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# Standard Practices for Simulated Service Testing for Corrosion of Metallic Containment Materials for Use With Heat-Transfer Fluids in Solar Heating and Cooling Systems<sup>1</sup>

This standard is issued under the fixed designation E 745; 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.

## 1. Scope

1.1 These practices cover test procedures simulating field service for evaluating the performance under corrosive conditions of metallic containment materials 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 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.

1.2 These practices describe test procedures used to evaluate the resistance to deterioration of metallic containment materials in the several conditions that may occur in operation of solar heating and cooling systems. These conditions include: (1) operating full flow; (2) stagnant empty vented; (3) stagnant, closed to atmosphere, non-draindown; and (4) stagnant, closed to atmosphere, draindown.

1.3 The recommended practices cover the following three tests:

1.3.1 *Practice A*—Laboratory Exposure Test for Coupon Specimens.

1.3.2 *Practice B*—Laboratory Exposure Test of Components or Subcomponents.

1.3.3 *Practice C*—Field Exposure Test of Components or Subcomponents.

1.4 Practice A provides a laboratory simulation of various operating conditions of solar heating and cooling systems. It utilizes coupon test specimens and does not provide for heating of the fluid by the containment material. Practice B provides a laboratory simulation of various operating conditions of a solar heating and cooling system utilizing a component or a simulated subcomponent construction, and does provide for heating of the fluid by the containment material. Practice C provides a field simulation of various operating conditions of solar heating and cooling systems utilizing a component or a simulated

subcomponent construction. It utilizes controlled schedules of operation in a field test.

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.* For a specific safety precaution statement see Section 6.

## 2. Referenced Documents

### 2.1 ASTM Standards:

E 712 Practice for Laboratory Screening of Metallic Containment Materials for Use With Liquids in Solar Heating and Cooling Systems<sup>2</sup>

G 1 Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens<sup>3</sup>

## 3. Terminology

### 3.1 Definitions:

3.1.1 *collector, n*—a device designed to absorb incident solar radiation and transfer the energy to a heat-transfer fluid. A collector has an absorber surface, a containment membrane, and may or may not have insulation and glazing.

3.1.2 *panel, n*—the absorber surface and containment membrane within the collector.

3.1.3 *component, n*—an individually distinguishable product that forms part of a more complex product, that is, a subsystem or system. The panel and collector are each components.

3.1.4 *simulated subcomponent, n*—a specimen fabricated in such a manner as to embody the major characteristics of a component with regard to material selection, design, forming, joining, and surface condition.

## 4. Significance and Use

4.1 At this time none of these practices have been demonstrated to correlate with field service.

4.2 Because these procedures do not restrict the selection of either the containment material or the fluid for testing, it is

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 12.02.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 03.02.

essential that consideration be given to the appropriate pairing of metal and fluid. 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.

4.3 It is important that consideration be given to each of the permitted variables in test procedure so that the results will be meaningfully related to field performance. It is especially important that the time of testing selected be adequate to correctly measure the rate of corrosion of the containment material.

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 corrosion 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 these practices, a test period of no less than 6 months be used. Furthermore, it is recommended that the effect of time of testing be evaluated to detect any significant time dependence of corrosion attack.

4.4 It is essential for the meaningful application of these procedures that the length of 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.

## 5. Materials

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

5.2 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 as to 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.

## 6. Safety Precautions

6.1 Particular attention must 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.

## 7. Calculations and Interpretation of Results

7.1 Determine the deterioration of the containment material by measurement of weight loss when possible, by measurement of metal thinning, and by examination at 10× magnification for incidence of localized attack.

7.1.1 Whatever cleaning method is used, the possibility of removal of solid metal is present; this results in error in the determination of the corrosion rate. One or more cleaned and examined specimens should be recleaned by the same method and re-examined. Loss due to this second cleaning may be used as a correction to the first one.

