Designation: C1617 – 15

Standard Practice for Quantitative Accelerated Laboratory Evaluation of Extraction Solutions Containing Ions Leached from Thermal Insulation on Aqueous Corrosion of Metals[1]

This standard is issued under the fixed designation C1617; 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 procedures for a quantitative accelerated laboratory evaluation of the influence of extraction solutions containing ions leached from thermal insulation on the aqueous corrosion of metals. The primary intent of the practice is for use with thermal insulation and associated materials that contribute to, or alternatively inhibit, the aqueous corrosion of different types and grades of metals due to soluble ions that are leached by water from within the insulation. The quantitative evaluation criteria are Mass Loss Corrosion Rate (MLCR) expressed in mils per year determined from the weight loss due to corrosion of exposed metal coupons after they are cleaned.

1.2 The insulation extraction solutions prepared for use in the test can be altered by the addition of corrosive ions to the solutions to simulate contamination from an external source. Ions expected to provide corrosion inhibition can be added to investigate their inhibitory effect.

1.3 Prepared laboratory standard solutions are used as reference solutions and controls, to provide a means of calibration and comparison. See Fig. 1 and Table 1.

1.4 Other liquids can be tested for their potential corrosiveness including cooling tower water, boiler feed, and chemical stocks. Added chemical inhibitors or protective coatings applied to the metal can also be evaluated using the general guidelines of the practice.

1.5 This practice cannot cover all possible field conditions that contribute to aqueous corrosion. The intent is to provide an accelerated means to obtain a non-subjective numeric value for judging the potential contribution to the corrosion of metals that can come from ions contained in thermal insulation materials or other experimental solutions. The calculated numeric value is the mass loss corrosion rate. This calculation is based on general corrosion spread equally over the test duration and the exposed area of the experimental cells created for the test. Corrosion found in field situations and this accelerated test also involves pitting and edge effects and the rate changes over time.

1.6 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.

1.7 *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:*[2]

A53/A53M Specification for Pipe, Steel, Black and Hot-Dipped, Zinc-Coated, Welded and Seamless
A105/A105M Specification for Carbon Steel Forgings for Piping Applications
C168 Terminology Relating to Thermal Insulation
C518 Test Method for Steady-State Thermal Transmission Properties by Means of the Heat Flow Meter Apparatus
C665 Specification for Mineral-Fiber Blanket Thermal Insulation for Light Frame Construction and Manufactured Housing
C692 Test Method for Evaluating the Influence of Thermal Insulations on External Stress Corrosion Cracking Tendency of Austenitic Stainless Steel
C739 Specification for Cellulosic Fiber Loose-Fill Thermal Insulation
C795 Specification for Thermal Insulation for Use in Contact with Austenitic Stainless Steel

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[1] This practice is under the jurisdiction of ASTM Committee C16 on Thermal Insulation and is the direct responsibility of Subcommittee C16.31 on Chemical and Physical Properties.

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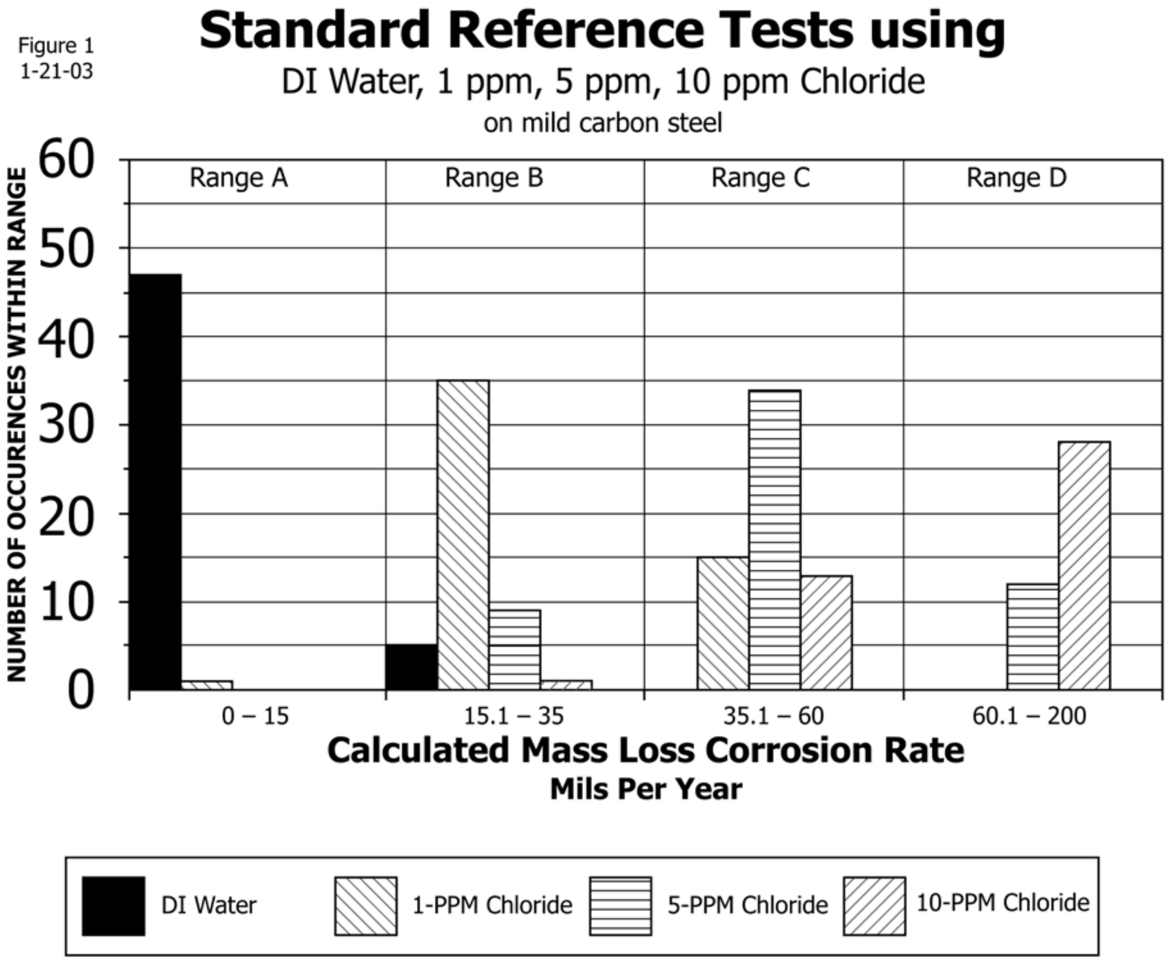
[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.



