



Standard Test Method for Trace Quantities of Sulfur in Light Liquid Petroleum Hydrocarbons by Oxidative Microcoulometry¹

This standard is issued under the fixed designation D 3120; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope *

1.1 This test method covers the determination of sulfur concentration in the range from 3.0 to 1000 mg/kg in light liquid hydrocarbons boiling in the range from 26 to 274°C (80 to 525°F).

1.2 Other materials falling within the distillation range specified in 1.1, but having sulfur concentrations above 1000 mg/kg, may be tested using appropriate dilutions to bring them within the specified limit. In addition, sample types that may be outside the specified distillation range, such as diesels and biodiesels, may be analyzed by this test method, but the precision statements for these types of materials have not yet been determined.

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are provided for information only. The preferred units are milligrams per kilogram (mg/kg).

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.* For specific hazard statements, see Sections 7 and 8.

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

D 1298 Test Method for Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method³

D 4052 Test Method for Density and Relative Density of Liquids by Digital Density Meter⁴

D 4057 Practice for Manual Sampling of Petroleum and

Petroleum Products⁴

D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products⁴

D 6299 Practice for Applying Statistical Quality Assurance Techniques to Evaluate Analytical Measurement System Performance⁵

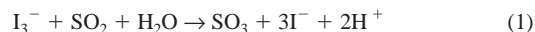
2.2 OSHA Regulations:

OSHA Regulations 29 CFR, paragraphs 1910.1000 and 1910.1200⁶

3. Summary of Test Method

3.1 A liquid sample is introduced into a pyrolysis tube maintained at a temperature between 900-1200°C, having a flowing stream of gas containing 50-80% oxygen and 20-50% inert gas (for example, argon, helium, etc.) Oxidative pyrolysis converts the sulfur to sulfur dioxide, which then flows into a titration cell where it reacts with triiodide ion present in the electrolyte. The triiodide ion consumed is coulometrically replaced and the total current ($I \times t$) required to replace it is a measure of the sulfur present in the sample.

3.2 The reaction occurring in the titration cell as sulfur dioxide enters is:



The triiodide ion consumed in the above reaction is generated coulometrically thus:



3.3 These microequivalents of triiodide ion (iodine) are equal to the number of microequivalents of titratable SO_2 ion entering the titration cell.

4. Significance and Use

4.1 This test method is used to determine the concentration of sulfur in light liquid hydrocarbons, gasoline, and diesels and their additives, where such concentrations of sulfur can be detrimental to their production, performance, and use. The measurement of sulfur in the production and final product of

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

³ *Annual Book of ASTM Standards*, Vol 05.01.

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

⁵ *Annual Book of ASTM Standards*, Vol 05.03.

⁶ Available from U.S. Government Printing Office, Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401.

***A Summary of Changes section appears at the end of this standard.**

gasoline and diesel is required for both regulatory purposes and to ensure maximum life expectancy of catalytic converters used in the automotive industry.

5. Interferences

5.1 This test method is applicable in the presence of total halide concentrations of up to 10 times the sulfur level and total nitrogen concentrations of up to 1000 times the sulfur level.

5.1.1 Stringent techniques shall be employed and all possible sources of sulfur contamination eliminated to attain the quantitative detectability capable with this test method.

5.2 This test method is not applicable in the presence of total heavy metal concentrations (for example, Ni, V, Pb, etc.) in excess of 500 mg/kg (ppm).

6. Apparatus

6.1 *Furnace*—Maintained at a temperature sufficient to completely pyrolyze the organic matrix, 900-1200°C, and completely oxidize the organically bound sulfur to SO₂. Independently controlled inlet and outlet temperature zones are optional. An electrical furnace has been found suitable to use.