7.1.2 To determine the corrosion rates based on weight loss, calculate the total surface area (making allowance for the change in surface area due to mounting holes) and divide the weight loss by the area to obtain the weight loss per unit area. This result may be divided by the duration of the test to obtain the corrosion rate in weight loss per unit area per unit time (such as mg/dm<sup>2</sup>·day = mdd). This result may be divided by the density of the metal to obtain a rate of loss in terms of thickness of the specimen (mils per year = mpy), for instance:

$$R_{\text{mdd}} = 100\,000 (W_o - W_t)/AT \quad (1)$$

where:

$R_{\text{mdd}}$  = the corrosion rate, mdd,  
 $W_o$  = original weight, g,  
 $W_t$  = final weight, g,  
 $A$  = area, cm<sup>2</sup>, and  
 $T$  = duration, days.

or

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

where:

$R_{\text{mpy}}$  = corrosion rate, mpy,  
 $W_o$  = original weight, g,  
 $W_t$  = final weight, g,  
 $A$  = area, cm<sup>2</sup>,  
 $T$  = duration, years, and  
 $D$  = density, g/cm<sup>3</sup>.

7.1.3 Identify any incidence of localized corrosion, whether pitting, crevice attack, intergranular attack, cracking, or any other form of localized attack, rate under at least 10× magnification, and report. Report the location, distribution, and maximum depth of attack for any localized attack.

7.2 Report any changes of the heat-transfer fluid, for example, appearance or odor, and include the results. Describe any changes in the appearance or condition of the test apparatus indicative of interaction with the metal specimen or fluid.

7.3 In the event of film formation and buildup, report the nature of the film and its degree of buildup.

7.4 For the evaluation of a 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 not be appropriate to report weight 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.

## 8. Report

8.1 Identify the containment material using a recognized standard test method, where applicable, or by chemical analysis. In case of identification by a standard method, supplemental identification by typical analysis for that standard, or by chemical analysis of the specimen is desirable.

8.2 Report the dimensions and configuration of the specimen. In the case of a metal couple, the report shall include at least the following elements: (1) description of the individual components of the couple; (2) 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 or connection, for example, surface preparation, conditions of attachment, and cleaning; (3) any change of the containment materials resulting from the coupling procedure; and (4) description of the relative areas of exposure of the components of the couple to the heat-transfer medium.

8.3 The heat-transfer fluid shall be identified by standard methods 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 fluids, the analysis of the water used shall be reported.

8.4 Identify the procedure used. Specify the test conditions used, including specimen preparation, time and temperature schedule, degree of atmospheric exposure of the heat transfer fluid, stirring, and flow rate, where applicable. Describe the method of temperature measurement and control, with comment on its accuracy and precision. Report any deviation from the standard procedure and so identify as a deviation.

8.5 Report the weight loss and average penetration rate, when applicable. If the time dependence of the corrosion rate is significant, (see Note 1), note this in the report, and include a plot of the corrosion rate as a function of time. Report all instances of localized deterioration of the test specimen. In the event of pitting or other non-uniform attack, report the frequency of attack and maximum penetration.

8.6 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 LABORATORY EXPOSURE TEST FOR COUPON SPECIMENS

### 9. Scope

9.1 This procedure is intended to evaluate the resistance to deterioration of metallic containment materials in contact with various heat-transfer fluids. By proper selection of test conditions, a wide range of operating conditions may be evaluated. However, the test procedure does not provide for a condition wherein heat is transferred from the containment material into the fluid.

### 10. Test Specimens and Sample

10.1 Select the test specimens from material that may reasonably represent that material as it would be applied in a solar heating and cooling system.

10.2 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-in.

#### 10.3 General Cleaning:

10.3.1 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 using a paste of magnesium oxide or aluminum oxide.

10.3.2 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 weighted. Dimensions determined to the third significant figure and weight determined to the fifth significant figure are usually satisfactory.

10.4 *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 condition may be encountered in service. In such 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.

10.5 *Alternative Specimen Designs*—Alternative specimen designs, particularly those incorporating crevices or metal coupling as may be encountered in service, are recommended.

10.6 *Electrolytic Cleaning*—For many metals, electrolytic cleaning is a satisfactory method for cleaning after testing.

10.6.1 The following method is typical: after scrubbing to remove loosely attached corrosion products, treat the specimen as a cathode in hot, dilute sulfuric acid under the following conditions:

10.6.1.1 *Electrolyte*—Sulfuric acid (5 weight %),

10.6.1.2 *Inhibitor*—0.2 volume % organic inhibitor (see Note 3),

10.6.1.3 *Anode*—Carbon or lead (see Note 4),

10.6.1.4 *Cathode*—Test specimen,

10.6.1.5 *Cathode Current Density*—2000 A/m<sup>2</sup>,

10.6.1.6 *Temperature*—165°F (75°C), and

10.6.1.7 *Exposure Period*—3 min.