Note 1—The Fig. 1 bar graph was created using the MLCR data shown in Table 1. Standard reference tests using de-ionized water, 1 ppm, 5 ppm, and 10 ppm chloride solutions were performed on mild carbon steel coupons. The calculated MLCR test results for mild carbon steel coupons were separated into four ranges. The rating criteria ranges were developed to accommodate the results obtained using this practice on the reference standards and experimental insulation samples. The ranges used are: MLCR = 0 to 15 mils = range A; MLCR = 15.1 to 35 mils = range B; MLCR = 35.1 to 60 mils = range C, MLCR = 60.1 and higher = range D. The bars on the graph represent the total number of occurrences within the range for each of the reference solutions.

Note 2—It is necessary for each laboratory to develop their own data, with their own individual plate or plates, metal, operators, cleaning procedures, and environmental conditions to establish the ranges of MLCR calculated for the reference standards. The insulation or other test solutions are only evaluated against the reference solution results run at the same time.

FIG. 1 Uncertainty Test

C871 Test Methods for Chemical Analysis of Thermal Insulation Materials for Leachable Chloride, Fluoride, Silicate, and Sodium Ions
D609 Practice for Preparation of Cold-Rolled Steel Panels for Testing Paint, Varnish, Conversion Coatings, and Related Coating Products
G1 Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens
G16 Guide for Applying Statistics to Analysis of Corrosion Data
G31 Guide for Laboratory Immersion Corrosion Testing of Metals
G46 Guide for Examination and Evaluation of Pitting Corrosion

3. Terminology

3.1 *Definitions:* Refer to Terminology C168 for definitions relating to insulation.

4. Summary of Practice

4.1 The practice uses controlled amounts of test solutions delivered drip wise onto a defined area of small flat coupons of selected test metals for the purpose of producing, comparing, and measuring the corrosion that occurs on the metals due to the exposure.

4.2 The test is conducted at elevated temperatures, greatly accelerating the corrosion in comparison with corrosion at room temperature. The heat makes the solution evaporate quickly, allowing an air (oxygen) interface and making thousands of wet-dry-wet cycles possible in a short time.

4.3 Quantitative measurements of corrosion are determined from the weight change (loss) due to the corrosion of the tested coupons. Reference tests prepared with known concentrations of solutions that are conducive to the corrosion of the tested metal are compared with water solutions containing ions extracted from insulation samples. Calculations of MLCR in

TABLE 1 Mass Loss Corrosion Rate (MLCR) Calculated Using Practice G1 (see Section 12)

NOTE 1—MLCR expressed in mils per year.

0-ppm De-ionized Water	1-ppm Chloride Solution	5-ppm Chloride Solution	10-ppm Chloride Solution
19.02	35.17	57.31	62.61
11.68	29.87	40.91	56.48
14.04	33.00	66.76	110.54
12.13	37.91	52.46	131.35
12.45	29.80	16.53	52.27
14.42	22.72	42.51	35.42
6.13	35.42	76.33	67.01
13.27	31.78	111.82	57.48
21.25	17.04	42.19	98.92
7.59	37.78	44.42	132.35
12.83	32.55	53.61	61.52
6.70	36.12	54.25	36.42
16.08	25.66	41.87	90.44
19.02	14.93	54.50	95.48
11.42	31.08	65.67	63.44
14.81	34.21	70.46	99.63
9.38	34.46	42.57	69.63
18.38	36.06	63.44	107.28
8.62	27.38	50.10	58.84
8.49	24.19	48.63	65.10
12.13	15.25	55.40	64.27
5.36	33.70	69.12	71.29
4.66	32.10	39.06	78.37
5.55	35.04	43.21	88.52
6.57	22.98	41.93	30.57
5.87	39.44	36.76	39.25
7.21	35.04	25.66	50.93
6.45	34.66	30.06	128.41
3.45	41.48	41.68	97.52
2.30	41.55	29.61	98.03
11.93	42.70	38.74	82.84
9.19	33.32	38.10	105.31
13.15	28.98	33.00	96.50
14.10	21.38	58.27	84.50
12.25	16.08	39.31	59.55
12.25	17.17	40.78	45.57
9.96	32.42	48.25	56.80
4.60	34.72	23.10	63.63
3.70	34.02	27.19	67.01
2.43	33.38	35.61	48.82
3.32	25.66	77.16	75.76
1.21	33.12	30.76	48.95
1.28	44.04	42.57	
5.87	37.46	42.63	
7.15	23.36	41.61	
3.96	28.15	61.27	
11.23	25.02	27.76	
10.02	36.83	49.27	
10.28	21.64	67.65	
9.38	27.63	68.54	
12.25	18.51	42.44	
9.38		40.14	
		36.76	
		54.12	
		67.40	
Average and (Standard Deviation)			
9.5 (4.8)	30.5 (7.4)	48.0 (16.4)	74.6 (26.0)

mils-per-year (MPY) made using the methods of Practice G1 are reported as the quantitative measurement.

