Designation: D1662 – 08 (Reapproved 2014)

Standard Test Method for Active Sulfur in Cutting Oils[1]

This standard is issued under the fixed designation D1662; 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 (302°F) in cutting fluids containing both natural and added sulfur.

NOTE 1—It has not been established by ASTM Subcommittee D02.L0 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 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]

D130 Test Method for Corrosiveness to Copper from Petroleum Products by Copper Strip Test

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *active sulfur*—sulfur in a cutting fluid that will react with metallic copper at a temperature of 150°C (302°F) under the prescribed conditions.

4. Summary of Test Method

4.1 A portion of the sample is treated with copper powder at 150°C (302°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 before and after treatment with copper.

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 *Filter Paper,* 2.5 µm retention size.

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 ± 2 mm (⅜ by 1⅜ in.) can be used.

6.3 *Stirring Apparatus,* 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 ± 2°C (302 ± 5°F).

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

7. Materials

7.1 *Diluent,* sulfur-free white oil, methyl lardate or dialkylbenzene.

7.2 *Copper Powder,* >99 %, <75 µm (200 mesh).

8. Procedure

8.1 Determine the sulfur concentration of the sample to be tested using any accepted method that has precision of ±0.15 % for sulfur.

NOTE 2—For best results, dilute the sample with sulfur-free white oil, methyl lardate or dialkylbenzene to a sulfur content of 2 to 4 %.

8.2 Place 50 ± 2 g of sample or sample dilution in a 200-mL tall-form beaker, lower the stirrer to within 5 mm of

[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.L0.01 on Metal Removal Fluids and Lubricants.

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This test method was prepared under the joint sponsorship of the American Society of Lubrication Engineers (ASLE) and accepted by ASLE in January 1969.

[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**

TABLE 1 Active Sulfur (%)

Materials	Average	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
	Xbar	Sr	SR	r	R
A	0.9454	0.0348	0.0706	0.0974	0.1976
B	3.3687	0.1022	0.5787	0.2862	1.6205
C	0.3902	0.082	0.2969	0.2296	0.8312
D	1.9643	0.1551	0.4668	0.4343	1.3070

the bottom of the beaker and add 5 ± 0.25 g of copper powder and heat to 150 ± 2°C (302 ± 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 (302 ± 5°F) is reached, add an additional 5 ± 0.25 g copper powder. Continue stirring at 150 ± 2°C (302 ± 5°F) for 30 ± 1 min. At the end of this period, stop stirring and insert a copper strip prepared in accordance with Test Method D130 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 ± 2°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 ± 2°C.

8.3 At the end of the heating period, filter the reaction mixture through the filter paper until the filtrate is clear with no visible precipitate.

NOTE 3—The filtration apparatus may be placed in an oven maintained at 100 ± 1°C (212 ± 2°F) for more rapid filtration.

8.4 Determine the sulfur content of the filtered sample using the same method as used in 8.1.

9. Calculation

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

$$\text{Active sulfur, wt \%} = A - B$$

where:

A = weight % sulfur of the untreated sample, and
B = weight % sulfur of the treated sample.

10. Precision and Bias[3]

10.1 The precision of this test method is based on an interlaboratory study conducted in 2006–2007. Four fluid samples having active sulfur levels of 0.9%, 3.6%, 0.3%, and 2.1% were run in triplicate by nine laboratories to determine the intralaboratory and interlaboratory precision of Test Method D1662.

10.1.1 *Repeatability*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the "*r*" value for that material; "*r*" is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

10.1.2 *Reproducibility*—Two test results should be judged not equivalent if they differ by more than the "*R*" value for that material; "*R*" is the interval representing the difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

10.1.3 Any judgment in accordance with the statements in 10.1.1 or 10.1.2 would have an approximate 95% probability of being correct.

10.1.4 Results from the interlaboratory study are summarized in Table 1 and Table 2 and are available as a research report.

10.2 *Bias*—No accepted reference material suitable for determining the bias was analyzed as part of this study, therefore no statement on bias can be made at this time.

11. Keywords

11.1 active sulfur test; cutting oils; metal removal fluids

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1628.

TABLE 2 Material Description

A	Fluid 1	1% total, 0.9% active sulfur source A in paraffinic mineral oil
B	Fluid 2	4% total, 3.6% active sulfur source A in paraffinic mineral oil
C	Fluid 3	1% total, 0.3% active sulfur source B in paraffinic mineral oil
D	Fluid 4	4% total, 2.1% active sulfur source A+B in paraffinic mineral oil

SUMMARY OF CHANGES

Subcommittee D02.L0 has identified the location of selected changes to this standard since the last issue (D1662–07) that may impact the use of this standard. (Approved May 1, 2008.)

(1) Added Precision and Bias information and accompanying research report.

Subcommittee D02.L0 has identified the location of selected changes to this standard since the last issue (D1662–92(2007)) that may impact the use of this standard. (Approved Dec. 1, 2007.)

(1) Test Method D129 is no longer the required sulfur determination method. Any accepted method in accordance with 8.1 may be used.
(2) The filter paper and copper powder to be used are more closely defined.
(3) Note 2 suggests diluting the sample if total sulfur is high.
(4) 8.3 requires that the filterate have no visible precipitate.