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Standard Test Method for Rust Protection by Metal Preservatives in the Humidity Cabinet¹

This standard is issued under the fixed designation D 1748; 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 test method has been adopted for use by government agencies to replace Method 5310 of Federal Test Method Standard No 791 b.

 ϵ^1 Note—Keywords were added in May 1993.

1. Scope

1.1 This test method is used for evaluating the rustpreventive properties of metal preservatives under conditions of high humidity.

1.2 The values stated in SI units are to be regarded as the standard.

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 consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

A 109 Specification for Steel, Strip, Carbon, Cold-Rolled²

D 91 Test Method for Precipitation Number of Lubricating Oils³

D 512 Test Methods for Chloride Ion in Water⁴ ist/a [Sel260

D 516 Test Methods for Sulfate Ion in Water⁴

2.2 Federal Specifications:⁵

RRS-366 (Method 5329 of VV-L-791e) Test Sieve Sizes

QQ-S-698 Steel Sheet and Strip, Low Carbon

PD-680 Standard Solvent

JAN-H-792 Operations of Humidity Cabinet

2.3 *Military Specifications:*⁵

MIL-C-16173D Corrosion Preventive Compound, Solvent Compound Cutback, Cold Application⁶

³ Annual Book of ASTM Standards, Vol 05.01.

MIL-C-15074C Corrosive Preventive Compound Finger Print Remover

2.4 Society of Automotive Engineers:⁷

1009C Tee Reducer, Bulkhead on Side, Flareless Tube

3. Summary of Test Method

3.1 Steel panels are prepared to a prescribed surface finish, dipped in the test oil, allowed to drain and then suspended in a humidity cabinet at 48.9 \pm °C (120°F) for a specified number of hours. The oil fails or passes the test according to the size and number of rust dots on the test surfaces of the panels.

4. Significance and Use

4.1 This test method is used for measuring the relative abilities of metal preservatives to prevent the rusting of steel panels under conditions of high humidity. It should not be relied upon to predict the effectiveness of a metal preservative in which high humidity is not the principal factor in the rusting.

4.2 There are published data indicating a useful degree of correlation with service performance, though these data are not extensive. Comparisons made by this method should normally be limited to similar metal preservative combinations designed for similar applications. The test life required for each type of metal preservative and for each intended application should be based on actual experience with that type of preservative in the intended service.

4.3 Since the precision of the method appears to be less than desired, a number of repeat tests may be necessary to establish the test life of a given metal preservative, and repeat tests by this method in more than one cabinet are sometimes desirable.

4.4 The data obtained from this accelerated test is of interest only in eliminating the most unsuitable materials or for indicating a probable relative order of protection against rust under conditions of high humidity. This method does not prescribe the exposure periods to be used for a specific product, nor the interpretation to be given to the results.

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 D 02.L0.03 on Corrosion Testing of Sheet Metal Processing Fluids.

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² Annual Book of ASTM Standards, Vol 01.03.

⁴ Annual Book of ASTM Standards, Vol 11.01.

⁵ Available from Standardization Documents Order Desk, Bldg. 4, Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

 $^{^{\}rm 6}$ Available from British Standards Institute, 2 Park St., London, England W1A2B5.

⁷ Available from Society of Automotive Engineers, 400 Commonwealth Dr., Warrendale, PA 15096.

5. Apparatus

5.1 The apparatus shall conform to the details shown in the Annex A1 and shall meet the requirements of Military Specification JAN-H-792.

6. Panel Cleaning Materials

6.1 Aluminum Oxide Cloth, 240-grit.

NOTE 1—Paper-backed abrasives, wet or dry, waterproof, or iron oxide abrasives are prohibited.