5. Significance and Use

5.1 Corrosion associated with insulation is an important concern for insulation manufacturers, specification writers, designers, contractors, users and operators of the equipment. Some material specifications contain test methods (or reference test methods contained in other material specifications), for use in evaluating the insulation with regard to the corrosion of steel, copper, and aluminum. In some cases these tests are not applicable or effective and have not been evaluated for precision and bias.

5.2 A properly selected, installed, and maintained insulation system will reduce the corrosion that often occurs on an un-insulated structure. However, when the protective weather-resistant covering of an insulation system fails, the conditions for the aqueous environment necessary for corrosion under insulation (CUI) often develop. It is possible the insulation contains, collects, or concentrates corrosive agents, or a combination thereof, often found in industrial and coastal environments. If water is not present, these electrolytes cannot migrate to the metal surface. The electrochemical reaction resulting in the aqueous corrosion of metal surfaces cannot take place in the absence of water and electrolytes. Additional environmental factors contributing to increased corrosion rates are oxygen, and elevated-temperature (near boiling point).

5.3 Chlorides and other corrosive ions are common to many environments. The primary corrosion preventative is to protect insulation and metal from contamination and moisture. Insulation covers, jackets, and metal coating of various kinds are often used to prevent water infiltration and contact with the metal.

5.4 This procedure can be used to evaluate all types of thermal insulation and fireproofing materials (industrial, commercial, residential, cryogenic, fire-resistive, insulating cement) manufactured using inorganic or organic materials, faced or unfaced, for which a filtered extraction solution can be obtained.

5.5 This procedure can be used with all metal types for which a coupon can be prepared such as mild steel, stainless steel, copper, or aluminum.

5.6 This procedure can also be applicable to insulation accessories including jacketing, covers, adhesives, cements, and binders associated with insulation and insulation products.

5.7 Heat treatment of the insulation (as recommended by the manufacturer up to the maximum potential exposure temperature) can be used to simulate possible conditions of use.

5.8 Adhesives can be tested by first drying followed by water extraction or by applying a known quantity of the test adhesive to a test piece of insulation and then extracting.

5.9 Insulating cements can be tested by casting a slab, drying, and extracting or by using the uncured insulating cement powder for extraction.

5.10 Reference tests prepared with various concentrations of solutions that are conducive to the corrosion of the tested metal serve as comparative standards. Solutions containing chloride, sodium hydroxide, various acids (sulfuric, hydrochloric, nitric, and citric acid), as well as "blank" tests using only de-ionized water and tap water are used.

5.11 Research can be done on insulation that has been specially formulated to inhibit corrosion in the presence of corrosive ions through modifications in basic composition or incorporation of certain chemical additives. Corrosive ions can also be added to the insulation extraction solutions to determine the effectiveness of any inhibitors present.

5.12 Protective surface treatments and coatings of different types and thickness can be applied to the metal coupons and compared using various corrosive liquids.

5.13 Several sets of tests are recommended because of the number of factors that affect corrosion. An average of the tests and the standard deviation between the test results are used on the data. Much of the corrosion literature recommends a minimum of three specimens for every test. Consult Guide G16 for additional statistical methods to apply to the corrosion data.

5.14 Results from this accelerated corrosion test shall not be considered as an indicator of the useful life of the metal equipment. Many factors need consideration for applicability to specific circumstances. Refer to Practice G31 for additional information.

6. Apparatus

6.1 The test apparatus must be housed in a reasonably clean and non-dusty environment to avoid any effects of contaminants.

6.2 *Heated Temperature Controlled Flat Hot Plate (see Appendix X1)*—A 1-ft (30.5-cm) square or circular plate that has uniform temperature across the surface provides the heated environment. See Appendix X1 for construct design and sources of assembled systems. Larger plates for testing more coupons are not excluded.

6.3 *Peristaltic Pump (see Appendix X1)*—A multi-channel peristaltic pump with individual cassettes and silicone tubes is recommended to supply 250 (±25) mL/day to each specimen.

6.4 *Silicone Rubber Tubing (see Appendix X1),* to deliver fluid to the test coupons.

6.5 *Miniature Barbed Fitting (see Appendix X1),* for connections of tubing (1/16 by 1/16 in.)(0.16 by 0.16 cm).

6.6 *Band Saw.*

6.7 *Balance,* capable of 0.0001 (±0.0002) g mass determination.

6.8 *Wet-Grinding Belt Grinder/Sander,* with used 80-grit (a belt previously used to make Test Method C692 stainless steel coupons is acceptable) or new 120-grit wet belt.

6.9 *Drying Oven.*

6.10 *Bottles,* plastic 1 L or equivalent, to individually supply each test specimen with test liquid.

6.11 *Nominal 1-in. Thin-wall PVC Pipe,* 1 5/16-in. (3.33 cm) OD; 1 3/16-in. (3.02 cm) ID by 1.25-in. (3.18 cm)lengths.

6.12 *High Temperature Grease or oil,* for use as heat transfer medium.

6.13 *Rubber O-Ring,* 1¼-in. (3.18 cm) ID, 1½-in. (3.81 cm) OD, ⅛-in. (0.32 cm)thick.

6.14 *Silicone Sealant,* 100% Silicone sealant.

6.15 *Plastic Straw,* ⅛-in. (0.32 cm) drink stirring straw (“swizzle stick”) .

6.16 *Cleaning Apparatus and Solutions,* for the coupons, stainless steel metal scourer pad, 3-M sanding pad (medium and fine) or equivalent sand paper, acetone, xylene, water, paper towels.

6.17 *Hand-Held Magnifier,* or 10 to 30× binocular microscope, or both.

6.18 *Filter,* 0.45 micron filter paper.

7. Reagents and Materials

7.1 *Distilled or De-Ionized Water,* containing less than 0.1 ppm chloride ions.

