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Standard Practice for Laboratory Screening of Metallic Containment Materials for Use With Liquids in Solar Heating and Cooling Systems¹

This standard is issued under the fixed designation E 712; 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 practice covers several laboratory test procedures for evaluating corrosion performance of metallic containment materials under conditions similar to those that may occur in solar heating and cooling systems. All test results relate to the performance of the metallic containment material only as a part of a metal/fluid pair. Performance in these laboratory test procedures, taken by itself, does not necessarily constitute an adequate basis for acceptance or rejection of a particular metal/fluid pair in solar heating and cooling systems, either in general or in a particular design. This practice is not intended to preclude the use of other screening tests, particularly when those tests are designed to more closely simulate field service conditions.

1.2 This practice describes apparatus and procedures for several tests, any one or more of which may be used to evaluate the deterioration of the metallic containment material in a metal/fluid pair. The procedures are designed to permit simulation, heating, and cooling systems including (1) operating full flow, (2) stagnant full, (3) stagnant partial fill, and (4) stagnant empty. Particular attention should be directed to properly reflecting whether the system is open or closed to atmosphere.

1.3 This practice covers the following six tests:

Practice A	Basic Immersion Test at Atmospheric Pressure
Practice B	Heat-Rejecting Surface Test at Atmospheric Pressure
Practice C	High-Pressure Test
Practice D	Repeated Dip Dry Test at Atmospheric Pressure
Practice E	Crevice Test at Atmospheric Pressure
Practice F	Tube Loop Test at Atmospheric Pressure

1.4 Practice A is concerned with the interaction of metal and fluid when both are at the same temperature with no heat transfer from one to the other. It is regarded as useful for plumbing, pumps, tanking, etc., but of less significance, taken by itself, for collector panels. Practices B and F are concerned with the deterioration of the metal when there is transfer of heat from the metal into the heat transfer fluid. These practices are

¹ These test methods are under the jurisdiction of ASTM Committee E44 on Solar, Geothermal, and Other Alternative Energy Sources and is the direct responsibility of Subcommittee E44.05 on Solar Heating and Cooling Subsystems and Systems.

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especially applicable to the collector panel. Practice C permits a variety of tests but is especially useful in relation to systems that experience high temperatures, or are closed to the atmosphere. Practices D and E evaluate specific corrosion problems that may be associated with particular metal/fluid pairs and particular designs of systems and components.

1.5 *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:

- D 1384 Test Method for Corrosion Test for Engine Coolants in Glassware²
- G 1 Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens³
- G 48 Test Methods for Pitting and Crevice Corrosion Resistance of Stainless Steels and Related Alloys by the Use of Ferric Chloride Solution³

3. Significance and Use

3.1 At this time, none of these tests has been demonstrated to correlate with field service.

3.2 It is essential that consideration be given to the appropriate pairing of metal and fluid since these procedures do not restrict the selection of either the containment material or the fluid for testing. Likewise, knowledge of the corrosion protection mechanism and the probable mode of failure of a particular metal is helpful in the selection of test conditions and the observation, interpretation, and reporting of test results.

3.3 The design of solar heating and cooling systems strongly affects the applicability of the results of the laboratory screening tests. Therefore, the results of these laboratory procedures should be confirmed by component and systems testing under actual or simulated service conditions.

3.4 Table 1 is provided to assist in an orderly consideration of the important factors in testing. It is expected that the user

² *Annual Book of ASTM Standards*, Vol 15.05.

³ *Annual Book of ASTM Standards*, Vol 03.02.

TABLE 1 Significant Variables in Evaluation of Containment Material/Heat Transfer Fluid Pairs^A

Test Aspect	Variable	
	Temperature	Flow Rate
I. <i>Operating Conditions of System:</i>		
A. Operating, full flow	normal operating	normal operating
B. Stagnant, full	fluid boiling point without pressurization or no-flow temperature with pressurization	convection
C. Stagnant, partial fill	same as stagnant, full	convection
D. Stagnant, empty	no-flow temperature	not applicable
II. <i>Test Specimen Design</i>	A. flat metal couple B. metal couple with crevice C. dissimilar metal couple D. dissimilar metal couple with crevice	
III. <i>Fluid Type</i>	A. fluid intended for use in system B. fluid pretreated by thermal exposure or chemical contamination	
IV. <i>Test Cycle</i>	A. long time, constant temperature B. cycles of heating, holding, and cooling C. cycles of operating full flow, and stagnation D. cycles of wetting and drying	

^AIn this table, the subdivisions are not necessarily related in correspondence to their lettering.

of the test procedure will investigate a range of test times and temperatures for the containment material in a metal/fluid pair, and adjust the time and temperature of testing as necessary.

