**Designation: D4048 – 16**

# Standard Test Method for Detection of Copper Corrosion from Lubricating Grease[1]

This standard is issued under the fixed designation D4048; 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 U.S. 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.

1.2.1 *Exception*—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 heal practices and determine the applicability of regulatory limitations prior to use.*For specific warning statements, see Sections 7, 8, and 10.

## 2. Referenced Documents

2.1 *ASTM Standards:*[2]

D97 Test Method for Pour Point of Petroleum Products

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

D2500 Test Method for Cloud Point of Petroleum Products and Liquid Fuels

D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants

2.2 *ASTM Adjuncts:*

Copper Strip Corrosion Standard[3]

## 3. Terminology

3.1 *Definitions*—See Terminology D4175.

3.2 *copper corrosion, n*—effect of a chemical attack on copper metal by a lubricant causing various levels of tarnishing and change in appearance.

3.2.1 *Discussion*—Acidic and other aggressive species, often sulfur-based, in a lubricant can attack copper or copper alloys present in bearings or other lubricated surfaces. The presence of this antagonistic interaction is often apparent in a well-defined series of color changes.

3.3 *lubricant, n*—any material interposed between two surfaces that reduces the friction or wear between them.

3.4 *lubricating grease, n*—a semi-fluid to solid product of a dispersion of a thickener in a liquid lubricant.

3.4.1 *Discussion*—The dispersion of the thickener forms a two-phase system and immobilizes the liquid lubricant by surface tension and other physical forces. Other ingredients are commonly included to impart special properties.

## 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 ± 1 °C (212 °F ± 2 °F) for 24 h ± 5 min. At the end of this heating period, the strip is removed, washed, and compared with the Copper Strip Corrosion Standard.

## 5. Significance and Use

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

NOTE 1—Because this test method requires the ability to determine subtle differences in color of copper strips, persons with certain types of color blindness may find it difficult to accurately compare a test strip to the Copper Strip Corrosion Standard.

## 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 3/16 in. to 1 5/16 in.)

[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.G0.01 on Chemical and General Laboratory Tests.

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[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.

[3] Available from ASTM International Headquarters. Order Adjunct No. ADJD0130. Originally produced in 1973.

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

inside diameter, and 115 mm to 125 mm (4½ in. to 5 in.) in height as described in Test Methods D97 and D2500.

6.2 *Test Jar Covers—Beakers,* 50 mL; borosilicate glass, approximately 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 °C ± 1 °C (212 °F ± 2 °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 Appendix X1 on Optional Useful Equipment in Test Method D130) 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 Appendix X1 on Optional Useful Equipment in Test Method D130.)

6.6 *Copper Strip Corrosion Standard*—consists 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 Copper Strip Corrosion Standard protected from light to avoid the possibility of fading. Inspect for fading by comparing two different 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 approximately 45°. 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 (¾ 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. (**Warning—**Extremely flammable. Harmful if inhaled. Vapors may cause flash fires.)

7.2 *Copper Strips*—Use strips 12.5 mm ± 2 mm (½ in. ± 1/16 in.) wide, 1.5 mm to 3.0 mm (1/16 in. to ⅛ in.) thick, cut 75 mm ± 5 mm (3 in. ± ¼ in.) long from smooth-surfaced, hard-temper, cold-finished copper of 99.9 + % purity; electrical bus bar stock is generally suitable. (See Appendix X1 on Optional Useful Equipment in Test Method D130.) 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 µm (220 grit CAMI-grade or P220 FEPA-grade) paper or cloth, also a supply of 105 µm (120 grit to 150 grit CAMI-grade or P120 to P150 FEPA-grade) silicon carbide grain and 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 ± 1 °C (or 122 °F ± 2 °F). Knock test-grade *iso*octane (**Warning—**see 8.1.2) or American Chemical Society Reagent Grade *n*-heptane (**Warning—**see 7.1) 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 7.1.) 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 or by wearing impervious gloves (see Note 2). Alternatively, the surface of the strip may be prepared by use of motor-driven machines using appropriate grades of dry paper or cloth.

NOTE 2—Disposable polyethylene is one type of glove that has been found to be satisfactory. However, any type of glove that is impervious to the reagents and materials used in this test method, while preventing fingers from contacting the strip directly, may also be used.

