



Standard Test Method for Active Sulfur in Cutting Oils¹

This standard is issued under the fixed designation D 1662; 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.

1. Scope

1.1 This test method covers the determination of active sulfur in cutting oils. This test method applies to sulfur reactive with copper powder at a temperature of 150°C (300°F) in cutting fluids containing both natural and added sulfur.

NOTE 1—It has not been established by ASTM as to how the active sulfur content thus determined may relate to field performance of the cutting fluid.

1.2 The values stated in SI 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.*

2. Referenced Documents

2.1 ASTM Standards:

D 129 Test Method for Sulfur in Petroleum Products (General Bomb Method)²

D 130 Test Method for Detection of Copper Corrosion from Petroleum Products by the Copper Strip Tarnish Test²

3. Terminology

3.1 Definition of a Term Specific to This Standard:

3.1.1 *active sulfur*—the sulfur in a cutting fluid that will react with metallic copper at a temperature of 149°C (300°F) under the prescribed conditions.

4. Summary of Test Method

4.1 A portion of the sample is treated with copper powder at 149°C (300°F). The copper powder is filtered from the mixture. Active sulfur is expressed as the difference between the sulfur contents of the sample, as determined by Test Method D 129 before and after treatment with copper.

¹ This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.L on Industrial Lubricants.

Current edition approved Sept. 15, 1992. Published November 1992. Originally published as D 1662 – 59. Last previous edition D 1662 – 86.

This test method was prepared under the joint sponsorship of the American Society of Lubrication Engineers. Accepted by ASLE January 1969.

² *Annual Book of ASTM Standards*, Vol 05.01.

5. Significance and Use

5.1 This test method measures the quantity of sulfur available to react with metallic surfaces to form solid lubricating aids at the temperature of the test. Rates of reaction are metal type, temperature, and time dependent.

6. Apparatus

6.1 The apparatus shall be the same as that described in Test Method D 129 in addition to the following:

6.2 *Stirrer*, constructed of glass in the form of an inverted T. A flat blade, approximate length 25 mm, height 6 mm, thickness 1 mm, shall be attached to a glass rod 6 mm in diameter, in such a way that the blade is symmetrical with the rod and has its flat surface in the vertical plane. Alternatively, a glass-coated magnetic stirring bar 9.5 by 34.9 mm ± 2 mm (3/8 by 1 3/8 in.) can be used.

6.3 *Stirring Apparatus*—An electric motor capable of maintaining a speed of 500 ± 25 rpm. Alternatively, when using the glass-coated stirring bar, a combination magnetic stirrer-hot plate is required.

6.4 *Hot Plate*, electric, or other convenient heat source capable of maintaining the sample at a temperature of 150 ± 3°C (300 ± 5°F).

6.5 *Beaker*, 200-mL, tall-form of heat-resistant glass, with a pour-out spout.

7. Materials

7.1 The materials shall be the same as described in Test Method D 129 in addition to the following:

7.2 *Copper Powder*.³

8. Procedure

8.1 Determine the sulfur concentration of the sample to be tested in accordance with Test Method D 129.

8.2 Place 50 ± 2 g of sample in a 200-mL tall-form beaker, lower the stirrer to within 5 mm of the bottom of the beaker and add 5 ± 0.25 g of copper powder and heat to 150 ± 2°C (300 ± 5°F) while stirring at 500 ± 25 rpm. If a magnetic stirrer is used, rotate the stirring bar at 500 ± 25 rpm. When 150 ± 2°C (300 ± 5°F) is reached, add an additional 5 ± 0.25 g copper powder. Continue stirring at 150 ± 2°C (300 ± 5°F) for 30 ±

³ The following copper powders have been found satisfactory: Atlantic Flake Powder No. 5010, Fisher C-431, Copper Dust, and LaPine No. 72696 Copper Powder.

 D 1662

1 min. At the end of this period, stop stirring and insert a copper strip prepared in accordance with Test Method D 130 for 10 ± 0.25 min. If there is any evidence of stain on the strip, recommence stirring and add an additional 5 ± 0.25 g of copper powder. Continue stirring the mixture at $150 \pm 2^\circ\text{C}$ for 30 ± 1 min. Again, insert a copper strip as previously stated for 10 ± 0.25 min. Repeat this procedure until the copper strip shows no stain, maintaining the temperature of the mixture at $150 \pm 2^\circ\text{C}$.

8.3 Filter the sample through filter paper at the end of the heating period. The filtration apparatus may be placed in an oven maintained at $100 \pm 1^\circ\text{C}$ ($212 \pm 2^\circ\text{F}$) for more rapid filtration.

NOTE 2—A filter paper sufficiently fine to prevent passage of any particulate matter must be used, as the presence of copper sulfide in the filtrate will give erroneous results.

8.4 Determine the sulfur content of the filtered sample using Test Method D 129.

9. Calculation

9.1 Calculate the active sulfur concentration of the sample as follows:

$$\text{Active sulfur, wt \%} = A - B \quad (1)$$

where:

A = weight % sulfur of the untreated sample, and

B = weight % sulfur of the treated sample.

10. Precision and Bias

10.1 *Precision*—It is not practical to specify the precision of the procedure in this test method for measuring active sulfur because a round robin has not been completed.

10.1.1 Work is underway to improve and determine the proper precision of this test method. Previously obtained precision on this test method is not known to have been obtained in accordance with currently accepted guidelines (for example in Committee D-2 Research Report RR-D-2-1007, “Manual on Determining Precision Data for ASTM Methods on Petroleum Products and Lubricants”).⁴

10.2 *Bias*—The procedure in this test method has no bias because values of active sulfur can be defined only in terms of a test method.

11. Keywords

11.1 active sulfur test; cutting oils; metalworking oils

⁴ Annual Book of ASTM Standards, Vol 05.03.

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 100 Barr Harbor Drive, West Conshohocken, PA 19428.