6.2 *Silica Sand*, white, dry, sharp, chloride free, or aluminum oxide, blasting grade. (The size shall be such that it meets the following sieve requirements to Federal Specification RR-S-366) (Method 5329 of VV-L-791e):

6.2.1 One hundred percent must pass through a No. 10 (2.00-mm) sieve.

6.2.2 Minimum of 90 % must pass through a No. 20 (850-μm) sieve.

6.2.3 Maximum of 10 % permitted to pass through a No. 50 (300- μ m) sieve.

6.3 *Petroleum Naphtha*, conforming to Federal Specification PD 668. Type 1 (37.8°C (100°F) min flash). (**Caution**— Combustible. Vapor harmful. See Annex A2.1. **Danger**— Extremely flammable, harmful if inhaled. See Annex A2.2.)

6.4 *Precipitation Naphtha*, conforming to the requirements in Section 4 of Test Method D 91.

6.5 *Methanol (Methyl Alcohol)*, absolute, ACS reagent grade. (**Danger**—Flammable. Vapor harmful. May be fatal or cause blindness if swallowed or inhaled, cannot be made nonpoisonous. See Annex A2.3.)

7. Humidity Cabinet Operating Conditions

7.1 During evaluation of a sample the cabinet shall be run continuously with the following standard conditions being maintained:

Air temperature:

48.9 ± 1.1°C (120 ± 2°F)
24.1 ± 5.5°C (75 ± 10°F)
(31 ± 1 ft ³ /h) at 25°C and 760
mm Hg
203 \pm 6.4 mm (8 \pm ¼ in.)
5.5 to 7.5
clear with no evidence of oil
less than 20 ppm (Test Method D 512)
less than 20 ppm as sulfate (Test
Method D 516)

^ABoil the water sample with 10 mL of saturated bromine water before making the test for sulfates.

Speed of rotating	$0.33 \pm 0.03 \text{ rpm}$
Cover	close fitting
Cloth layers in cover	shall not be torn, contaminated, nor contain droplets of water
Cover opening	to a height of 355 mm (14 in.) at the front

7.2 Rate of air to the cabinet, air temperature, pH, and water level shall be checked and regulated if necessary in the morning and afternoon of each day. Remaining standard conditions shall be closely checked once each week. The pH measurement may be made with wide-range indicator paper.

NOTE 2-Values for pH outside the limits shown indicate contamina-

tion which should be investigated and corrected. A persistent low pH along with a positive sulfate test indicates that the air supply is contaminated with sulfur oxides. In this case, the water in the cabinet should be replaced, and a suitable alkali scrubber system installed in the air train.

NOTE 3—Details of the cabinet operation described in 7.1 and 7.2 and the details of panel preparation described in Section 8, must be carefully carried out. Only by such standardization can results be obtained that are significant and comparable to those run at another time or in another laboratory.

8. Panel Preparation

8.1 Remove the protective packaging from all the panels to be used for a particular day and wash away the rust preventive material in a beaker of petroleum naphtha (swabbing is permissible). Carefully inspect each panel and use only those which comply with requirements given in Annex A1.10. Identify each panel by an appropriate number in the right-hand lower corner, outside of the significant area, or by attaching a small metal tag to the outside wire hook after the panel is polished.

8.2 The following are pertinent to the polishing operations:

8.2.1 Do not allow the bare fingers to touch the panel. Tongs, metal hooks, or pieces of lint-free paper are suitable helps for manipulating and holding the panel.

8.2.2 Always keep the panel on a clean, dry surface.

8.3 Alternative Surface Finishes—Polishing:

8.3.1 The amount of polishing of the panel by the operator conducting the humidity cabinet test should only be that required to give it a fresh, clean, and active surface. This requires only a few minutes per panel. A fast-moving belt sander should not be used since the heat of friction may change the surface characteristics of the panel. Surface finish limits for the panel are not defined here. There should be no appreciable change of the finish from the 0.25 to .51 μ m (10 to 20 μ m) obtained by the original surface grinding A1.10.1.3.

