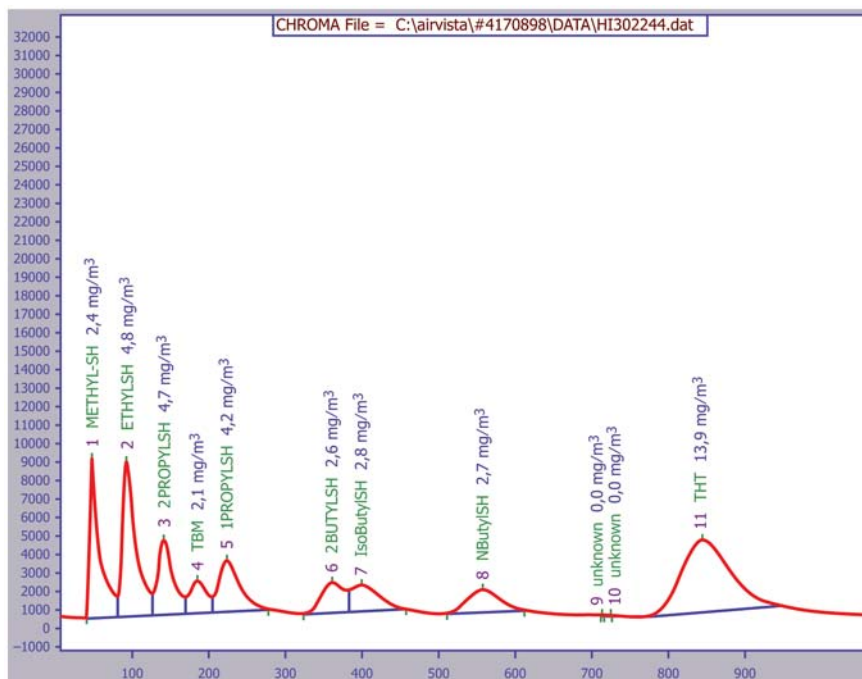


FIG. 2 Typical GC-EC Sulfur Chromatogram of a Mixed Sulfur Standard

TABLE 3 Typical Gas Chromatographic Operating Parameters

Compound	Relative RF vs. DMS
Hydrogen sulfide, H ₂ S	2.3
Methyl mercaptan (MM)	0.75
Dimethyl sulfide (DMS)	1
i-Propyl mercaptan (IPM)	2.12
t-Butyl mercaptan (TBM)	1.8
Thiophane (THT)	0.94

$$\begin{aligned} \text{DMS Response Factor (RF}_{\text{DMS}}) &= 1 \text{ PPMv}/2000 \\ &= 0.0005 \text{ PPMv DMS per area count} \end{aligned}$$

9. Procedure

9.1 Sampling and Preparation of Sample Aliquots:

9.1.1 *Gas Samples*—Sample is transported from a gas source to the GC-EC instrument using an inert on-line sampling system as described in Practice [D7165](#) and [D7166](#). Alternatively, grab, time-proportional (time integrated), or flow-proportional samples may be brought to the laboratory in specially conditioned high-pressure sample containers such as silanized steel flow-through vessels or in Tedlar® bags at atmospheric pressure for off-line analysis.

9.2 *Instrument Setup*—Set up the GC-EC according to the chromatograph operating parameters listed in [Table 2](#).

9.3 *External Standard Calibration*—At least once a day, analyze the calibration standard mix to verify the calibration curve determined in [8.5](#) is still valid and to determine the standard response factors for sample analysis to follow. The difference of response factors found at the beginning and the end of each run or series of runs within a 24-hour period should not exceed 5%. If these criteria are not satisfied, re-calibrate the instrument.

9.4 *Instrument Performance Check*—After calibration has been performed or verified as per [9.3](#), analyze control standards or samples, in replicates, to verify the chromatographic performance including ability to identify components by retention time and accurately determining their concentrations within ±5 % for components present at 0.1 ppmv or greater concentration. System maintenance and re-calibration are required if these criteria cannot be met.

