Designation: D 1280 – 00

Standard Guide for
Total Immersion Corrosion Test for Soak Tank Metal Cleaners[1]

1. Scope

1.1 This guide covers determination of the corrosive effects of soak tank metal cleaners on all metals other than aluminum and its alloys, under conditions of total immersion, by quantitative measurement of weight change or by qualitative visual determination of change. The test determines the effects of the cleaner on metals being cleaned, and does not determine the life of the cleaner or of the containing equipment.

1.2 When the test is used to assist in the choice of material for a specific use, the test conditions should simulate the conditions of use as closely as practicable.

1.3 Where no further processing subsequent to cleaning is indicated, a test for the effect of residual cleaner on the corrosion behavior of the material may be required.

1.4 *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.* Material Safety Data Sheets are available for reagents and materials. Review them for hazards prior to usage.

2. Referenced Documents

2.1 *ASTM Standards:*

D 930 Test Method for Total Immersion Corrosion Test of Water-Soluble Aluminum Cleaners[2]

3. Significance and Use

3.1 This guide is used to distinguish soak tank metal cleaners that are excessively corrosive to metals (other than aluminum and its alloys which are covered by Method D 930). The users will define the degree of corrosion considered excessive, in terms of weight loss per unit of surface area or in terms of changes in appearance, or both.

4. Apparatus

4.1 *Test Tubes*, of a suitable material 38 mm in diameter by 300 mm in length, having a capacity of about 300 mL. The tubes shall be chosen so that the specimens will remain fully immersed during the test, and the ratio of area of immersed metal to the volume of solution will be prescribed in 7.1, with sufficient space provided for foam formation.

4.2 *Condensers*, Allihn-type, having jackets 200 to 250 mm in length, except that condensers will not be required for tests run at room temperature.

4.3 *Stoppers or Joints*—The connection between test tube and condenser optionally may be either standard-taper joints or rubber stoppers preboiled in aqueous caustic soda to remove free sulfur, and boiled in a sufficient number of changes of distilled water until neutral.

4.4 *Constant-Temperature Device*—Any suitable means may be employed for maintaining the solutions actively at the boiling point for tests conducted at that temperature. For tests made at other temperatures, a suitable constant-temperature bath shall be provided to maintain the required temperature within ±2°F (1°C). For control testing at room temperature, it is suggested that the solution be maintained at 95 ± 2°F (35 ± 1°C), which, being slightly above most room temperatures, is easy to maintain by heat input.

4.5 *Coating Apparatus*, shall be provided suitable for producing a uniform film of cleaner on the surface of the test specimen, for testing the effects of residual cleaner on the surfaces of cleaned metals (Section 10).

4.6 *Humidity Chamber*, for exposing the contaminated specimens at 90 % relative humidity as specified in 10.1.3.

5. Test Specimens

5.1 The test specimens shall be identical in composition, metallurgy, and surface finishing, with the conditions of the metal at the stage where cleaning will be applied in practice. The specimens shall have an area between 0.300 and 0.375 dm^2, and a length not to exceed 200 mm. (A specimen 18 by 85 by 1 mm in thickness would have an area of 0.327 dm^2.) At least two, and preferably four, replicates shall be tested in each concentration of cleaner solution prescribed in 7.2. The number of replicates under test shall be recorded.

6. Precleaning Test Specimens

6.1 Make ready the following pre-cleaning materials for removal of soil present on the metal surface prior to corrosion testing:

6.1.1 Place 200 mL of trichloroethylene in a 250-mL beaker

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[2] *Annual Book of ASTM Standards*, Vol 15.04.

and maintain at room temperature.

6.1.2 Prepare a vapor degreasing bath by heating 150 to 200 mL of trichloroethylene in a 1-L beaker, the degree of vaporization being controlled to permit vapor degreasing of the specimens when held in the beaker; or, alternatively, fill a wash bottle containing either of these solvents at room temperature.

6.1.3 Maintain a 250-mL beaker of either anhydrous methanol or isopropanol at 122°F (50°C).

6.1.4 Maintain a 250-mL beaker of distilled water at 122°F (50°C).

6.1.5 Maintain a 250-mL beaker of redistilled acetone at room temperature.

6.1.6 Place a small swab of fresh absorbent cotton in each of the beakers (6.1.1-6.1.5).

6.2 Immediately prior to their use in the test, clean the test specimens as follows:

6.2.1 Immerse the test specimens in the beaker of trichloroethylene (6.1.1) and immediately swab the surfaces of the individual specimens thoroughly, using clean forceps to hold both the cotton swab and the test specimen.

6.2.2 Shake off excess solvent and either transfer the specimen to the vapor degreasing bath (6.1.2) long enough to observe the vapor completely covering and condensing on the specimen, or thoroughly wash the specimen with a stream of fresh solvent from the wash bottle.