8.3.2 Divide the 240-grit aluminum oxide abrasive cloth into convenient size strips for the subsequent polishing operations. Observing the precautions given in 8.2 and 8.3, buff all four of the rounded edges with even strokes in the direction of each edge. Ream out the two holes used for suspension and wipe clean, using gauze wet with petroleum naphtha.

8.3.3 While polishing, place the panel on a clean, dry surface with a suitable thickness of clean paper under it to help prevent contamination. The panel may be held by hand using paper between the fingers and the steel surface. Alternatively it may be held in a special holder such as a wooden block having about a 1.6-mm ($\frac{1}{16}$ -in.) depression slightly larger in area than the 51- by 102-mm (2- by 4-in.) dimension of the panel. The abrasive cloth may be held in the palm of the hand with the fingers applying pressure to the panel. Alternatively, the aluminum oxide cloth may be held on a block of size convenient to the hand and convenient for polishing with smooth strokes without marking the ends of the panel.

8.3.4 Polish the unnumbered or leading surface of the panel with careful even strokes, always parallel to the 102-mm (4-in.) dimension. Use a polishing pressure of about 4.5 to 8.9 N (1 to 2 lb). Do not scratch the surface by using short or curved strokes. Ensure that the panel is held firmly so that only the abrasive cloth moves. After several polishing strokes, inspect

the abrasive cloth and when necessary make another fold to expose a new and effective area. Continue to polish the leading surface until it contains a completely fresh surface. The finish should be within the range from 0.25 to 0.51 μ m (10 to 20 μ in) (rms). Examine the entire surface and if scratches or other imperfections are noted, continue polishing until corrected. In the same manner polish the second side of the panel, unless the test specification requires only one polished side per panel.

NOTE 4—It is advisable for each operator to finish several panels to determine exactly what technique is required to attain the correct surface finish using a profilometer or other surface roughness gage. After techniques have been established, the use of visual comparison standards is sufficient as a check on surface roughness.

8.3.5 Remove the dust from the abrasive operation using clean gauze wet with petroleum naphtha. (**Caution**—Combustible. See Annex A2.1). Finally, wipe with clean surgical gauze until there is no dark stain on a clean section of the gauze. Remove any dust in the holes by use of a pipe cleaner. (This may be followed by an ultrasonic cleaning bath procedure.) Submerge the panel completely in absolute methanol at room temperature. (**Danger**—Flammable. Vapor harmful. See Annex A2.3).

NOTE 5—The following should be carried out periodically as a check on surface cleanliness: Place the cleaned panel directly under a buret on a table free of vibrations and drafts. Place the buret so its tip is exactly 300 mm above the panel. The buret contains distilled water and shall have a tip of proper dimensions to deliver 0.05 ± 0.01 mL of distilled water per drop. Allow one drop of distilled water to fall onto the panel surface. If the surface is absolutely clean, successive droplets on various parts of the surface will spread out completely in spots of closely reproducible dimensions. A clean panel should give a spread of 21 to 23 mm for each 0.05 mL of distilled water. This test is considered necessary and important because of variations found in different abrasive materials and the personal factors involved in the procedure require some method of check on final results. Panels used for this cleanliness check test are not suitable for use in the protection test.

8.3.6 To minimize differences in activity of the steel surfaces as a result of time in various air atmospheres, cleaning and polishing of the panels should be standardized in respect to time. For this reason carry out the procedure described in 8.3.2-8.3.5 one panel at a time, and after each one is prepared, store it immediately in absolute methanol (**Danger**— Flammable. Vapor harmful. See Annex A2.3) at room temperature until all the panels for one day's operations are prepared.

8.4 Alternative Surface Finishes—Sand or Aluminum Oxide Blasting:⁸

8.4.1 Blast the edges and lightly blast the backs of the panels with the blasting material.

8.4.2 Blast the unnumbered side, or test surface, of the panels to a fresh, uniformly abraded surface. (Operation 177.9–355.8 N of the blasting equipment at (40- to 80-lb.) pressure and holding the workpiece 50.8 to 76.2 mm (2 to 3 in.) from the nozzle is recommended.)

