

An American National Standard

AMERICAN SOCIETY FOR TESTING AND MATERIALS 100 Barr Harbor Dr., West Conshohocken, PA 19428 Reprinted from the Annual Book of ASTM Standards. Copyright ASTM

# Standard Test Method for Detection of Copper Corrosion from Lubricating Grease<sup>1</sup>

This standard is issued under the fixed designation D 4048; 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 ( $\epsilon$ ) 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 detection of the corrosiveness to copper of lubricating grease.

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. For specific hazard statements, see Note 1.

#### 2. Referenced Documents

2.1 ASTM Standards:

D 97 Test Method for Pour Point of Petroleum Oils<sup>2</sup>

D 130 Test Method for Detection of Copper Corrosion from Petroleum Products by the Copper Strip Tarnish Test<sup>2</sup> D 2500 Test Method for Cloud Point of Petroleum Oils<sup>2</sup> 2.2 Adjuncts: *ASTM* 

Copper Strip Corrosion Standard<sup>3</sup>

#### 3. Terminology

3.1 There are no terms in this method that require new or 6 other than dictionary definitions.

#### 4. Summary of Test Method

4.1 A prepared copper strip is totally immersed in a sample of grease and heated in an oven or liquid bath at a specified temperature for a definite period of time. Commonly used conditions are 100°C (212°F) for 24 h. At the end of this heating period, the strip is removed, washed, and compared with the ASTM Copper Strip Corrosion Standards.

#### 5. Significance and Use

5.1 This test method measures the tendency of lubricating grease to corrode copper under specific static conditions. It

<sup>2</sup> Annual Book of ASTM Standards, Vol 05.01.

may be of some value in predicting possible chemical attack on lubricated parts, such as bearings that contain copper or copper alloys. Such corrosion, for example, can cause premature bearing failures. However, no correlations with actual field service, most of which are under dynamic conditions, have been established. It does not measure either the ability of the lubricant to inhibit copper corrosion caused by factors other than the lubricant itself nor does it measure the stability of the grease in the presence of copper.

### 6. Apparatus

6.1 *Test Jars*—Cloud and pour jars, cylindrical jars of clear glass with flat bottoms, 30 mm to 33.5-mm ( $1\frac{3}{16}$  in. to  $1\frac{5}{16}$  in.) inside diameter, and 115 mm to 125 mm ( $4\frac{1}{2}$  in. to 5 in.) in height as described in Test Methods D 97 and D 2500.

6.2 Test Jar Covers—Beakers, 50-mL; borosilicate glass, 40 mm (1.6 in.) by 50 mm (2.0 in.) suitable for covering individual test jars. Small porcelain or glazed silica crucibles or crucible covers or watch glasses or vented corks that lightly cover the test jars, allowing pressure equalization between the inside and outside of the jars, yet minimizing exposure of the inside of the jars to foreign vapors present in the oven will also be satisfactory.

6.3 *Oven*—A circulating air oven or liquid bath capable of maintaining a temperature of  $100 \pm 1^{\circ}$ C (212  $\pm 2^{\circ}$ F) or other desired temperatures with the same precision.

6.4 *Polishing Vise*—For holding the copper strip firmly without marring the edges while polishing. Any convenient type of holder (see the Appendix of Test Method D 130) may be used, provided that the strip is held tightly and that the surface of the strip being polished is supported above the surface of the holder.

6.5 *Viewing Test Tubes*—Flat glass test tubes may be used to protect corroded strips for close inspection or storage. (See the Appendix of Test Method D 130.)

6.6 ASTM Copper Strip Corrosion Standards<sup>2,4</sup> consist of reproductions in color of typical strips representing degrees of tarnish and corrosion, the reproductions being encased in plastic in the form of a plaque.

6.6.1 Keep the plastic-encased printed ASTM Copper Strip Corrosion Standards protected from light to avoid the possibility of fading. Inspect for fading by comparing two different

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<sup>&</sup>lt;sup>3</sup> Available from ASTM Headquarters for a nominal charge. Request Adjunct PCN 12-401300-00.

<sup>&</sup>lt;sup>4</sup> Available from commercial sources.

plaques, one of which has been carefully protected from light (new). Observe both sets in diffused daylight (or equivalent), first from a point directly above and then from an angle of  $45^{\circ}$ . If any evidence of fading is observed, particularly at the left end of the plaque, it is suggested that the one that is the more faded with respect to the other be discarded.

6.6.1.1 Alternatively, place a 20-mm ( $\frac{3}{4}$  in.) opaque strip (masking tape) across the top of the colored portion of the plaque when initially purchased. At intervals, remove the opaque strip and observe. If there is any evidence of fading of the exposed portion, it is suggested that the standard be replaced.

6.6.1.2 These plaques are full-color productions of typical strips. They have been printed on aluminum sheets by a four-color process and are encased in plastic for protection. Directions for their use are given on the reverse side of each plaque.

6.6.2 If the surface of the plastic cover shows excessive scratching, it is suggested that the plaque be replaced.

## 7. Reagents and Materials

7.1 *Acetone*—American Chemical Society Reagent Grade acetone is suitable.