7.2 *Metal Test Coupons,* meeting the composition requirements of applicable ASTM Specification for Mild Steel, Stainless Steel, Copper, or Aluminum. Mill certificates of chemical composition and mechanical properties are required.

7.2.1 Some researchers will want to maintain traceability to the metals used in other C16 corrosion procedures. Specification C739 uses cold rolled, low carbon (<0.30 %) commercial quality shim steel. Specification C665 uses cold rolled, low carbon, quarter hard, temper No. 3, strip steel. It is possible other metal grades meeting Specification A53/A53M, Specification A105/A105M, and other common ferrous steel specifications are of interest for use in the tests. If stainless steel coupons are to be used, it is recommended that they be 16-gage and prepared following the sensitization procedure described in Test Method C692 Section 9 on Test Coupons (sensitize stainless steel coupons by heating at 1200°F (649°C) in an argon (inert) or air (oxidizing) atmosphere for three hours). Galvanized steel is not suitable for test because the elevated temperatures recommended by the practice are above the recommended use temperature of galvanized metal. However, with suitable adjustments to slow the drip rate and lower the temperature of the hot plate, there are possibilities for the development of test practices.

7.2.2 Carbon Steel Coupons ; style: 0.032 Steel, Type R, Dull Matte Finish. Specs: ASTM D609-Type 1, Temper = ¼ hard, Carbon = 0.13; size = 0.032 by 2 by 3.5in. (0.8 x 51 x 89 mm)

7.2.3 It is likely that different results will be found when switching between various metal grades. The use of standard solutions of corrosive ions provides a benchmark against which the leachable ions contained in the insulation are evaluated. The standard solutions are run during every test sequence, after having previously established the range of results for the individual laboratory and the type, grade, and lot of metal.

7.3 *Chemically Pure Salts and Reagent Grade Acids* shall be used for preparation of corrosion solutions used as reference standards for plate calibration and comparison with extraction solutions.

7.4 *Chloride Reference Standards* are prepared from a 1000 ppm (mg/L) chloride solution using 1.64 g of sodium chloride to one liter of de-ionized water. For a liter of a 1-mg/L chloride solution, mix 1 mL of 1000 ppm chloride solution to one liter. Quantity and concentration of the reference standards are made as needed for the desired test.

8. Metal Coupon and Test Cell Preparation

8.1 Carbon steel coupons referenced in 7.2.2 are used as received from the manufacturer.

NOTE 1—The previous coupon preparation technique has been moved to Appendix X3 (History).

8.2 Permanently mark each coupon for identification. If metal stamp impressions are used to mark the coupon, do not allow the impression to deform the back face of the coupon.

8.3 Heat the coupons to drive off surface moisture and obtain a constant weight. Cool the coupons in a moisture-free environment and weigh using a precision balance to 0.1 mg. Record the weight and coupon identification.

8.4 Cut the polyvinylchloride (PVC) pipe into 1.25in.(3.175 cm) lengths. Remove the ragged edges to make smooth flat-sanded ends. Drill a ⅛-in. hole in the side of the pipe, ⅛ in. from the top end and then clean the pipe in de-ionized water and dry.

8.5 Position an O-ring approximately 0.5 in. (91.5 cm) from a smooth flat-sanded end of the PVC pipe. Put a 0.125-in.(0.32 cm) bead of silicone sealant completely around the space formed by the pipe and O-ring. Position the pipe in the center of the coupon with the hole oriented to the corner for easy access. While tightly holding the pipe down, push the O-ring into contact with the coupon, squeezing out some silicone sealant to form a continuous, watertight seal. Avoid silicone sealant on the inside of the pipe and metal. Allow the silicone to cure completely (overnight) before testing.

8.6 Cut 1-in. (2.54 cm) pieces of the plastic straw with one end at a 45° angle. Insert the straw into the hole in the PVC pipe so that the angle is down and the drip falls in the approximate center of the coupon. The barbed fitting is used to attach the straw to the peristaltic pump tube. Fig. 2 shows a completed test coupon with the components labeled. Figs. 3 and 4 show a hot plate with the coupons installed.

9. Solution Preparation

9.1 *Procedure A:*

9.1.1 Many industrial insulation materials are required to meet the requirements of Specification C795 using Test Methods C692 and C871. If the material has been extracted for Test Method C871 testing, a suitable procedure is filtration of the concentrated extraction solution through a 0.45 micron filter followed by the dilution of the concentrated extraction solution with de-ionized water for use in this test. Refer to Test Method C871 for the details of the extraction. Briefly described, the procedure involves extracting duplicate ground-up samples of 20 g each in 450 g of boiling water for 30 min, adjusting the final solution weight to 500 g, and then filtering to remove the solids.

9.1.2 Combine 375 mL from each of the two extraction solutions described in 9.1.1 to provide a uniform 750-mL solution. Dilute 375 mL of the solution with 2625 mL of de-ionized water to total 3000 mL. One thousand millilitres of the resulting solution is used in a 4-day test for one metal coupon. The two extractions provide enough diluted solution for six coupon tests of four-day duration. The minimum recommended number of specimens per test set is three. Additional test sets are used to provide greater confidence in the results. The unused 125 mL from each of the extraction solutions are available for Test Method C871 or other chemical analysis.

9.2 *Procedure B:*

9.2.1 There are insulation materials that do not readily wick water, and cannot be made to wick by heat treatment. Some manufacturers consider it inappropriate to subject them to a severe leaching of soluble ions by Procedure A because it exposes a maximum surface area to water for extraction, which would not happen under ordinary conditions of use. An alternative extraction procedure is as follows:

9.2.2 Slice the material cross-sectionally on a band saw into 0.25-in. (0.64 cm) wide pieces. Cut enough slices so that the exposed surface area totals 2 ft^2 (1858 cm^2) . A 2-in.(5.08 cm) thick block sample would require 12 slices that are 5.11-in. (12.98 cm) long. A 1½-in. (3.81 cm) thick block sample would require 16 slices that are 4.93-in. (12.52 cm) long.