6.2 *Pyrolysis Tube*—Fabricated from quartz and constructed so the sample is vaporized in a heated zone before the furnace and swept into the oxidation zone by an inert carrier gas, where the vaporized sample mixes with oxygen and is pyrolyzed. The inlet shall be constructed large enough to accommodate a sample boat completely into the oxidation zone of the pyrolysis tube or allow the direct injection of the sample into the heated zone before the furnace. The pyrolysis tube shall have side arms for the introduction of oxygen and inert carrier gas.

6.3 *Titration Cell*—Consisting of a sensor/reference pair of electrodes to detect changes in triiodide ion concentration, a generator anode-cathode pair of electrodes to maintain a constant triiodide ion concentration, an inlet for gaseous sample from the pyrolysis tube, and an outlet to vent the exit gases from the titration cell. The reference electrode can be either an Ag/AgCl double junction reference electrode or a platinum wire in a saturated triiodide half-cell. The sensor electrode and both the anode and cathode electrodes of the generator are made of platinum. The titration cell shall require mixing, which can be accomplished with a magnetic stir bar, stream of gas, or other suitable means. Other sensor and reference electrodes may be used if they meet the performance criteria of this test method.

NOTE 1—Take care not to use excessive stirring and possibly damage the electrodes with the stir bar. The creation of a slight vortex is adequate.

6.4 *Microcoulometer*—The apparatus' microcoulometer, with variable attenuation and gain control, shall be capable of measuring the potential of the sensing-reference electrode pair and compare this potential to a bias potential. By amplifying this potential difference and applying the difference to a working-auxiliary pair of electrodes (the generator), a titrant is generated. The microcoulometer integrates the amount of current used, calculates the equivalent mass of sulfur titrated and calculates the concentration of sulfur in the sample.

6.5 *Strip Chart Recorder (Optional)*—To monitor and plot the mV potential of the titration cell during the analysis.

6.6 *Flow Control*—The apparatus shall be equipped with flow controllers capable of maintaining a constant supply of oxygen and inert carrier gas.

6.7 *Dryer Tube*—The oxidation of samples produces water vapor which, if allowed to condense between the exit of the pyrolysis tube and the titration cell, will absorb the SO₂ formed and result in low recovery. Steps shall be taken to prevent such an occurrence. This is easily accomplished by placing a phosphoric acid dehydration tube between the titration cell and exit of the pyrolysis tube. Other approaches, such as heating tape or permeation tubes, can be used if precision and accuracy are not degraded.

6.8 *Sampling Syringes*—Microlitre syringes able to accurately deliver 5 to 80 mL of sample are required. The volume injected should not exceed 80% of a syringe's capacity.

6.9 *Sample Inlet System*—Either type of sample inlet system described can be used.

6.9.1 *Boat Inlet System*—The inlet of the pyrolysis tube is sealed to the boat inlet system. The system provides a cooled area before the furnace for the sample boat prior to quantitative introduction of sample into the boat and is purged with the inert carrier gas. The boat driving mechanism then fully inserts the boat into the oxidation zone of the furnace. The drive mechanism shall advance and retract the sample boat into and out of the oxidation zone of the furnace at a controlled and repeatable rate (see Note 2).

6.9.1.1 *Boat Inlet Cooler (Optional)*—Sample volatility and injection volume may require an apparatus capable of cooling the sample boat prior to sample introduction. Thermoelectric coolers (peltier) or recirculating refrigerated liquid devices are strongly recommended. Switching sample boats between each analysis may prove effective, provided sample size is not too large.

6.9.1.2 *Sample Boats*—Quartz or other suitable material which will not react with the sample or sulfur compounds being analyzed and able to withstand the temperatures extremes of the test method.

6.9.2 *Syringe Inlet System*—The system shall deliver a quantitative amount of sample from a microlitre syringe into a heated area before the oxidation zone of the pyrolysis tube at a controlled and repeatable rate. There the sample is volatilized and the inert carrier gas stream purging the heated area transports the volatilized sample into the oxidation zone of the pyrolysis furnace. An adjustable drive mechanism capable of injecting the sample from a microlitre syringe at a constant rate between 0.5 to 1.0 mL/s is required (see Note 2).