NOTE 1—Corrosion, whether general or localized, is a time-dependent phenomenon. This time dependence can show substantial nonlinearity. For example, formation of a protective oxide will diminish corrosion with time, while certain forms of localized attack accelerate with time. The minimum time required for a test to provide a corrosion rate that can be extrapolated for the prediction of long-term performance varies widely, depending on the selection of metal and fluid, and on the form of corrosion attack. Therefore, it is not possible to establish a single minimum length of test applicable to all materials and conditions. However, it is recommended that for the tests described in this practice, a test period of no less than 30 days be used. Furthermore, it is recommended that the effect of time of testing be evaluated to detect any significant time dependence of corrosion attack.

3.5 It is essential for the meaningful application of these procedures that the length of the test be adequate to detect changes in the nature of the fluid that might significantly alter the corrosivity of the fluid. For example, exhaustion of chemical inhibitor or chemical breakdown of the fluid may occur after periods of months in selected cycles of operation.

NOTE 2—Many fluids that may be considered for solar applications contain additives to minimize the corrosivity of the fluid. Many such additives are useful only within a specific concentration range, and some additives may actually accelerate corrosion if the concentration falls below a critical level. Depletion kinetics can be a strong function of the exposed metal surface area. Therefore, for tests involving fluids with such additives, consideration must be given to the ratio of metal surface area to fluid volume as it may relate to an operating system.

4. Selection of Materials and Reagents

4.1 Any metallic material may be selected for evaluation. The material shall be capable of being described with sufficient accuracy to permit reproduction of the test.

4.2 Any heat-transfer fluid may be selected for evaluation. However, it is expected that the fluid will be selected with consideration given to possible interactions of material and fluid under the conditions of testing. The fluid should be

capable of being described chemically, as to its basic components and the presence or absence of minor components that affect the interaction with the metal. It is permitted to precondition the fluid before testing. Any such preconditioning treatment shall be described in the report.

4.3 Particular attention shall be directed to avoidance of materials, fluids, or metal/fluid pairs that can be hazardous to the operator. The flammability, vapor pressure, and toxicity of the heat-transfer fluid shall be known prior to initiation of testing and appropriate precautionary measures shall be taken to ensure the safety of all test personnel.

5. Sampling and Test Specimens

5.1 The test specimens shall be selected from material that may reasonably represent that material as it would be applied in a solar heating and cooling system.

5.2 For laboratory corrosion tests that simulate exposure to service environments, a commercial surface, such as a mill finish, closely resembling the one that would be used in service, will yield the most significant results. For more searching tests of either the metal or the environment, standard surface finishes may be preferred. Ideally, the surface finish should be recorded in surface roughness terms, such as rms inches.

5.3 General Cleaning:

5.3.1 General cleaning may be accomplished with a wide variety of cleaning media. Water-based cleaners should be followed by an alcohol dip after thorough rinsing. Solvent cleaners such as petroleum fractions, aromatic hydrocarbons, and chlorinated hydrocarbons are generally acceptable. Chlorinated solvents, however, should not be used on titanium, stainless steel, or aluminum. Mechanical cleaning of very smooth surfaces may be accomplished by the use of a paste of magnesium oxide or alumina.

5.3.2 Any of the methods suitable for cleaning a given corroded specimen may be used to complete the cleaning of specimens prior to test, provided that they do not cause

localized attack. The cleaned specimens should be measured and weighed. Dimensions determined to the third significant figure and mass determined in the fifth significant figure are usually satisfactory.

5.4 Metallurgical Condition—Specimen preparation may change the metallurgical condition of the metal. For example, shearing a specimen to size will cold-work and possibly fracture the edges. The specimen may be tested in this condition if it is believed that such a condition may be encountered in service. In this case, the condition shall be described in the report of results. However, it is recommended that changes in metallurgical condition be corrected for customary testing. For example, sheared edges should be machined or the specimen annealed.

5.5 Alternative specimen designs, particularly those incorporating crevices or metal couplings as may be encountered in application, are recommended.

5.6 For many metals, electrolytic cleaning is a satisfactory method for cleaning after testing. The following method is typical:

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

5.6.1.1 Electrolyte—Sulfuric acid (H_2SO_4) (5 mass %).

5.6.1.2 Inhibitor—0.2 vol % of organic inhibitor (see Note 3).

5.6.1.3 Anode—Carbon or lead (see Note 4).

5.6.1.4 Cathode—Test specimen.

5.6.1.5 Cathode Current Density—2000 A/m².

5.6.1.6 Temperature—75°C (165°F).

5.6.1.7 Exposure Period—3 min.

NOTE 3—Instead of using 0.2 vol % of any proprietary inhibitor and 0.5 kg/m³ of inhibitors such as diorthotolyl thiourea, quinoline ethiodide or betanaphtol quinoline may be used.