9.2.3 Record the weight of the slices.

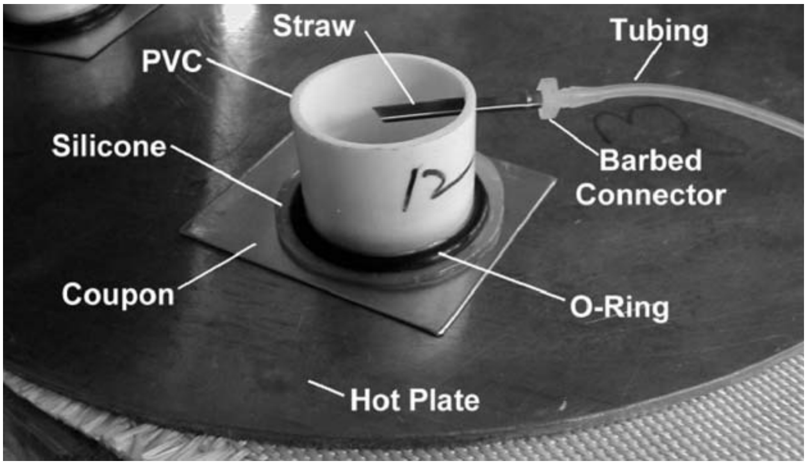


FIG. 2 Test Coupon with Components Identified

FIG. 3 Test Coupons on Hot Plate

FIG. 4 Test Cells on Hot Plate

9.2.4 Stack the slices using plastic spacers (flattened plastic stir-straws) between the slices, and secure the stack with rubber bands or monofilament fishing line.

9.2.5 Place the stack or stacks in the bottom of a suitable container. If the material floats, an appropriate means is necessary to weight the material so it remains submerged.

9.2.6 Pour in enough heated de-ionized water to cover the stack completely. If boiling water exceeds the desired extraction temperature, the manufacturer needs to specify the water temperature.

9.2.7 Agitate the contents 3 times over a 15-min period. After 15 min, filter the water though a Whatman number 41 filter or equivalent. Rinse the container and slices with de-ionized water. Record the total volume of water obtained from the extraction. Filter the extraction solution through a 0.45 micron filter.

9.2.8 Adjust the final volume to 3000 mL to test three coupons for four days.

9.3 *Reference Standards:*

9.3.1 The use of reference tests to compare the measured corrosion resulting from the insulation solutions to that of known corrosive solutions allows for a degree of calibration of the practice. Ideally the number of test coupons for each solution is three. Conduct the tests on the same plate at the same time as the insulation solutions.

9.3.2 The reference solutions for mild steel and copper coupons include de-ionized water and various solutions of chloride ranging from 1 to 5 mg/L and ideally bracket the corrosion found for the insulation coupons. The reference solutions for aluminum coupons include de-ionized water and various solutions of sodium hydroxide. Solutions that are more concentrated than 5 mg/L produce high corrosion and are better tested using reduced exposure times.

10. Test Procedure

10.1 *Test Plate Conditions:*

10.1.1 Start the heated plate previously tested and regulated to operate at 230°F (± 10°F) (100°C ±6°C) with water dripping into the test cells. The hot plate shall be maintained at this temperature throughout the test. It is important to establish this control prior to beginning tests for data collection. Variables influencing temperature control are: the individual heated plate, digital controller (when used), thermocouple position, and the insulation covering the thermocouple and coupons (when used). When any changes are made it is necessary to re-establish the temperature control of the test set-up. Temperature stability is improved by using thermal insulation on top of the coupons and between the cell tubes.

NOTE 2—Glass fiber felt, 0.5-in (1.5 cm) thick with an aluminum foil barrier to prevent heat transfer fluid uptake, and also EPDM-based elastomeric foam insulation have been successfully used for temperature control.

10.1.2 It is useful to test the evaporation rate of each coupon, especially on newly constructed plates, to verify that the coupons are being heated evenly. Start the peristaltic pump with the feed tubes in de-ionized water and allow the temperature controller to stabilize. Turn off the peristaltic pump and quickly fill all the test coupon cells with 1 mL of de-ionized water using an automatic pipette. Determine the time it takes for the first cell to evaporate the water (expect 2 to 3 min) and verify that the other cells dry within 45 s of the first. When necessary, reposition or otherwise adjust the coupons.

10.1.3 New plates are evaluated by performing a number of tests using only standard solutions to determine the range of the results for each standard solution. A Frequency Histogram similar to Fig. 1 is developed for the individual lab, test equipment, and metal used in the test. Guide G16 is helpful in analyzing the data.

10.1.4 A small fan used to circulate the air above the test apparatus is recommended to help the evaporation process by moving the air saturated with water.

10.2 *General Procedure:*

10.2.1 Place each coupon with the attached PVC tube on the flat plate using sufficient high temperature grease between the coupon and the plate to maintain good contact (no air space). Place temperature stability insulation on top of the coupons.

10.2.2 Fill the liquid reservoirs for the peristaltic pump with the test and standard reference solutions and attach the individual feed tubes to the barbs in the plastic stir-straws. Record the coupon identification and solution information.

10.2.3 Start the peristaltic pump previously calibrated to deliver 250 mL/day to each sample.

10.2.4 Monitor the reservoir bottles daily to ascertain that the delivery to each sample is 250 ± 25 mL/day. Refill if needed for longer duration tests.

10.2.5 At the conclusion of the test period (normally 96 h), carefully remove the coupons. In the event of a power outage, or a plugged tube during the test, add additional test time to the end of the test to allow the delivery of the full amount of liquid. Include this information in the final report.

