



Designation: **D1552—15 D1552 – 16**

Standard Test Method for Sulfur in Petroleum Products by High Temperature Combustion and IR ~~Detection~~Infrared (IR) Detection or Thermal Conductivity Detection (TCD)¹

This standard is issued under the fixed designation D1552; 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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

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

1. Scope*

1.1 This test method covers ~~a procedure~~procedures for the determination of total sulfur in petroleum products including lubricating oils containing additives, and in additive concentrates. This test method is applicable to samples boiling above 177 °C (350 °F) and containing a mass fraction of sulfur ~~not less than 0.06 %~~. ~~This procedure uses IR detection following pyrolysis in a resistance between 0.22 % and 24.2 %~~. Other sulfur concentrations may be analyzed, but the precision stated may or may not apply. These procedures use IR detection or TCD following combustion in a furnace.

1.2 Petroleum coke containing ~~up to 8 mass % sulfur can be analyzed~~a mass fraction of sulfur between 2.53 % to 3.79 % sulfur may be analyzed. Other sulfur concentrations may be analyzed, but the precision stated may or may not apply.

NOTE 1—The D1552–08 (2014) version of this standard contained two other procedures using iodate titrations. Since these procedures are no longer being used in the industry laboratories based on a survey of D02.SC 3 laboratories conducted in September 2014, they are being deleted. For earlier information on the deleted procedures, D1552–08 (2014) may be perused.

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses 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:²

~~D1266~~D4057 Test Method for Sulfur in Petroleum Products (Lamp Method)Practice for Manual Sampling of Petroleum and Petroleum Products

~~D4057~~D4177 Practice for Manual Automatic Sampling of Petroleum and Petroleum Products

D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance

D6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products and Lubricants

D6792 Practice for Quality System in Petroleum Products and Lubricants Testing Laboratories

3. Summary of Test Method

3.1 The sample is weighed ~~into a special ceramic boat which is then~~ and placed into a combustion furnace at 1350 °C (2460 °F) ~~in an oxygen atmosphere~~. furnace having an oxygen atmosphere sufficient to combust the entire sample and a temperature between 1150 °C and 1450 °C for Procedure A or 1150 °C for Procedure B. Most sulfur present is combusted to SO₂, which is then measured with an infrared a detector after moisture and dust are removed by traps. ~~A microprocessor~~The instrument calculates the mass percent sulfur from the sample ~~weight~~mass, the integrated detector signal, and a predetermined calibration factor. The calibration factor is determined using standards approximating the material to be analyzed.

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.03 on Elemental Analysis.

Current edition approved April 1, 2015 July 1, 2016. Published May 2015 August 2016. Originally approved in 1958. Last previous edition approved in 2014 2015 as D1552–08 (2014) D1552 – 15.^{ε1}. DOI: 10.1520/D1552-15.10.1520/D1552-16.

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.

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

3.1.1 *Procedure A*—After combustion of the sample and subsequent moisture/dust removal, SO₂ is measured using infrared (IR) detection.

3.1.2 *Procedure B*—After combustion of the sample and subsequent moisture/dust removal, SO₂ is measured using thermal conductivity detection. An apparatus utilizing TCD may require the sample gas to pass an oxygen scrubber and adsorption/desorption traps to allow passing of contaminants prior to measuring SO₂.

4. Significance and Use

4.1 This test method provides a means of monitoring the sulfur level of various petroleum products and additives. This knowledge can be used to predict performance, handling, or processing properties. In some cases the presence of sulfur compounds is beneficial to the product and monitoring the depletion of sulfur can provide useful information. In other cases the presence of sulfur compounds is detrimental to the processing or use of the product.

5. Apparatus

5.1 ~~Resistance Type Furnace, Combustion Furnace:~~ capable of maintaining a temperature of at least 1350 °C (2460 °F).

5.1.1 *Procedure A*—Furnace capable of maintaining a constant temperature (between 1150 °C minimum and 1450 °C maximum) sufficient to ensure quantitative recovery of sulfur as its corresponding gas SO₂.

5.1.2 *Procedure B*—Furnace capable of maintaining a temperature (1150 °C) sufficient to ensure quantitative recovery of sulfur as its corresponding gas SO₂.

5.2 ~~Absorber,~~ as described in Test Method [D1266](#).

5.2 ~~Combustion and IR Sulfur Detection System,~~ comprised of automatic balance, oxygen gas flow controls, drying tubes, combustion furnace, infrared detector and microprocessor. ~~The furnace shall be capable of maintaining a nominal oxygen scrubber, adsorption/desorption traps as required, combustion furnace, combustion boats or tin (Sn) containers as required and either an IR (Procedure A) or TCD (Procedure B) SO₂ operating temperature of 1350 °C (2460 °F) detector.~~

5.3 Sieve, 60 mesh (250 μm).

6. Reagents and Materials

6.1 *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.³ 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.

6.2 ~~Combustion Promoter—Magnesium Oxide (Com-Aid) oxide (MgO), tungsten trioxide (WO₃), or Alumina—aluminum oxide (Al₂O₃).~~ Alternatively, COM-CAT, a dual promoter that is both a fixing agent and oxidizing agent, may be used.⁴

6.3 ~~Magnesium Perchlorate: Anhydrous—Drying Agent,~~ Magnesium perchlorate: anhydrous (Mg(ClO₄)₂) or phosphorus pentoxide (P₂O₅). (**Warning**—In addition to other precautions, handle magnesium perchlorate with care. Avoid contacting it with acid and organic materials. Reactions with fuel may be violent.)