6.2.3 Swab the specimens separately in the beaker of alcohol (6.1.3) and shake free from excess alcohol.

6.2.4 Transfer the specimens to the beaker of distilled water (6.1.4). Swab carefully and shake free from excess water.

6.2.5 Immerse the specimens separately several times in the beaker of acetone (6.1.5), and shake free from acetone.

6.2.6 If the specimens are to be weighed, dry in a vacuum desiccator or in a low-temperature oven at 100°F (37.7°C).

7. Test Conditions

7.1 *Ratio of Area of Immersed Metal to Volume of Solution*—The ratio of the area of the immersed metal to the volume of solution shall be less than 1.5 dm^2/L of solution. (A specimen 18 by 85 by 1 mm in thickness would require 220 mL of solution, and a specimen having an area of 0.375 dm^2 would require 250 mL of solution.) Use fresh solution for each set of replicates and record the ratio used.

7.2 *Solution Concentration*:

7.2.1 In the absence of the manufacturer's recommendations, test the specimens in solutions of the cleaner of concentrations of 0.25, 0.5, 1.0, 4.0, and 8.0 weight % made up from stock solutions which shall be freshly prepared in 2000-g quantities. Boil the solutions for 30 min unless otherwise specified. In case the cleaner is not soluble to the extent noted in the stock solutions indicated, record this fact, but nevertheless continue the test with the specified total amounts of cleaner present in the test tubes. A blank test of either two or four replicates shall be made in freshly boiled distilled water.

7.2.2 When the manufacturer's recommendations are available, make the test at the following relative concentrations (based on the average concentrations suggested), recording the percentages these represent: (*1*) one half the concentration recommended, (*2*) the concentration recommended, and (*3*) twice the concentration recommended. Make a blank test of either two or four replicates in freshly boiled distilled water, submitting them otherwise to exactly the same procedure followed for the specimens immersed in cleaner solution.

7.3 *Water*—The water used in preparing the water-soluble cleaner solutions shall be freshly boiled distilled water.

7.4 *Temperature*— In the absence of manufacturer's recommendations, make the test at the boiling point of the solution. With available recommendations, make the test at 20 ± 2°F (11 ± 1°C) below and 20 ± 2°F (11 ± 1°C) above the average recommended temperature, or at the boiling point of the solution if it is lower. Record the temperature of the test in any case.

8. Procedure for Quantitative Weight Loss Test

8.1 Weigh the cleaned dry test specimens to the nearest 0.001 g.

8.2 Transfer the specimens individually to test tubes containing the preheated cleaner solutions, using only one specimen per tube. Attach the tubes to reflux condensers and maintain at the required temperature, adding a small piece of unglazed porcelain to prevent bumping. Expose for a period of 2 h. Other exposure periods may also be observed where appropriate.

8.3 Remove the strips at the end of the exposure period and proceed as follows:

8.3.1 Hold the specimen in forceps and rinse thoroughly in a 1-L beaker into which tap water is flowing rapidly.

8.3.2 Rinse thoroughly in distilled water at room temperature.

8.3.3 Rinse with a stream of acetone from a wash bottle. Shake free from acetone and dry.

8.3.4 Examine for, and record any of the following visible changes in comparison with the original uncleaned specimen: (*1*) discoloration, (*2*) dulling, (*3*) etching, (*4*) presence of accretions and relative amounts and areas, (*5*) types of pitting—wide, medium, or narrow, and (*6*) presence of selective or localized attack.

8.3.5 Remove corrosion products. (Methods of removal of corrosion products from different metals are given in Appendix X1 and Appendix X2.)

8.3.6 Rinse and dry as indicated in 8.3.2 and 8.3.3.

8.3.7 Weigh to the nearest 0.001 g.

9. Procedure for Qualitative Surface Corrosion Test

9.1 Proceed as for the quantitative weight loss test, except omit drying and weighing the specimens before the test, and dispense with the removal of corrosion products unless the corrosion is severe enough to obscure observation.

10. Procedure for Residual-Cleaner Corrosion Test

10.1 Where it is desired to determine the effect of residual cleaner on the corrosion behavior of the materials tested, make the following test:

10.1.1 Immerse clean test panels in solutions of the cleaner of the following concentrations and temperatures, allowing the panels to come to the temperature of the solution, and then slowly withdrawing them by a suitable apparatus such as a Fisher-Payne Dip Coater in such a manner as to leave a thin uniform coating of cleaner on the surface of the specimen:

TABLE 1 Weight Losses of 0.5 dm^2 Specimens Subjected to Electrolytic Cleaning Treatment

Material	Total Weight Loss, g
Copper-nickel-zinc (75-20-5)	0.0000
Brass (admiralty)	0.0001
Brass (red)	0.0000
Brass (yellow)	0.0002
Bronze (phosphor, 5 percent tin)	0.0000
Bronze (silicon)	0.0002
Bronze (case) (85-5-5-5)	0.0010
Copper	0.0001
Copper-nickel (70-30)	0.0000
Iron and steel	0.0003
Nickel-molybdenum-iron (60-20-20)	0.0004
Nickel-chromium-iron (80-13-7)	0.0000
Lead (chemical)	0.0030
Nickel-copper (70-30)	0.0000
Nickel	0.0011
Stainless steel	0.0000
Tin	0.0003
Magnesium and zinc	too high to be useful

10.1.1.1 The concentration of the test solution shall be one tenth the concentration recommended by the manufacturer, or 0.5 weight % when no concentration is recommended.