NOTE 4—If lead anodes are used, lead may deposit on the specimen and cause an error in the mass loss. If the specimen is resistant to nitric acid, the lead may be removed by a flash dip in 1 + 1 nitric acid. Except for the possible source of error, lead is preferred as an anode as it gives more efficient corrosion product removal.

5.6.2 After the electrolytic treatment, scrub the specimens with a brush, rinse thoroughly, and dry.

5.6.3 It should be noted that this electrolytic treatment may result in the redeposition of metal, such as copper, from reducible corrosion products, and thus, lower the apparent mass loss.

5.7 Chemical cleaning of specimens after testing is satisfactory provided the following procedures are used:

5.7.1 Copper and Nickel Alloys—Dip for 1 to 3 min in HCl (1 + 1) or H_2SO_4 (1 + 10) at room temperature. Scrub lightly with bristle brush under running water, using fine scouring powder if needed.

5.7.2 Aluminum Alloys—Dip for 5 to 10 min in a water solution containing 2 mass % of chromic acid (chromium trioxide, $CrCO_3$) and 5 vol % of orthophosphoric acid (H_3PO_4 , 85 %) maintained at 80°C (175°F). Ultrasonic agitation will facilitate this procedure. Rinse in water to remove the acid, brush very lightly with a soft bristle brush to remove any loose film, and rinse again. If film remains, immerse 1 min in

concentrated nitric acid and repeat previous steps. Nitric acid alone may be used if there are no deposits.

5.7.3 Tin Alloys—Dip for 10 min in boiling trisodium phosphate solution (15 %). Scrub lightly with bristle brush under running water and dry.

5.7.4 Iron and Steel—Suitable methods are as follows:

5.7.4.1 Preferably, use electrolytic cleaning (see 5.6).

5.7.4.2 Immerse in Clark's solution (hydrochloric acid—100 parts, antimonious oxide—2 parts, stannous chloride—5 parts) for up to 25 min. Solution may be cold, but it should be stirred vigorously.

5.7.4.3 Remove scales formed on steel under oxidizing conditions in 15 vol % concentrated phosphoric acid containing 0.15 vol % of organic inhibitor at room temperature.

5.7.4.4 Clean stainless steel in 20 % nitric acid at 60°C (140°F) for 20 min.

5.7.4.5 In place of chemical cleaning use a brass scraper or brass bristle brush, or both, followed by scrubbing with a wet bristle brush and fine scouring powder.

NOTE 5—Such vigorous mechanical cleaning is applicable when mass loss is large and hence errors in mass loss will produce only small errors in corrosion rates. Blank corrections will be difficult to apply.

5.7.4.6 Other methods of cleaning iron and steel include immersion in hot sodium hydride, and cathodic pickling in molten caustic soda.

NOTE 6—These methods may be hazardous to personnel. They should not be carried out by untrained personnel or without supervision.

5.7.5 After cleaning and thorough rinsing, dry and weigh the samples.

6. Calculations and Interpretation of Results

6.1 The deterioration of the containment material shall be determined by measurement of mass loss and by examination at 10× magnification for incidence of localized attack.

6.1.1 Whichever cleaning method is used, the possibility of removal of solid metal is present. Such removal would result in error in the determination of the corrosion rate. One or more cleaned and weighed specimens should be recleaned by the same method and reweighed. Loss due to this second weighing may be used as a correction of the first one.

NOTE 7—The use of suitable inhibitors will diminish the attack and will permit reasonable degree of reproducibility with specimens varying in degree of rusting.

6.1.2 The total surface is calculated (making allowance for the change in surface area due to mounting holes). The mass loss is divided by the area to get a mass loss per unit area. This again may be divided by the duration of the test to get a corrosion rate in mass loss per unit area per unit time (such as mg/dm²·day = mdd). This value may be divided by the density of the metal and modified by appropriate conversion factors to obtain a figure in terms of rate of loss in thickness of the specimen (such as mils per year = mpy).

6.1.2.1 For example:

$$R_{mdd} = 100\,000 [(W_o - W_i)/AT] \quad (1)$$

where:

R_{mdd} = corrosion rate, mdd,

W_o = original mass, g,
 W_t = final mass, g,
 A = area, cm², and
 T = duration, days.

or

$$R_{\text{mpy}} = 393.7 [(W_o - W_t)/ATD] \quad (2)$$

where:

R_{mpy} = corrosion rate, mpy,
 W_o = original mass, g,
 W_t = final mass, g,
 A = area, cm²,
 T = duration, years, and
 D = density, g/cm³.