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 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 impervious gloves (see Note 2) or with stainless steel forceps; *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. (**Warning—**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 g 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.

NOTE 3—The use of a syringe to prepare a grease sample with minimal entrained air and then to transfer it to the test jar has been found to be helpful in reducing the amount of air trapped in the sample during filling. A syringe with a wide nozzle and a diameter just less than that of the test jar may be most helpful in this regard. This may reduce the potential risk of breaking the somewhat fragile test jar during the action of tapping it to remove air from the grease sample.

9.2 To prevent touching the strip with fingers, wear impervious gloves (see Note 2) or use stainless steel forceps; 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 Cover the jar(s) loosely with a beaker, crucible, watch glass, or vented cork (see 6.2).

9.4 Place the jar(s) in an oven or liquid bath so that they are supported approximately vertical. Regulate the oven or liquid bath to maintain a specified temperature for a specified period of time. Commonly used conditions are 100 °C ± 1 °C (212 °F ± 2 °F) for 24 h ± 5 min.

9.5 At the end of the test period, remove the jar(s) from the oven or liquid bath and allow them to cool to room temperature.

9.6 *Strip Examination:*

9.6.1 Wearing impervious gloves (see Note 2), or using stainless steel forceps, remove the strip from the grease, and carefully remove excess grease clinging to the strip. Take care not to disturb the tarnish on the strip, and immediately immerse it in wash solvent. Withdraw the strip at once, dry with quantitative filter paper by blotting and not by wiping, and inspect, in diffused daylight (or equivalent), for evidence of tarnishing or corrosion by comparison with the Copper Strip Corrosion Standard. Hold both the test strip and the standard strip plaque in such a manner that light reflected from them at an angle of approximately 45° will be observed.

NOTE 4—It has been suggested that a very light grease may be blotted off the strip with filter paper or a laboratory wiper. Heavier greases may require wiping or removal with a plastic, rubber, or stainless steel spatula. However the grease is removed from the strip, exercise care not to disturb the tarnish or corrosion on the strip.

9.6.2 In handling the test strip during the inspection and comparison, the danger of marking or staining can be avoided if it is inserted in a flat tube (see Appendix X1 on Optional Useful Equipment in Test Method D130), which can be stoppered with absorbent cotton.

## 10. Interpretation

10.1 Interpret the corrosiveness of the sample according to how the appearance of the test strip agrees with one of the strips of the Copper Strip Corrosion Standard.

10.1.1 When a strip is in the obvious transition state between that indicated by any two adjacent standard strips, judge the sample by the more tarnished standard strip. Should a strip appear to have a darker orange color than standard strip 1b, consider the observed strip as still belonging in this Classification 1b; however, if any evidence of red color is noticed, the observed strip belongs in Classification 2.

10.1.2 A claret red 2a strip can be mistaken for a 3a strip, if the brassy underlay of the 3a strip is completely masked by a magenta overtone. To distinguish between them, immerse the strip in acetone (**Warning—**see 7.1). A 2a strip will appear as a 1b strip, while a 3a strip will not change.

10.1.3 To distinguish a 2c strip from a 3b strip, place it in a 20 mm by 150 mm test tube, and bring it to a temperature of 315 °C to 370 °C (600 °F to 700 °F) in 4 min to 6 min using a hot plate. Adjust the temperature by observing an ASTM high temperature distillation thermometer inserted in a second test tube lying on the hot plate. A 2c strip will assume the color of a 2d strip and successive stages of tarnish, while a 3b strip will take on the appearance of a 4a strip.

10.1.4 Repeat the test if blemishes due to fingerprints or due to spots from any water droplets or entrained air that may have touched the test strip during the test period or from other unknown causes are observed. If blemishes on the strip are still

**TABLE 1 Copper Strip Classifications**

| Classification | Designation | Description[A] |
|---|---|---|
| Freshly Polished Strip[B] | | |
| 1 | Slight tarnish | a. Light orange, almost the same as a freshly polished strip |
| | . . . | b. Dark orange |
| 2 | Moderate tarnish | a. Claret red |
| | . . . | b. Lavender |
| | . . . | c. Multicolored with lavender, blue or silver, or both, overlaid on claret red |
| | . . . | d. Silvery |
| | . . . | e. Brassy or gold |
| 3 | Dark tarnish | a. Magenta overcase on brassy strip |
| | . . . | b. Multicolored with red and green showing (peacock) but no gray |
| 4 | Corrosion | a. Transparent black, dark gray, or brown with peacock green barely showing |
| | . . . | b. Graphite or lusterless black |
| | . . . | c. Glossy or jet black |

[A] The Copper Strip Corrosion Standard is a colored reproduction of strips characteristic of these descriptions.