NOTE 3—Instead of using 0.2 volume % of any proprietary inhibitor, about 0.5 kg/m<sup>3</sup> of inhibitors such as diorthotolyl thiourea, quinoline ethiodide, or betanaphthol quinoline may be used.

**NOTE 4**—If lead anodes are used, lead may deposit on the specimen and cause an error in the weight 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.

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

10.6.3 This electrolytic treatment may result in the redeposition of metal, such as copper, from reducible corrosion products and, thus, lower the apparent weight loss.

10.7 *Chemical Cleaning*—Chemical cleaning of specimens after testing is satisfactory provided the following methods are used:

10.7.1 *Copper and Nickel Alloys*—Dip for 1 to 3 min in HCl (1 + 1) or H<sub>2</sub>SO<sub>4</sub> (1 + 10) at room temperature. Scrub lightly with bristle brush under running water, using fine scouring powder if needed.

10.7.2 *Aluminum Alloys*—Dip for 5 to 10 min in a water solution containing 2 weight % of chromic acid (chromium trioxide, CrO<sub>3</sub>) and 5 vol % of orthophosphoric acid (H<sub>3</sub>PO<sub>4</sub>, 85 %) maintained at 175°F (80°C). 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.

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

10.7.4 *Iron and Steel*—Suitable methods are as follows:

10.7.4.1 Preferably, use electrolytic cleaning (see 10.6).

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

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

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

10.7.4.5 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 weight losses are large and hence errors in weight loss will produce only small errors in corrosion rates. Blank connections will be difficult to apply.

10.7.4.6 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 the uninitiated or without professional supervision.

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

## 11. Apparatus

11.1 *Test Cell*—For those portions of the test procedure that may be performed at atmospheric pressure, the test cell is

typically a 4-L container of heat-resistant glass (see Note 7). Provision is made for closing the top of the test cell with a machinable nonmetallic cover (for example, polypropylene, acetal copolymers), while providing opening for temperature-measuring devices, tubes for simulating draindown, and tubes for gas sparging (see Fig. 1). All materials used in the apparatus shall be selected so that there is no significant interaction, other than the possibility of a mechanically imposed crevice, with the containment material or the heat-transfer fluid.

**NOTE 7**—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.

11.2 *Specimen Holders*—Specimens shall be secured to nonmetallic holders utilizing a nonmetallic fastener or a fastener of the same material, similar in composition and processing. The specimen holders are in turn secured to the mixing shaft. For the purpose of simulating field service conditions, dissimilar metals that exist in operating systems (for example, steel storage tanks, Pb-Sn solders, copper plumbing) may be included in the cell. Galvanic couples may be established between materials that are in electrical contact in operating systems.

11.3 *Mixing Shaft*, capable of rotating, such that the outer edge of the specimen encounters a fluid-to-metal speed approximately equivalent to fluid flow rates encountered in service.

11.4 *Heating Device*—The test cell may be heated by mantle, hot plate, or bath. Use of the bath may minimize the variation in heating and cooling rates when metal/fluid pairs are being evaluated. For certain fluids, the more localized heating typical of a hot plate in comparison with the other methods, may produce changes in the heat-transfer fluid.

11.5 Metal/fluid heating and cooling rates, and the upper and lower temperature limits, should be selected to simulate field service conditions. Control of temperature at the upper and lower limits should be  $\pm 5^\circ\text{F}$  ( $3^\circ\text{C}$ ). Cooling below ambient may be accomplished by a refrigeration unit.

11.6 Conditions of aeration and deaeration may be accomplished by gas sparging.

11.7 *Autoclave*—For test conditions that cannot be achieved in open atmosphere, the mixing shaft and attached specimens can be transferred to an autoclave.

## 12. Procedure

12.1 Cut metallic containment test specimens from sheet having a gage similar to the wall of the solar absorber channel. The metallurgical condition of the test specimen should be similar to that of the finished absorber product. Typical test specimen dimensions are 1 $\frac{5}{8}$  (41 mm) in. long by 1 in. (25 mm) wide. Dimensions should be maintained to within  $\pm 0.005$  in. (0.10 mm).