9.5 *Sample Analysis*—Purge the sampling lines from the sample receiving point through the sample loop in the gas chromatograph. Automatically or manually inject sample into the sample loop affixed to a gas-sampling valve. If the sample volume results in a response above the linear range of the detector, change the sample loop. Run the analysis per the conditions specified in [Table 2](#). Obtain the chromatographic data via a computer-based chromatographic data system. Examine the graphic display for any errors (for example, over-range component data), and repeat the injection and analysis if necessary or as required. The difference between corresponding peak areas of repeated runs should not exceed 5 % for compounds present at concentrations equal to or higher than 50 times of their corresponding detection limits. Standard addition and matrix dilution should be carried out to identify possible interferences and improve qualitative and quantitative determination.

10. Calculation

10.1 Determine the chromatographic peak area of each component and use the response factor ([Eq 4](#)) obtained from the calibration run to calculate the amount of each sulfur compound present.

$$C_n = A_n \times RF_n \quad (4)$$

where:

C_n = Concentration of the compound in the gas on a common unit basis (ppmv, mL/m³, etc.)
 A_n = peak area of the compound measured
 RF_n = response factor of the compound (ppmv, mL/m³ per unit area).

11. Quality Assurance

NOTE 4—The following quality assurance (QA) procedures are suggested and may be required in certain applications.

11.1 *Spiked Samples*—A spiked sample is analyzed each day as part of a QA/QC program. Spikes are prepared by quantitative addition of a mixed component calibration gas to a known volume of sample gas. Typically, 20 or 50 mL of a mixed component standard is added to a sample to give a final volume of 100 mL. Acceptable recoveries for components present should fall within 10 % of the theoretical amounts of substances present at 50 times higher than its detection limits to verify nominal system performance. Unacceptable recoveries indicate matrix interference or system malfunction.

11.2 *Calibration Standard Reanalysis*—A standard is reanalyzed after samples every day as part of a QA/QC program. A calibration standard or a control standard or sample should be used daily to verify the response factor (RF) of target sulfur compounds. The day-to-day variation of retention time should not exceed RSD ± 1 %. The day-to-day variation of RFs should not be greater than 5 %. The detector electrolytic level should be maintained and the detector should be re-calibrated for optimal sensitivity if RFs exceeds this limitation. Auto calibration at a specific interval is strongly recommended for continuous on-line monitoring.

11.3 *Blank Analysis*—Confirmation of a lack of carry-over or contamination is recommended and may be required for certain applications. This is accomplished through analysis of a nitrogen or zero air blank. Observation of carryover is typical of injection valve deterioration.

12. Report

12.1 Report the identification and concentration of each individual sulfur compound in common units.

13. Precision and Bias

13.1 *Precision*—This standard has not yet undergone an interlaboratory study to substantiate the listed precision data. The statistical examination of the laboratory test results is as follows:

13.1.1 *Repeatability (Single Operator and Apparatus)*—The difference between successive test results obtained by an automatic online GC-EC analyzer on a single sulfur compound under a constant operating condition on an identical testing sample would, in the long run, in the normal and correct

operation of the test method, exceed the following values by only one case in twenty. See [Table 4](#).

13.1.2 *Reproducibility (Different Operators, Apparatus and sites)*—The difference between two single and independent

results obtained by different operators working on different online GC-EC analyzers using an identical test sample would, in the long run, exceed the following values only one case in twenty. (Experimental results to be determined.)

13.2 *Bias*—Since there is no accepted true sulfur reference material for determining the bias of sulfur measurement, no statement on this can be made.

TABLE 4 Repeatability of Specific Sulfur Compounds Measurement in Nitrogen

Compound	ppmv	Repeatability
H ₂ S	5.85	± 0.047
MM	6.34	± 0.046
IPM	1.15	± 0.016
TBM	2.49	± 0.011
THT	4.91	± 0.070

14. Keywords

14.1 electrochemical detection; gas chromatography; odorants; on-line measurement; sulfur compounds

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