NOTE 2—Take care not to introduce the sample too fast into the oxidation zone of the furnace and overload the combustion capacity of the pyrolysis tube. Program the sample inlet system to deliver the sample at a sufficiently controlled and repeatable rate to prevent any incomplete combustion by-products (coke or soot) from forming at the exit of the pyrolysis tube.

6.10 *Balance*—With a weighing precision of ± 0.01 mg.

7. Reagents and Materials

7.1 *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,

TABLE 1 Recommended Preparation of Working Standards

Working Standard Concentration, µg-S/mL	Stock Standard Concentration, µg-S/mL	Volume Stock Standard, mL	Total Volume Working Standard, mL
500	1000	25	50
50	1000	5	100
10	500 ^A	2	100

^A Working standard.

TABLE 2 Recommended Calibration Standards versus Desired Calibration Range

Calibration Range, mg-S/kg	Calibration Standards Concentration		
	Concentration 1, mg-S/mL	Concentration 2, mg-S/mL	Concentration 3, mg-S/mL
Trace–2.5	0.2	1.0	2.5
0.5–50	0.2	5	50
1.0–250	0.5	10	250
2.5–500	1.0	25	500
5.0–1000	2.5	50	1000

where such specifications are available.⁷ 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.

7.2 Purity of Water—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193, Type II and III.

7.3 Quartz Wool—Grade fine.

7.4 Acetic Acid (CH₃COOH)—Glacial acetic acid with specific gravity = 1.05. (**Warning**—Poison. Corrosive. Combustible. May be fatal if swallowed. Causes severe burns. Harmful if inhaled.)

7.5 Phosphoric Acid (85 % w/w)—Orthophosphoric acid (H₃PO₄). (**Warning**—Poison. Corrosive. May be fatal if swallowed. Causes severe burns.)

7.6 Inert Gas—Argon or helium, high purity grade (HP),⁸ used as carrier gas. (**Warning**—Compressed gas under high pressure. Gas reduces oxygen available for breathing.)

7.7 Oxygen—High purity grade (HP),⁸ used as the reactant gas. (**Warning**—Oxygen vigorously accelerates combustion.)

7.8 Gas Regulators—Two-stage gas regulators shall be used for the oxygen and inert carrier gas.

7.9 Cell Electrolyte Solution—Dissolve 0.5 g of potassium iodide (KI) and 0.6 g of sodium azide (NaN₃) in approximately 500 mL of high-purity water, add 6 mL of glacial acetic acid (CH₃COOH), and dilute to 1000 mL or follow the manufacturer's specifications.

NOTE 3—Take care to store bulk quantities of the electrolyte in a dark place. It is recommended to prepare fresh electrolyte at least every three months.

7.10 Sodium Azide (NaN₃), fine granular. (**Warning**—Toxic. Causes eye and skin irritation. Explosive.)

7.11 Potassium Iodide (KI), fine granular.

7.12 Potassium Chloride (KCl), fine granular. Used for the 1M Ag/AgCl double junction reference electrode.

7.13 Potassium Nitrate (KNO₃), fine granular. Used for the 1M Ag/AgCl double junction reference electrode.

7.14 Iodine (I), 20 mesh or less, for saturated reference electrode.

7.15 Toluene, Xylenes, Isooctane—Reagent grade. (Other solvents similar to those occurring in the samples being analyzed are acceptable.) A solvent blank correction is required due to the inherent sulfur present in the solvents used for standard preparation and sample dilution. (**Warning**—Flammable solvents. Harmful if inhaled. Vapors may cause flash fire.)

NOTE 4—The use of solvents with non-detectable levels of sulfur relative to the sulfur content in the sample can make the solvent blank correction unnecessary.