6.1.3 Any incidence of localized corrosion, whether pitting, crevice attack, intergranular attack, cracking, or any other form of localized attack, shall be identified and rated under at least 10× magnification, and shall be reported. The location, distribution, and maximum depth of attack shall be reported for any localized attack.

6.2 Any changes of the heat transfer fluid, such as appearance or odor, should be reported with the results. Any changes in the appearance or condition of the test apparatus indicative of interaction with the metal specimen or fluid shall be described.

6.3 In the event of film formation and buildup, the nature of the film and its degree of buildup shall be reported.

6.4 For the evaluation of containment material couple, an effort should be made to utilize the same procedures as for a single material test. However, because of the variability permitted in the design of the specimen for the couple, it may be appropriate to report mass loss or penetration. For all tests of metal couple/fluid performance, special attention should be given to observation and reporting of localized corrosion and evidence of galvanic attack.

7. Report

7.1 The containment material shall be identified by standard specification where applicable, or by chemical analysis. In case of identification by standard specification, supplemental identification by typical analysis for such specification, or by chemical analysis of the specimen is desirable.

7.2 The dimensions and configuration of the specimen shall be reported. In the case of metal couple, the description shall include at least the following elements: (1) a description of the individual components of the couple; (2) a description of the method of attachment or association of the couple including any third material introduced as a binder or for other function and the procedures of connection, for example, surface preparation, conditions of attachment, and cleaning; (3) any change of the containment materials resulting from the coupling procedure; and (4) a description of the relative areas of exposure of the components of the couple to the heat transfer medium.

7.3 The heat-transfer fluid shall be identified by standard specification where applicable, by initial chemical analysis, or by proprietary designation. Use of trademarks or names of patented or proprietary products, without accompanying

chemical description, is discouraged but not prohibited. For aqueous transfer fluid, the analysis of the water used shall be reported.

7.4 The test used shall be identified. The test conditions used shall be specified, including specimen preparation, time and temperature schedule, degree of atmospheric exposure of the heat-transfer fluid, stirring, and flow rate, where applicable. The method of temperature measurement and control, with comment on its accuracy and precision, shall be described. The nature of boiling of the fluid shall be described if boiling was observed during the test. Any deviation from the standard procedure shall be reported and so identified as a deviation.

7.5 The report shall provide both mass loss and average penetration rate when applicable. The time dependence of the corrosion rate shall be commented upon (see Note 1) with a plot of corrosion rate as a function of time being provided when this time dependence is significant. All instances of localized deterioration of the test specimen shall be reported. In the event of pitting or other nonuniform attack, the frequency of attack and maximum penetration shall be reported.

7.6 A commentary on the results and their interpretation, particularly their applicability to various designs for solar heating and cooling systems, is optional but desirable.

PRACTICE A—BASIC IMMERSION TEST AT ATMOSPHERIC PRESSURE

8. Scope

8.1 This test is intended to provide a simple, rapid exposure test for evaluation of metal and fluid interaction. The apparatus, as typically constructed, is open to the atmosphere. Therefore, the results of this test may not be applicable to closed systems.

9. Apparatus

9.1 The vessel is typically a 1000-mL beaker or reaction flask or heat-resistant glass (see Note 8). Provision is made for closing the top of the reaction vessel while providing openings for temperature-measuring devices, reflux condenser, and stirring device, as necessary. The specimen may be suspended in a cradle of nonmetallic material or supported by a rack either constructed of a nonmetallic material or insulated so as to prevent galvanic interaction of specimen and rack.

NOTE 8—For certain containment materials there exists the possibility that silicate from the glass of the apparatus contaminating an aqueous heat transfer fluid, would significantly affect the corrosion observed in this test. The effect of silicate from this source would be minimal in those cases in which silicates are a part of the corrosion inhibitor system, or in which silicates are otherwise present in the heat transfer fluid. However, for those cases in which the effect of silicate from glassware could have a significant effect on the results, it is recommended that other materials be used for the apparatus, such that no significant extraneous effects will be introduced in the results of the test (see Ref (1)).

9.2 The vessel may be heated by mantle, hot plate, or bath. Selection of heating method can affect accuracy of temperature control. For certain fluids the more localized heating typical of a hot plate in comparison with the constant-temperature bath, may produce changes in the heat-transfer fluids.

9.3 The fluid may be stirred to simulate flow conditions. For those fluids in which aeration or deaeration can be simulated by