11. Cleaning Coupons

11.1 The cleaning procedure is important to the accurate determination of the weight loss due to corrosion. The goal of any cleaning is to remove the corrosion product but minimize the loss of intact metal. The use of a weighed cleaning blank coupon, that is subjected to the same cleaning procedures but not otherwise tested, is necessary to determine of the weight of metal loss due to the specific cleaning procedures. Additional information about cleaning coupons after testing is written in Practice G1 Section 7 on Methods for Cleaning After Testing.

11.2 Remove the coupons and clean the heat transfer medium from the back of the coupon.

11.3 Remove the PVC cells leaving the O-ring and silicone intact when possible. At times the corrosion may be under the O-ring on highly corroded coupons. Carefully remove the O-ring while leaving the silicone intact under these circumstances. If there is loose rust or solids it is permitted to use a dental pick and water to remove any large pieces.

11.4 The following steps are for 2-3 coupons at a time and protective gloves and glasses shall be worn for the following steps. Put approximately 2 ml of 1:3 HCL (Dilute 125 ml of concentrated, 36.5-38%, HCl to 500 ml to make 1:3) in the coupon test area (within o-ring / silicone area circle only). Use a dental pick or similar instrument to gently scrape away and etch away the corrosion products. The technician shall pay close attention to the tested area and attempt to gently remove as much corrosion products as possible within any pits or groves. If necessary, the technician is permitted to scratch into the by-product to allow the HCL to react and remove the material. The HCL will become muddy during this process depending on the amount of corrosion present. The HCL stays on the tested area of the coupon for a total of 10 minutes even if the coupon is very clean.

11.5 Rinse under running tap water, if clean go to next step. If not, use a few more drops of HCL and work on any deep pits with the dental pick until clean. Most of the corrosion products will be removed in the previous step and only very corroded coupons with deep pits will require this extra cleaning step. Do not leave the HCL on the coupon for another 10 minutes, only long enough to remove any excess corrosion products.

11.6 Remove the O-ring and silicone using a razor blade widget. Do not gouge or remove metal. An additional optional procedure is to then soak the coupons in Xylene for a few minutes. This will swell the remaining silicone and more of it will be removed with the razor blade. Dry with a clean cloth.

11.7 Moisten fiberglass felt paper or similar material with 1:3 HCL and rub down both sides of the coupon. Pay particular attention to the area which contained the silicone to remove any trace amounts and to the tested area to remove all traces of corrosion products.

11.8 Rinse the coupon under running water and rub all surfaces with your fingers to remove trace HCL. Soak the coupon in diluted baking soda (~ 2 tablespoons in 500ml) to neutralize the HCL.

11.9 Dry and polish the coupons with a lint free cloth. Put in 180 degree oven to fully dry. Cool in a desiccator. Weigh and record the weight of the coupons.

12. Inspection of Coupons

12.1 Weigh using a precision balance to 0.1 mg. Record the weight and coupon identification. Calculate the average Mass Loss Corrosion Rate (MLCR) expressed in mils per year using Practice G1 Section 8 on Assessment of Corrosion Damage, as shown below.

$$\mathrm{MLCR} = \frac{(K \times W)}{(A \times T \times D)} \quad \text{mils per year} \qquad (1)$$

where:

K = 3.45×10^6 (a constant to convert cm/h to mils/year),
T = time of exposure in h,
A = area of exposure (cm^2); based on the inner radius (cm) of the plastic pipe: $A = \pi \times r^2$ (3.142 times radius of circle (squared)),
W = mass loss from the coupon (g) = (initial recorded weight of coupon) – (weight of coupon after exposure and cleaning), and
D = bulk density of the test metal (g/cm^3); carefully measure with a micrometer and weigh several stacks of coupons, average the results to obtain: D = weighed mass (g) / length × width × height (all cm).

12.1.1 *Example Calculations (for De-Ionized Water):*

$K = 3.45 \times 10^6$
T (h) = 96
$A = 3.142 \times (0.594\ \text{in.})^2 = 1.108\ \text{in.}^2 \times 6.452\ \text{cm}^2/\text{in.}^2 = 7.149\ \text{cm}^2$
W (de-ionized (DI) water) = 16.5971 – 16.5875 = 0.0096
D (mild steel) = 7.88 g/cm^3; D (aluminum) = 2.72 g/cm^3; D (copper) = 8.81 g/cm^3

12.1.1.1 *Mathematical Simplifications:*

$A \times T \times D$: mild steel = 5408.08, Al = 1866.75, Cu = 6046.34
Factor = ($K = 3.45 \times 10^6$) / ($A \times T \times D$): mild carbon steel = 637.93, Al = 1848.13, Cu = 570.59

12.1.1.2 *Measured Data:*

W (DI water) = 16.5971 – 16.5875 = 0.0096 × 637.93 (factor mild steel) = 6.124 mils per year
MLCR = 6.124 (mils per year)

12.2 Rate and categorize the MLCR of the insulation or other test solutions against the MLCR of standard reference tests to determine an estimate of the corrosiveness of the soluble ions contained in the solution. Utilize data collected from a number of test sets to develop a chart similar to Fig. 1. The data shown in Table 1 are standard reference tests used to create the Fig. 1 bar graph. It is necessary for each laboratory to develop their own data, with their own individual plate or plates, metal, operators, cleaning procedures, and environmental conditions to establish the ranges of MLCR calculated for the reference standards. The insulation or other test solutions are then evaluated against the reference solution results. An example rating criteria for estimating corrosiveness is: MLCR = 0 to 15 mils = range A; MLCR = 15.1 to 35 mils = range B; MLCR = 35.1 to 60 mils = range C, MLCR = 60.1 and higher = range D. The rating criteria were developed to accommodate the results obtained using this practice on reference standards and experimental insulation samples tested on mild carbon steel coupons.