7.16 Dibenzothiophene—FW 184.26, 17.399 % (mass/mass) S (see Note 4).

7.17 n-Butyl Sulfide—FW 146.29, 21.92 % (mass/mass) S (see Note 4).

7.18 Thionaphthene (Benzothiophene)—FW 134.20, 23.90% (mass/mass) S (see Note 4).

NOTE 5—A correction for chemical impurity can be applied if deemed necessary.

7.19 Sulfur, Standard Solution (approximately 1000 mg-S/mL)—Prepare a stock solution by accurately weighing approximately 0.5748 g of dibenzothiophene or 0.4652 g of n-butyl sulfide or 0.4184 g of thionaphthene into a tared 100 mL, type A volumetric flask. Dilute to volume with a selected solvent. This stock can then be further diluted to prepare sulfur working and calibration standards as outlined in Tables 1-3 (see Notes 6-8).

$$\mu\text{g-S/mL} = \frac{A \times B \times C \times 10^6}{100 \text{ mL}} \quad (3)$$

where:

A = grams of standard.

B = weight of fraction sulfur (S) in standard.

C = weight of fraction standard purity.

⁷ Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

⁸ High-purity grade gas has a minimum purity of 99.995 %.

TABLE 3 Recommended Preparation of Calibration Standards

Concentration Working Standard, µg-S/mL	Volume of Working Standard, mL	Total Volume Calibration Standard, mL	Concentration Calibration Standard, µg-S/mL
10	2	100	0.2
10	5	100	0.5
50	2	100	1.0
50	5	100	2.5
50	10	100	5.0
500	5	100	25
500	10	100	50
1000 ^A	25	100	250
500	500
1000 ^A	1000 ^A

^A Standard stock solution.

7.20 Sulfur, Standard Working Solution—Prepare the working standards using standard type A volumetric pipet as outlined in Table 1.

NOTE 6—Working and calibration standards prepared on a regular basis recommended, depending upon frequency of use and age. Stock solutions typically have a useful life of about 3 months.

NOTE 7—Calibration standards can be prepared and diluted on a mass/mass basis, when calculation results are adjusted to accommodate them.

NOTE 8—Stock, working and calibration standards from commercial sources can be used if checked for accuracy and can meet the performance criteria of this test method.

7.21 Quality Assurance (QA) Samples—Samples that are liquid petroleum materials or standards products of known sulfur content that were not used in the generation of the apparatus' calibration curve. These (QA) samples are to be used to check the validity of the testing process as described in Section 11. An ample supply of QA sample material should be available for the intended period of use and shall also be homogenous and stable under the anticipated storage time and conditions.

8. Hazards

8.1 High temperature is employed in this test method. Extra care shall be exercised when using flammable materials near the furnace.

8.2 Consult current OSHA regulations and supplier's Material Safety Data Sheets (MSDS) for materials used in this test method.

9. Sampling

9.1 Obtain a test sample in accordance with Practice D 4057 or Practice D 4177. To preserve volatile components which are in some samples, do not uncover samples any longer than necessary. Samples should be analyzed as soon as possible after collection from bulk supplies to prevent loss of sulfur, or contamination due to exposure or contact with sample container.

9.1.1 (Warning)—Samples collected at temperatures below room temperature can undergo expansion and rupture the container. For such samples, do not fill the container to the top; leave sufficient air space above the sample to allow room for expansion.)

9.2 If the test sample is not used immediately, thoroughly mix the container prior to taking a test specimen.

10. Preparation of Apparatus

10.1 Carefully insert the quartz pyrolysis tube into the furnace, and connect the oxygen and inert carrier gas lines.

10.2 Connect the boat drive system or syringe drive system to the pyrolysis tube.

10.3 Perform a leak check according to the manufacturer's instructions.

10.4 Assemble the titration cell, and add all required solutions, including cell electrolyte, according to the manufacturer's instructions.