10.1.1.2 The temperature of the solution shall be that recommended by the manufacturer or, in the absence of any recommendation, the boiling point of the solution.

10.1.2 Dry specimens at a moderate temperature (about 212°F (100°C)).

10.1.3 Expose the dried specimens to 90 % relative humidity at 95°F (35°C) for one week.

10.1.4 Treat the specimens as indicated in 8.3.4-8.3.7.

11. Report

11.1 *Quantitative Weight Loss Test*—Report the following data for each test performed:

11.1.1 Temperature,

11.1.2 Specimens, size and number,

11.1.3 Cleaner concentrations,

11.1.4 Metal, alloy, surface treatment, and metallurgical state,

11.1.5 Ratio of surface area to volume of solution,

11.1.6 Type of cleaner and other conditions of test peculiar to type,

11.1.7 Weight loss in milligrams per square decimetre hour,

11.1.8 Range in weight loss values, and

11.1.9 Appearance before and after removal of corrosion products with regard to the following: (*1*) discoloration, (*2*) dulling, (*3*) etching, (*4*) presence of accretions and relative amounts and areas, (*5*) type of pitting—wide, medium, or narrow, and (*6*) presence of selective or localized attack.

11.2 *Qualitative Surface Corrosion Test*—Report the same information as for the quantitative weight loss test (11.1), except to omit 11.1.7 and 11.1.8.

12. Precision and Bias

12.1 Since the conditions for testing are to be selected by the user, and therefore vary, no precision or bias data have been obtained.

APPENDIXES

(Nonmandatory Information)

X1. TEST METHOD FOR ELECTROLYTIC CLEANING OF CORROSION TEST SPECIMENS AFTER EXPOSURE

X1.1 After scrubbing to remove loosely attached corrosion products, treat the specimen as a cathode in hot, dilute sulfuric acid under the following conditions:

Test solution	sulfuric acid (5 weight %)
Inhibitor	2 mL organic inhibitor per litre of solution
Anode	carbon
Cathode	test specimen
Cathode current density	20 A/dm^2(6.5 A/0.327 dm^2)
Temperature	165°F (74°C)
Exposure period	3 min

X1.2 After the electrolytic treatment, scrub the specimens, while wet, with a stiff bristle brush. The weight losses of specimens 0.5 dm^2 in area, treated by the test method described, have been found to be of the order shown in Table 1.

X1.3 It should be noted that this electrolytic treatment may result in the redeposition of adherent metal from reducible corrosion products, and thus lower the apparent weight loss. However, general experience has indicated that in most cases of corrosion in liquids the possible errors from this source are not likely to be serious. Instead of using 2 mL of any proprietary inhibitor, about 0.5 g/L of such inhibitors as diorthotolyl thiourea, quinoline ethiodide, or betanaphthol quinoline may be used.

X2. CHEMICAL TEST METHODS FOR CLEANING CORRODED SPECIMENS WITHOUT USE OF ELECTRIC CURRENT

X2.1 *Copper and Nickel Alloys*—Dip for 2 to 3 min in hydrochloric acid (1 + 1) or sulfuric acid (1 + 10) at room temperature. Scrub with a bristle brush under running water, and dry.

X2.2 *Tin Alloys*—Dip for 10 min in boiling trisodium phosphate solution (15 weight %) prepared from material conforming to Specification D 538, Trisodium Phosphate. Scrub with a bristle brush under running water, and dry.

X2.3 *Lead Alloys*—Dip for 10 min in boiling acetic acid (1 volume %). Brush with a bristle brush under running water, and dry.

X2.4 *Zinc*—Immerse the specimens in warm (140 to 180°F) ammonium chloride (10 weight %) for several min. Rinse in water, and scrub with a soft brush. The accumulation of corrosion products is removed almost completely by this treatment. Then immerse the specimens for 15 to 20 s in a boiling solution of chromic acid (5 weight %) and silver nitrate (1 weight %). Rinse in hot water, and dry.

NOTE X2.1—In making up the chromic acid solution, it is advisable to dissolve the silver nitrate separately and add it to the boiling chromic acid to prevent excessive crystallization of the silver chromate. The chromic acid must be free from sulfate to avoid attack on the zinc.

X2.5 *Magnesium Alloys*—Clean for approximately 1 min in chromic acid (20 weight %) to which has been added, with agitation, 1 weight % of silver nitrate in solution form. Operate the bath at 200 to 212°F.

X2.6 *Iron and Steel*—Clean for 2 to 3 min by immersion in a boiling solution of ammonium citrate (10 weight %).

X2.7 *Stainless Steel*—Clean for 5 min by immersion in a solution of nitric acid (30 volume %) at a temperature of 120 to 130°F.