8.4.3 Immediately after blasting, place the panels in a beaker of anhydrous methanol or an ultrasonic cleaning bath containing anhydrous methanol.

8.4.4 Heat the methanol so that the solvent will evaporate from the panels immediately upon withdrawal from the solvent.

8.4.5 Remove remaining residue by holding the panels in a rack at 20° from the vertical and spraying downward with naphtha.

8.4.6 Spray the test surface, then the back of the panel, and the test surface again.

8.4.7 Rinse the panels in hot naphtha and hot methanol and store in a desiccator until cool.

8.4.8 Panels are to be used the same day as prepared.

9. Procedure

9.1 Bring the sample oil to a temperature of $23.3 \pm 0.5^{\circ}$ C ($74 \pm 1^{\circ}$ F) and pour into a clean, dry 400-mL tall-form glass beaker⁹ to a height of at least 114 mm (about 375 mL). By use of one clean suspension hook remove a panel from the methanol and hang it in the vapor space above boiling ASTM precipitation naphtha (**Danger**—Flammable. See Annex A2.1) for 5 min, ensuring that the panel is completely wet with the refluxing solvent.

NOTE 6—Cleaning the panel with naphtha vapors is conveniently done using approximately 100 mL of naphtha in a 400- to 600-mL tall-form beaker. Perform this operation in a well-ventilated hood and make sure there are no sources of ignition in the area. Heat-resistant glass beakers have been used for this purpose, but use of a metal beaker is preferred from the standpoint of possible breakage.

9.2 Then slush the panel for 10 s in a beaker of boiling absolute methanol. (Danger—Flammable. Vapor harmful. See Annex A2.3). Withdraw it from the methanol and observe for any stains on the surfaces, with attention to any contamination from the holes. If stains are present, repeat the panel preparation beginning at 8.3.2. After 10 to 20 s in the air, place the clean panel in the sample oil and agitate for 10 s while submerged in the oil. Withdraw the panel with a continuous motion, drain for 10 s, and replace in the sample oil for 1 min with slight agitation. Remove from the test oil with a continuous motion, taking from 2 to 4 s. Handle the panel carefully and do not jar nor shake it. Dip the end of a second clean suspension hook into the sample oil and insert it into the second hole of the panel. Drain the panel suspended by two hooks in the box described in the Annex A1 (Fig. A1.8), at a temperature of 24.1 \pm 3°C (75 \pm 5°F) for 2 h \pm 20 min, unless another draining time is specified (Note 7). More than one panel may be dipped in one beaker of the preservative oil, provided its temperature does not rise above 26.7°C (80°F). If this occurs, then dip subsequent panels in a second beaker of the test oil at 23.3 \pm 0.5°C (74 \pm 1°F).

Note 7—Sample holders, rates of removal of panel from the test sample, and draining time for some preservatives may differ from those given above—according to particular specifications. For example, for some of the more highly compounded preservatives that have been cut back with volatile solvents, the panel is removed using two hooks at the rate of 102 mm (4 in.)/min, and a 24 ± 1 h draining period is used.

9.3 At the end of the draining period, suspend the panels in the humidity cabinet described in Annex A1, with the "back,"

⁸ Blasting equipment can be obtained from Pressure Blast Manufacturing Co., Inc., 41 Chapel St., Manchester, CT USA 06040.

⁹ Borosilicate glass has been found satisfactory for this purpose.

that is, the numbered side of the panel, trailing as the stage rotates. Allow the one or more test panels treated with the sample oil to remain in the humidity cabinet for the number of hours specified. Maintain operating conditions as specified in Section 7. Check and adjust the air temperatures, pH, air rate, and water level twice each day, at 7- to 8-h intervals.