12.3 Individual laboratories working in conjunction with material manufacturers, specification writers, designers, contractors, and operators of the equipment are encouraged to develop and share their own rating criteria through testing, experimentation, refinement, and experience, for particular insulation types, metal classification, and metal types.

12.4 Visually examine test coupons under good lighting for evidence of corrosion. Personnel involved in the inspection shall demonstrate natural or corrected near distance vision acuity of 20/25 or greater Snellen fraction with at least one eye and be trained for corrosion detection. Examine all cleaned coupons using 10 to 30× magnification for areas of pitting and corrosion and characterize using Practices G1 and G46.

12.5 Guide G16 is helpful for guidance.

12.6 Stainless steel coupons are inspected and evaluated for corrosion cracking using the procedures of Test Method C692 Section 14 on Inspection of Coupons.

13. Report

13.1 Report the following information:

13.2 *Insulation Solution:*

13.2.1 Manufacturer, product name, lot number, type, size, density, and other identifying information for insulation used to prepare the solution.

13.2.2 Method used to prepare the solution (A or B). Indicate the quantity and temperature of the water, and the length of time of the extraction.

13.2.3 The weight of the insulation extracted and final volume of the extraction solution.

13.2.3.1 The volume of this extraction solution used and the volume of de-ionized water used to make the final test solution.

13.2.4 Information on heat treatment or other special treatment of the insulation.

13.2.5 Information on the reference solutions tested for comparison.

13.3 *Metal:*

13.3.1 Type or types of metal used (mild steel, copper, aluminum, stainless steel) including ASTM classification and any mill certificate of chemical composition.

13.3.2 Provide information on treatment of the metal surface and surface coatings if used.

13.4 *Results:*

13.4.1 Number of specimens tested, and the time period exposed.

13.4.2 Describe the severity of corrosion (density, size, and depth) in the test area of each coupon using procedures in Practices G46 and G1 for guidance.

13.4.3 Report the actual mass loss and calculated average MLCR in "mils per year" for the test coupons, the reference test coupons and the cleaning blank coupon using 12.1.

13.4.4 Rate the insulation results against the reference tests for an estimate of the corrosiveness of the leachable ions following the guidelines of 12.2 or 12.3 or both.

13.5 *Optional:*

13.5.1 Chemical analysis data run in accord with Test Method C871.

13.5.2 Report other statistical and graphical representations of the data, such as pictures, maximum pit depth, and precise area within the test exposure area where corrosion occurred, where obtainable and desirable for the particular investigation.

14. Precision and Bias

14.1 Appendix X2 provides a discussion and review of controlled and uncontrolled variables. Table 1 is a summary of all individual standard reference coupons tested using the general guidelines of this practice. The tests were conducted over a 2-year period by a single lab using three heating plates and two operators. The precision and bias results are required within five years of document publication.

14.2 *Precision*—The precision of the procedure in Practice C1617 for measuring corrosion is being determined and will be available on or before 2010. This practice is not necessarily suitable for use in specifications or in case of disputed results as long as these data are unavailable. Contact the Chair, Committee C16, ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428–2959 in order to participate in the development of precision and bias statements through interlaboratory testing.

15. Keywords

15.1 chloride; corrosion; corrosion under insulation; inhibition; metal; protective coatings; steel; thermal insulation

APPENDIXES

(Nonmandatory Information)

X1. ACCESSORIES

X1.1 Hot Plates

X1.1.1 A commercial hot plate with ⅛-in. thick copper plate on surface to provide uniform surface temperature has been used by a lab with mixed results.

X1.1.2 Test Method C518 hot plate with ⅛-in. thick copper plate on surface to provide uniform surface temperatures has been used by a lab with mixed results.

X1.1.3 A flat plate heater was topped with a 1-in. thick aluminum plate and a ⅛-in. thick copper plate to achieve equal heat distribution at the surface. The perimeter of the plate is insulated. See Fig. X1.1.

X1.1.4 Temperature control is obtained with a digital PID temperature controller with the control thermocouple mounted on the top surface, covered by a small section of ceramic wool.

X1.2 Peristaltic Pump and Accessories

X1.2.1 A multi-channel peristaltic pump with silicone rubber tubing and barbed fittings must be used to deliver test liquid at the rate of 250 mL/day (±10 %) to each coupon.

FIG. X1.1 A One-Foot Square Flat Plate Heater

X2. RUGGEDNESS VARIABLES

X2.1 Uncontrolled Variables

X2.1.1 *Atmosphere Within Laboratory*—Humidity and air temperature of the room are normal for personnel comfort but vary with location, season, and time of day. A fan is helpful in circulating the air above the cells and reducing the localized humidity from the water evaporating from the cells. Elevation and atmospheric pressure will also influence the boiling temperature and evaporation rate from the cells.

X2.1.2 *Rate of Evaporation from Coupon*—While directly related to the hot plate temperature and delivery rate of solution, which are controlled, the time that the coupon remains wet can be different in each cell because of the way the solution spreads out and the atmosphere within the laboratory.

X2.1.3 *Distribution of Localized and General Corrosion Pattern*—The method calculates all metal loss as general corrosion, that is, having occurred evenly over the entire exposed area. However, the actual corrosion is almost entirely localized, with deeply pitted areas and edge effects accounting for most of the metal loss within the exposed area. The effect is more pronounced when strong corrosive agents generate significant metal loss.

X2.1.4 *Change in Corrosion Rate*—The method assumes the rate to be constant over the test period and then extrapolates that rate to calculate one year of corrosion reported as mil per year. A shorter duration test for strong corrosive agents will help minimize the changes that occur as the products of corrosion and the concentration of ions from the solutions build up.