10.5 Connect the titration cell to the apparatus according to the manufacturer's instructions.

10.6 Adjust the flow of gases and set the furnace temperatures and instrumental parameters according to the manufacturer's instructions.

10.7 If using a boat inlet system, pre-bake the sample boats used for the analysis per the manufacturer's instructions.

11. Calibration and Standardization

11.1 Following the manufacturer's recommended procedures, set the operational parameters of the apparatus for internal calibration. If the apparatus is not so equipped, analyze the solvent blank and calibration standards, record their values as µg or ng of sulfur, and manually generate a calibration curve.

11.2 Calibration Standards—Select the desired calibration range from Table 2. Prepare the appropriate calibration standards for the range selected as outlined in Table 3. The calibration standards concentrations are calculated based on the formula below:

$$\text{Calibration Standard Concentration: } \mu\text{g-S/mL} = \frac{A \times B}{100 \text{ mL}} \quad (4)$$

where:

A = µg-S/mL of working or stock solution.

B = mL of working or stock solution.

NOTE 9—A calibration curve with three standards is recommended. It is recommended to use standards covering the range of sulfur concentrations expected in the samples being analyzed. Calibration curves with one or two standards can be used if it can be shown the calibration curve is linear over the concentration range being measured and the standards meet the performance criteria of this test method. Larger series of standards can also be used if it can be shown the calibration curve is linear over the concentration range being measured and the standards meet the performance criteria of this test method.

NOTE 10—The use of calibration standards with different concentrations is possible provided they cover the expected concentration range for the samples being analyzed and meet the performance criteria of this test method.

11.3 Select the appropriate syringe based on the recommended sample size in Table 4 for the concentration of standards used to calibrate the apparatus.

11.4 Flush the microlitre syringe several times with the sample prior to analysis. If bubbles are present in the liquid column, flush the syringe and withdraw a new sample.

11.5 The sample size can be determined volumetrically or by mass with a syringe. The sample size should be 80% or less of the syringe's capacity.

11.5.1 *Volumetric Measurement*—Obtain the volume of injected material by filling the syringe to the selected level, retracting the plunger so the lower meniscus falls on the 10% scale mark, and recording the volume of liquid in the syringe. After the sample has been injected, retract the plunger again so the lower liquid meniscus falls on the 10% scale mark, and record the volume of liquid in the syringe. The difference between the two readings is the volume of sample injected.

NOTE 11—An automatic sampling and injection device can be used in place of the procedure described for the manual syringe injection.

11.5.2 *Mass Measurement*—The sample syringe may be weighed before and after the injection to determine the amount of sample injected. This technique provides greater precision than the volume delivery method, provided a balance with a precision of ± 0.01 mg is used.

11.5.3 Once the appropriate sample size has been measured into the microlitre syringe, promptly and quantitatively deliver the sample into the apparatus. Again, there are two alternative techniques available.

11.5.4 *Boat Inlet System*—Add the sample by slowly discharging quantitatively the contents of the syringe into a sample boat containing quartz wool, being careful to include the last drop from the tip of the syringe needle. Remove the syringe and immediately start the analysis. The apparatus' baseline should remain stable until the boat approaches the furnace and volatilization of sample begins (see Note 12). The program of the inlet system should keep the boat completely in the oxidation zone of the furnace a time (residence time) sufficient to totally oxidize the sample before withdrawing the sample boat out of the furnace (see Note 13). Allow at least 2 min for cooling before the next sample injection after the boat has reached the fully retracted position (see Note 14).

NOTE 12—It is recommended, if necessary, to reduce the boat speed, or briefly pause the boat, or both, as it is introduced into the oxidation zone of the furnace to ensure complete sample combustion and avoid overload-

ing the combustion capacity of the system.

NOTE 13—The residence time required to completely oxidize any remaining residue in the boat is dependent on the sample matrix. A 120-s residence time is usually sufficient. Increase the residence time, if necessary, to ensure complete oxidation of the sample.