6.4 *Oxygen (Extra Dry)*—The oxygen shall be at least 99.5 % pure and show no detectable sulfur by blank determination. (**Warning**—Oxygen vigorously accelerates combustion.)

6.5 ~~Phosphorus Pentoxide—Inert Gas—(Helium, Argon),~~ argon, high purity grade, 99.995 % minimum purity, as required by manufacturer's recommendations.

6.6 ~~Ascarite,~~ 8 mesh to 20 mesh.

6.7 ~~Vanadium Pentoxide,~~ anhydrous, powdered V₂O₅.

6.6 *Quality Control (QC) Sample(s)*, preferably are portions of one or more petroleum products that are stable and representative of the samples of interest. These QC samples can be used to check the validity of the testing process and performance of the instrument as described in Section 11.

7. Sampling

7.1 Take samples in accordance with the instructions in Practice [D4057](#) or [D4177](#).

³ *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.

⁴ The sole source of supply of COM-CAT known to the committee at this time is LECO Corporation, 3000 Lakeview Ave. St. Joseph, MI 49085 USA. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

8. Preparation of Apparatus

8.1 ~~Resistance-Type Furnace-IR Detection~~—Assemble and adjust apparatus according to manufacturer’s instructions. Initialize microprocessor; instrument, check power supplies, set oxygen gas pressure and flows, and set furnace temperature to 1350 °C (2460 °F); temperature.

8.1.1 Condition a fresh anhydrous scrubber with four coal samples when analyzing petroleum coke samples, or with four petroleum product samples the instrument with samples that are representative or typical of the sample types to be analyzed. During the interlaboratory study, laboratories analyzed between one and five conditioning samples.

8.1.2 Calibrate the automatic balance according to manufacturer’s instructions.

9. Standardization

9.1 *Determination of Standardization Factor:*

9.1.1 Because effects such as sample volatility can also affect the relative recovery as SO₂ of the sulfur originally present in the sample, it is necessary to determine a standardization factor. Proceed as described in Sections 9 through 12, using an oil sample of similar type to the unknown sample and of accurately known sulfur content.⁵

9.1.2 Determine the calibration factor for the particular type of sample to be analyzed (lubricating oil, petroleum coke, residual fuel) as recommended by the manufacturer.

9.2 *Quality Control*—Run a suitable analytical quality control sample several times daily. When the observed value lies between acceptable limits on a quality control chart, proceed with sample determinations.

10. Preparation of Coke

10.1 It is assumed that a representative sample has been received for analysis. If the sample of coke received is not dry, it is recommended that the sample be dried prior to grinding.

10.2 Grind and sieve the sample received so as to pass a 60 mesh (250 μm) sieve.

10.3 Dry the sieved material to constant weight at 105 °C to 110 °C.

11. Analysis of Quality Control Samples

11.1 A QC sample shall be analyzed each day samples are analyzed to verify the testing procedure and instrument performance. Additional QC samples may be analyzed. The QC samples shall be treated as outlined in Section 12, depending upon the type of furnace set-up used by the lab.

11.2 When QC/Quality Assurance (QA) protocols are already established in the testing facility, these may be used to confirm the reliability of the test result.

11.3 When there is no QC/QA protocol established in the testing facility, Appendix X1 can be used as the QC/QA system.

12. Procedure with Resistance Furnace-IR Detection Procedures

12.1 Allow the system to warm up and the furnace to reach operating temperature.

12.2 After homogeneity of the sample is assured, select the appropriate sample size as recommended by the instrument manufacturer. As an example, for liquid samples, take up to 0.13 g for analysis and for solid samples, take up to 0.4 g for analysis.

12.3 Determine and store the system blank value.

12.1 Weigh solid samples into combustion boats and record the net weights. For liquid samples, follow 12.4.1 – 12.4.4. It is possible to weigh and store several weights in the microprocessor before beginning a series of burns. *Procedure A—Combustion with Infrared (IR) Detection:*

12.1.1 Fill the combustion boat to one-third capacity with evenly spread MgO powder. Following instrument manufacturer recommendations, allow the system to warm up and the furnace to reach an operating temperature between 1150 °C minimum and 1450 °C maximum.

12.1.2 Form a slight trench in the MgO powder with a scoop. Mix or swirl the test sample thoroughly to ensure homogeneity. Select the appropriate test specimen size as recommended by the instrument manufacturer. As an example, for liquid samples, take up to 0.13 g for analysis and for solid samples, take up to 0.4 g for analysis.

12.1.3 Determine and store the system blank value.

12.1.4 Place the combustion boat on the balance and weigh an appropriate amount of the sample into the trench in the MgO. Weigh solid samples into combustion boats or containers and record the net masses. For liquid samples, follow 12.1.4.1 powder to 12.1.4.4 Record and enter the weight.

12.1.4.1 Following manufacturer recommendations; add combustion promoter, if used, to combustion boat or container using scoop or spatula. Fill the combustion boat to one-third capacity with evenly spread MgO powder.

⁵ Residual fuel oil Standard Reference Materials may be obtained from the National Institute of Standards and Technology or other sources.