NOTE 1—Warning: Extremely flammable. Harmful if inhaled. Vapors may cause flash fires.

7.2 *Copper Strips*—Use strips 12.5 mm ( $\frac{1}{2}$  in.) wide, 1.5 mm to 3.0 mm ( $\frac{1}{16}$  in. to  $\frac{1}{8}$  in.) thick, cut 75 mm (3 in.) long from smooth-surfaced, hard-temper, cold-finished copper of 99.9 + % purity; electrical bus bar stock is generally suitable. (See the Appendix of Test Method D 130.) The strips may be used repeatedly but should be discarded when the surfaces become deformed on handling.

7.3 *Polishing Materials*—Silicon carbide grit paper of varying degrees of fineness, including 65- $\mu$ m (240-grit) paper or cloth, also a supply of 105- $\mu$ m (150-mesh) silicon carbide grain<sup>4</sup> and pharmaceutical-grade absorbent cotton (cotton wool).

7.4 Wash Solvent—Volatile, sulfur-free hydrocarbon solvent may be used provided that it shows no tarnish at all when tested at 50°C (or 122°F). Knock test-grade *is*ooctane (**Warning**—see Note 2) or American Chemical Society Reagent Grade *n*-heptane (**Warning**—see Note 3) are suitable solvents. Because of possible toxic effects, the use of benzene should be avoided.

## 8. Preparation of Strip

#### 8.1 Mechanical Cleaning of Strips:

8.1.1 *Surface Preparation*—Remove all surface blemishes from all six sides of the strip with silicon carbide paper of such degrees of fineness as are needed to accomplish the desired results efficiently. Finish with 65-μm (240-grit) silicon carbide paper or cloth, removing all marks that may have been made by other grades of paper used previously. Immerse the strip in wash solvent (**Warning**—see Note 1.) from which it can be withdrawn immediately for final polishing or in which it can be stored for future use.

8.1.1.1 As a practical manual procedure for surface preparation, place a sheet of the paper on a flat surface, moisten it

with wash solvent, and rub the strip against the paper with a rotary motion, protecting the strip from contact with the fingers with an ashless filter paper or by wearing disposable polyethylene gloves. Alternatively, the surface of the strip may be prepared by use of motor-driven machines using appropriate grades of dry paper or cloth.

8.1.2 Final Polishing-Remove strip from the wash solvent. Holding it in the fingers protected with the ashless filter paper, polish first the ends and then the sides with the 105-µm (150-mesh) silicon carbide grains<sup>5</sup> picked up from a clean glass plate with a pad of cotton (cotton wool) moistened with a drop of wash solvent. Wipe vigorously with fresh pads of cotton (cotton wool) and subsequently handle only when wearing disposable polyethylene gloves or with stainless steel forceps (see Note 2); do not touch with naked fingers. Clamp the strip in a vise, and polish the main surfaces with silicon-carbide grains on absorbent cotton. Rub in the direction of the long axis of the strip, carrying the stroke beyond the end of the strip before reversing the direction. Clean all metal dust from the strip before reversing the direction. Clean all metal dust from the strip by rubbing vigorously with clean pads of absorbent cotton until a fresh pad remains unsoiled. When the strip is clean, immediately immerse it in the prepared sample.

NOTE 2-Caution, forceps can scratch copper, affecting the results.

8.1.2.1 It is important to polish the whole surface of the strip uniformly to obtain a uniformly stained strip. If the edges show wear (surface elliptical), they will likely show more corrosion than the center. The use of a vise will facilitate uniform polishing.

## 9. Procedure

9.1 Place about 60 mL (about 50 to 70 g, depending on its specific gravity) of grease sample into the bottom of a clean test jar (see 6.1.). Bring the depth of the grease to about 80 mm (3.2 in.). Tap the grease down into the jar carefully. Smooth the top to give a relatively flat surface. Wipe excess grease off the jar walls.

9.2 To prevent touching the strip with fingers, wear disposable polyethylene gloves or use stainless steel forceps (see Note 2); and insert cleaned copper strip into the grease until it touches the bottom of the tube and is completely immersed and covered by at least 5 mm (0.2 in.) of grease.

9.3 Place the jar in a rack so it is supported approximately vertically and cover it loosely with a beaker, crucible, watch glass, or vented cork (see 6.2).

9.4 Place rack with the samples in an oven or liquid bath regulated to maintain a specified temperature for a specified period of time. Commonly used conditions are  $100 \pm 1^{\circ}$ C (212  $\pm 2^{\circ}$ F) for 24 h.

9.5 At the end of the test period, remove the rack of samples from the oven or liquid bath and allow them to cool to room temperature.

9.6 Strip Examination:

9.6.1 Wearing disposable polyethylene gloves or using stainless steel forceps (see Note 2), remove the strip from the

<sup>&</sup>lt;sup>5</sup> Supporting data are available and on loan from ASTM Headquarters. Request RR: D02-1187.