NOTE 14—The level of boat cooling required prior to sample injection is directly related to the volatility and size of sample. The use of a boat cooling device, an increase in boat cooling time, or both, can be used to control the vaporization of sample until the boat approaches the furnace.

11.5.4.1 Reduce the boat drive introduction rate, sample size or both if coke or soot is observed on the exit end of the pyrolysis tube. Increase the residence time for the boat in the oxidation zone of the furnace if coke or soot is observed on the boat after the analysis is finished.

11.5.5 *Syringe Inlet System*—For direct injection, carefully insert the syringe needle into the inlet of the combustion tube and attach the syringe to the driving mechanism. Allow time for the sample residues to be volatilized from the needle (needle blank). Once the baseline has been reestablished, immediately start the analysis. Remove the syringe once the sample has been syringe once the sample has been completely injected and the analysis is finished.

NOTE 15—To avoid overloading the combustion capacity of the system, injection rates between 0.5-1.0 $\mu\text{L/s}$ are recommended.

11.5.5.1 Reduce the sample size, rate of injection, or both, for the direct injection the test sample into the furnace if coke or soot is observed on the exit end of the pyrolysis tube.

11.6 Calibrate the instrument using one of the following two techniques:

11.6.1 *Automatic Internal Calibration*—Measure the calibration standards and solvent blank using one of the procedures described in 11.3–11.5.5. Analyze the solvent blank and each standard a minimum of three times. Calibrate the apparatus per the manufacturer's instructions. Use either a first or second order linear regression and use this calibration curve to determine the amount of sulfur in the unknown sample. The system's performance shall be checked each day of use. (See Section 12.)

11.6.2 *Manual Calibration Curve*—Measure the solvent blank and calibration standards using one of the procedures described in 11.3–11.5.5. Analyze the solvent blank and each calibration standard a minimum of three times and record their absolute values in either μg or ng sulfur. Subtract the averaged blank value from each calibration standard and construct a curve plotting the mass of sulfur measured for each calibration standard (y-axis) versus the theoretical mass of sulfur injected. Perform a first or second order linear regression and use the calibration curve to determine the amount of sulfur in the unknown sample. The system's performance shall be checked each day of use. (See Section 12.)

NOTE 16—Not all of the sulfur in the sample exits the oxidation zone of the furnace as titratable SO_2 . In the strongly oxidative conditions of the pyrolysis tube, some of the sulfur is also converted to SO_3 , which does not react with the titrant. Accordingly, sulfur standards appropriate to the sample's boiling range and sulfur type is recommended to guarantee adequate calibration. Recoveries less than 75% are to be considered suspect. If recoveries below 75% are observed, it is recommended the operator check the coulometric system, measuring parameters, and operating techniques are in proper order. If the apparatus is being operated

TABLE 4 Recommended Sample Size for Expected Sample Concentrations

Sample Concentrations, mg-S/kg	Injected Volume, μL	Mass Sulfur, $\mu\text{g-S}$
Trace–5.0	80	Trace–0.25
0.5–50	60	0.03–1.5
1.0–250	30	0.03–5.0
2.5–500	20	0.05–5.0
5.0–1000	10	0.05–10

properly, recoveries between 75 to 90% are to be expected.

11.7 If the fraction of sulfur converted to SO₂(recovery factors) drops below 75% of the standard solutions, prepare fresh standards to check if the standards have been altered. Procedural details should be reviewed if a low recovery factors persist.