9.4 Open the humidity cabinet twice each day, except Saturday and Sunday, as follows: (1) For a 15-min period, and (2) after an interval of at least 3 h (preferably 6 to 8 h to permit checking of operating conditions during these openings) from the first opening, for a 5-min period. Generally, it will be found convenient to do the inspection of panels during the 15-min opening, and the insertion of new panels during the 5-min opening. To standardize the effect of panel cooling and other variables, keep the cabinet open for the entire 15- and 5-min periods even though the time required to inspect or install panels may be less. The front edge of the cover should be propped open to a distance of 356 mm (14 in.) from the top of the cabinet.

9.5 Panels being evaluated against specified times in the cabinet should not be withdrawn, except at the end of the required time. Panels used in hours-to-failure evaluations, such as in developmental studies, should be very carefully withdrawn and inspected one at a time: a panel should not leave the cabinet, except for the actual time each day required for its inspection.

10. Examination

10.1 Remove the panels at the completion of the specified time in the cabinet. Wash them with petroleum naphtha and within 10 min examine each one under a fluorescent light for pass or failure as follows:

10.2 Consider the significant area, as indicated in the Annex A1 (Fig. A1.7) of each side of each panel as a separate test surface; each panel thus represents two test surfaces. Rate each test surface as follows:

Pass: A test surface shall pass if it contains no more than three dots of rust, no one of which is larger than 1 mm in diameter.

Fail: A test surface shall fail if it contains one or more dots of rust larger than 1 mm in diameter or if it contains four or more dots of any size.

10.3 Alternately, the panels may be rated for pass or failure in accordance with the criteria stated in the specification or requirement for which the method is being used.

11. Report

- 11.1 The report shall include the following:
- 11.1.1 Hours in the humidity cabinet,
- 11.1.2 Number of test surfaces (or panels),
- 11.1.3 Number of passing test surfaces (or panels), and
- 11.1.4 Type of panel preparation (sandblasted or polished).

NOTE 8—This method is intended as a detailed standardized procedure for running the humidity test. Whether a metal preservative passes or fails the test depends upon the criteria given in the specification or requirement for which the method is being used.

12. Precision¹⁰ (See 4.4)

12.1 This procedure is believed to represent the best available practice. Operational procedures that might affect precision have been defined as closely as appears practicable.

12.2 Table 1 summarizes the effect of panel preparation on the repeatability and reproducibility of ASTM Test Method D 1748 (95 % confidence level).

13. Keywords

13.1 humidity; humidity cabinet; metal preservatives; rust protection

¹⁰ Supporting data have been filed and may be obtained from ASTM Headquarters by RR: D02 - 1136.

TABLE 1 Panel Preparation

- <u>8-83(1993)e1</u> 0c9-4326-9d78-a	Sandblasted,	A1 ₂ O ₃ Blasted, %	Polished, 748-8%1993e
Repeatability	27.8	46.1	97.3
Reproducibility	141.0	71.2	264.0
A			

^AThe sandblasted panels used in obtaining the precision data all came from one source.

ANNEXES

(Mandatory Information)

A1. HUMIDITY CABINET APPARATUS

A1.1 Location

A1.1.1 The location of the humidity cabinet shall provide for continuous controlled operation during the test. The humidity cabinet shall be in a room maintained at a temperature between 24.1 ± 5.5 °C (75 ± 10 °F). The room shall be free of strong air drafts and of exposure to any acid fumes or gases known to promote corrosion, such as sulfur dioxide, hydrogen sulfide, ammonia, and so forth.

A1.2 Humidity Cabinet¹¹

A1.2.1 The equipment used in this test method is the humidity cabinet conforming to Military Specification JAN-H-792. The general arrangement of the cabinet components is shown in Fig. A1.1. It consists of a metal-lined wooden cabinet, with the open top equipped with a hinged lid consisting of two thicknesses of desized airplane cloth. The cabinet holds approximately 94 L (25 gal) of distilled water up to the

¹¹ The Humidity Cabinet can be obtained from Koehler Instrument Co., Inc., 1595 Sycamore Ave., Bohemia, L.I., NY 11716.