X2.1.5 *Different Metal Qualities and Variables Between or Within a Lot*—The metal meets the ASTM specification requirements but there are possible localized variances within the sheet of metal the coupons are cut from.

X2.2 Controlled Variables

X2.2.1 *Temperature of Hot Plate During the Test*—A specific set point is provided but it is acceptable to raise or lower it slightly to adjust for local conditions and include the information in the report.

X2.2.2 *Duration of the Test*—The test is designed to run for four days but it is acceptable for different control or insulation solutions to be tested for longer or shorter duration if needed.

X2.2.3 *Rate and Volume*—The amount of solution used over a specific time period is controlled and reported. The described equipment will deliver approximately 0.17 mL as 5 drops per minute. It is possible to change the rate that the solution is delivered. This will influence the evaporation rate and the time that the cell is wet and dry. The use of intermittent delivery to allow larger volumes of solution to collect in the cell and then pausing while the solution evaporates is an option to be investigated. This option will cover the exposed area completely and is expected to result in less localized corrosion.

X2.2.4 *Solution Concentrations*—The standard reference solutions are made from chemically pure reagent grade materials and de-ionized water. Standard laboratory practice will allow different laboratories to make identical solution to test and establish a basis for comparison to the insulation solutions. The insulation solutions are prepared using specifically described extraction and dilution procedures.

X2.2.5 *Cell Tubes*—The material and height of the cell tubes are specifically described. A test using Lucite tubes rather than PVC tubes showed no significant difference. This reinforces the conclusion, based on the low corrosion of the "blank" de-ionized water tests, that the chloride content of the PVC did not contribute to the corrosion rate because it does not leach from the cell tube. The height of the tube and initial tests using capped tubes showed that condensation for the evaporating solution will re-enter the cell and change the overall time the cell is wet and will affect corrosion.

X2.2.6 *Cleaning*—Mechanical methods have been used with good success on bare coupons and the cleaning materials used are described. Chemical methods following the guidance of Practice G1 section 7.2 are necessary if the purpose of the test is to check metal coating, as mechanical methods will unintentionally remove the coating. Chemical means of cleaning bare steel coupons increased the measured weight loss but made cleaning much easier.

X3. SHORT HISTORY OF THE TASK GROUP

X3.1 The task group was formed to investigate various approaches to obtaining a non-subjective, numeric value for judging the potential contribution from thermal insulation materials to the corrosion of metal. It is suspected that an important motivation to beginning this work was corrosion resulting from chemical fire retardant treatments that were used during in the late 1970s. Several analytical approaches were tried, including electrochemical evaluation. The results from these tests and a Round Robin interlaboratory study were unsatisfactory and the approach was discontinued.

X3.2 A test procedure referred to as the Stansbury test was investigated and advanced to a Round Robin interlaboratory study. Metal coupons were sandwiched between insulation samples in a water-tight container. De-ionized water was added to partially immerse the metal. The procedure attempted to measure the pit depth and categorize the general corrosion on metal coupons at the metal-air-wet insulation interface. The results from the Round Robin interlaboratory study were inconsistent and the corrosion depressions were too small for accurate measurement. The approach was discontinued.

X3.3 A test protocol developed by Tutco Scientific Inc. and referred to as the Tutco Accelerated Corrosion Test (TACT) was started in 1998 after the unsatisfactory round robin trials of the Stansbury procedure. TACT was further refined, expanded, and tested with the goal of developing an ASTM test method. The document was written as this practice to provide greater latitude to describe multiple sample preparation and evaluation criteria. This will allow different material standards and specifications to specify exactly how the tests are to be run within the framework of the practice.

X3.4 A major problem faced over the years in the development of a non-subjective measurement is that corrosion is not a well-behaved physical phenomenon that acts consistently and predictably every time. The ASTM G01 Committee on Corrosion of Metals suggests it is usually necessary to run a large number of tests to develop a meaningful statistical data base for comparison and measurement. This requires a large amount of work by the primary investigators and others in support of the method and to conduct Round Robin interlaboratory studies.

X3.5 It is important to be aware that accelerated test methods usually represent a worst case scenario and are not normally useful for predicting the life expectancy of equipment or actual rate of corrosion that will be found in real life situations. These methods are primarily useful for comparative and qualitative evaluations of the tested materials. It is difficult to correlate the laboratory result with the in-service situations.

X3.6 The uncertainty tests for Table 1 reported in this document were done using coupons prepared using the following procedures. Future testing will be done using commercial corrosion coupons.

X3.6.1 Shear 2 by 7-in. (51 by 178-mm) coupons from the test metal, with the long dimension parallel to the long dimension of the sheet.

X3.6.2 Grip coupon with suction cup holder (see Fig. 1 of Test Method C692) or other means to facilitate wet grinding on belt grinder. Grind parallel to the long dimension of the coupon using a well-used 80-grit (a belt previously used to make Test Method C692 stainless steel coupons is acceptable) or new 120-grit wet belt with just enough pressure to remove the dull finish and leave the metal bright. The degree of surface roughness is expected to affect the test, do not over-grind. A reference coupon or comparative roughness standard is useful. The belt-ground face is the test surface. Immediately rinse in de-ionized water and dry with a clean paper towel to prevent flash corrosion.

X3.6.3 Cut the coupon into approximately three equal pieces using a band saw equipped with a metal cutting blade.

X3.6.4 Prepare the edges of the coupons with a sander to produce smooth, even, and flat surfaces.

X3.6.5 Clean the surface to be tested by lightly wet sanding with a fine sanding pad in distilled or de-ionized water. Wet sand the back surface of the coupon to establish a clean condition that can be reproduced after testing. Rinse in distilled or de-ionized water, followed by rinsing in acetone. Dry and polish the test surface using a clean paper towel. Do not touch the test surface with bare hands thereafter.