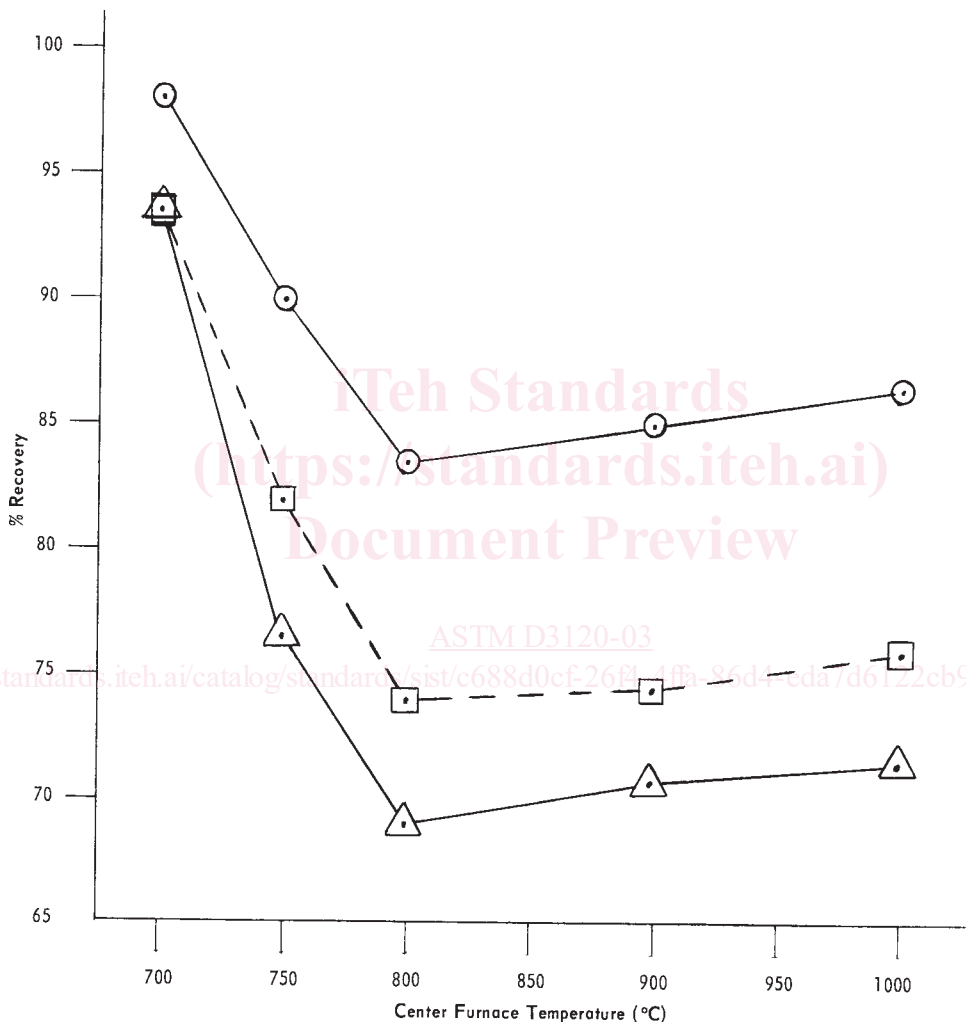
11.8 *Cleaning and Recalibration*—Clean any coked or sooted parts per the manufacturer’s instructions. Assemble and leak check the apparatus per the manufacturer’s instructions after any cleaning or adjustment. Repeat instrument calibration prior to reanalysis of test samples.

12. Quality Assurance (QA)

12.1 Confirm the performance of the instrument of the test procedure by analyzing a quality control (QC) sample after each calibration and at least each day of use thereafter.

12.2 When QC/QA protocols are already established in the testing facility, these can be used when they confirm the reliability of the test result.

12.3 When there is not QC/QA protocol established in the testing facility, Appendix X1 can be used as the QC/QA system.



Oxidative sulfur system: Thiophene in cyclohexane (10 ppm S) using 0.06% azide electrolyte

Legend	Flow rate (cc/min)		
	Oxygen	Argon	O ₂ /Ar ratio
○—○	40	160	1:4
□- -□	100	100	1:1
△—△	160	40	4:1

FIG. 1 Percent Recovery versus Temperature (°C)