[B] The freshly polished strip is included in the series only as an indication of the appearance of a properly polished strip before a test run; it is not possible to duplicate this appearance after a test, even with a completely noncorrosive sample.

observed, the rating for the more tarnished portion of the strip should be applied to the whole strip.

10.1.5 Repeat the test also if the sharp edges along the flat faces of the strip appear to be in a classification higher than the greater portion of the strip; in this case, it is likely that the edges were burnished during polishing.

## 11. Report

11.1 Report the corrosiveness in accordance with one of the classifications and descriptions in Table 1. State the duration of the test and the test temperature.

## 12. Precision and Bias[4]

12.1 Due to the nature of the results, the precision of this test method was not obtained in accordance with RR:D02-1007.

12.1.1 *Repeatability*—can be judged by the fact that 96 % of duplicate results obtained in the normal simultaneous procedure by twelve laboratories with six samples were in agreement (see Note 5).

12.1.2 *Reproducibility*—may be judged by the fact that twelve laboratories matched consensus within a numerical classification (1, 2, 3, 4) 92 % of the time and within a numerical and descriptive classification (a, b, c, d) 69 % of the time with five samples showing good or poor protection against copper corrosion. A sample (Grease F) with somewhat marginal corrosion protection properties found agreement between laboratories only 50 % of the time by either numerical or numerical and descriptive classification.

NOTE 5—Actual data on which this precision statement is based can be found in Appendix X1. Test conditions were 100 °C, 24 h.

12.2 *Bias*—The procedure in Test Method D4048 has no bias because the rating of copper corrosion is defined only in terms of this test method.

## 13. Keywords

13.1 copper corrosion; copper strip; copper tarnish; lubricating grease

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1187.

# APPENDIX

**(Nonmandatory Information)**

## X1. PRECISION STATEMENT DATA

| Grease Run No. | A | | B | | C | | D | | E | | F | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 1 | 2 | 1 | 2 | 1 | 2 | 1 | 2 | 1 | 2 | 1 | 2 |
| 1 | 2c | 2c | 1a | 1a | 1b | 1b | 1a | 1a | 4a | 4a | 2c | 2c |
| 2 | 1a | 1a | 1a | 1a | 1b | 1b | 1a | 1a | 4a | 4a | 1b | 1b |
| 3 | 1a | 1a | 1a | 1a | 1b | 1a | 1a | 1a | 4a | 4a | 1b | 1b |
| 4 | 1a | 1a | 1b | 1b | 1b | 1b | 1b | 1b | 3b | 3b | 2c | 2c |
| 5 | 1a | 1a | 1a | 2 | 2 | 2 | 2 | 2 | 4 | 4 | 3 | 3 |
| 6 | 1a | 1a | 1a | 1a | 1a | 1a | 1a | 1a | 4b | 4b | 4a | 4a |
| 7 | 1a | 1a | 1b | 1b | 1b | 1b | 1a | 1a | 4a | 4a | 3a | 3b |
| 8 | 1a | 1a | 1a | 1a | 1a | 1a | 1a | 1a | 4a | 4a | 1b | 1b |
| 9 | 1a | 1a | 1a | 1a | 1a | 1a | 1a | 1a | 4a | 4a | 1b | 1b |
| 10 | 1a | 1a | 1a | 1a | 1a | 1a | 1a | 1a | 4b | 4b | 1b | 1b |
| 11 | 2c | 1b | 1a | 1b | 1b | 1b | 1a | 1a | 4a | 4a | 2c | 2c |
| 12 | 1a | 1a | 1a | 1a | 1a | 1a | 1a | 1a | 4b | 4b | 1b | 1b |

## SUMMARY OF CHANGES

Subcommittee D02.G0 has identified the location of selected changes to this standard since the last issue (D4048 – 10) that may impact the use of this standard. (Approved Dec. 1, 2016.)

*(1)* Revised subsection 7.3.

*(2)* Added new Note 3.