Designation: D 6676 – 01$^{\epsilon 1}$

Standard Test Method for Cathodic Disbonding of Exterior Pipeline Coatings at Elevated Temperatures Using Interior Heating[1]

This standard is issued under the fixed designation D 6676; 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.

ε[1] NOTE—Editorial changes were made throughout in October 2001.

1. Scope

1.1 This test method describes an accelerated procedure for determining comparative characteristics of coating systems applied to the exterior of steel pipe for the purpose of preventing or mitigating corrosion that may occur in underground or immersion where the pipe is carrying heated media and is under cathodic protection. This test method is intended for use with samples of coated pipe, or with a specimen cut from the section of coated pipe or flat plates, and is applicable to such samples when the coating is characterized by function as an electrical barrier.

1.2 This test method is intended to simulate conditions when external coatings are exposed to high temperature inside the pipe and to an ambient temperature outside, and thus are subjected to temperature gradient. If elevated temperatures are not required, see Test Method G 8. If a specific test method is required with no options, see Test Method G 80. If elevated temperatures are required but without temperature gradient, see Test Method G 42.

1.3 The values stated in SI units to three significant decimals are to be regarded as the standard. The values given in parentheses are for information only.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

G 8 Test Methods for Cathodic Disbonding of Pipeline Coatings[2]

G 12 Test Method for Nondestructive Measurement of Film Thickness of Pipeline Coatings on Steel[2]

G 42 Test Method for Cathodic Disbonding of Pipeline Coatings Subjected to Elevated Temperatures[2]

G 62 Test Method for Holiday Detection in Pipeline Coatings[2]

G 80 Test Method for Specific Cathodic Disbonding of Pipeline Coatings[2]

G 95 Test Method for Cathodic Disbondment Test of Pipeline Coatings (Attached Cell Method)[2]

3. Summary of Test Method

3.1 The test method described, subjects the coating on the test specimen to electrical stress in a highly conductive alkaline electrolyte. Electrical stress is obtained from an impressed direct- current system. An intentional holiday is to be made in the coating prior to starting of test.

3.1.1 Electrical instrumentation is provided for measuring the current and the potential throughout the test cycle. At the conclusion of the test period, the test specimen is physically examined.

3.1.2 Physical examination is conducted by comparing the extent of loosened or disbonded coating at the intentional holiday in the immersed area with extent of loosened or disbonded coating at a reference holiday made in the coating in an area that was not immersed.

3.1.3 The cathodic stress is applied under conditions of a constant temperature gradient, simulating a heated pipeline with an exterior coating.

3.1.4 Specimens that can be used are: (*a*) piece of pipe (Fig. 1) or (*b*) samples cut from pipe or flat plate (Fig. 2 and Fig. 3).

3.1.4.1 Some coatings rely on application tension (such as tape) for maximum cathodic disbondment resistance. Cut coupons or flat plates must not be used.

4. Significance and Use

4.1 Damage to a pipe coating is almost unavoidable during transportation and construction. Breaks or holidays in pipe coatings may expose the pipe to possible corrosion since, after a pipe has been installed underground, the surrounding earth will be moisture-bearing and will constitute an effective electrolyte. Applied cathodic protection potentials may cause

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[1] This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.48 on Durability of Pipeline Coatings and Linings.

Current edition approved May 10, 2001. Published July 2001.

[2] *Annual Book of ASTM Standards*, Vol 06.02.

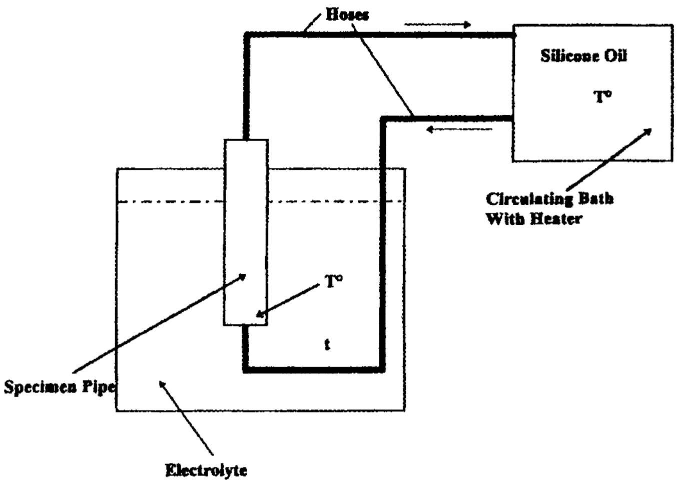

FIG. 1 Pipe Specimen Heated Inside

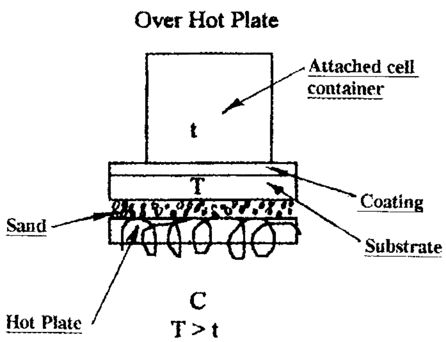

FIG. 2 Flat Specimen Heated Over Hot Plate

loosening of the coating, beginning at holiday edges. Spontaneous holidays may also be caused by such potentials. Usually exterior pipeline coatings applied over pipes carrying hot media (oil, gas) are exposed to high temperature inside the pipe and low temperature outside and subjected to temperature gradient. Heat flux is directed from metal (substrate) to the coating. This test method provides accelerated conditions for cathodic disbondment to occur under simulated heating and provides a measure of resistance of coatings to this type of action.

4.2 The effects of the test are to be evaluated by physical examinations and monitoring the current drawn by the test specimens. Usually there is no correlation between the two methods of evaluation, but both methods are significant. Physical examination consists of assessing the effective contact of the coating with the metal surface in terms of observed differences in the relative adhesive bond. It is usually found that the cathodically disbonded area propagates from an area where adhesion is zero to an area where adhesion reaches the original level. An intermediate zone of decreased adhesion may also be present.

4.3 Assumptions associated with test results include:

4.3.1 Maximum adhesion, or bond, is found in the coating that was not immersed in the test liquid, and

4.3.2 Decreased adhesion in the immersed test area is the result of cathodic disbondment.

4.4 Ability to resist disbondment is a desired quality on a comparative basis, but disbondment in this test method is not necessarily an adverse indication of coating performance. The virtue of this test method is that all dielectric-type coatings now in common use will disbond to some degree, thus providing a means of comparing one coating to another.

4.5 The amount of current flowing in the test cell is a relative indicator of the extent of areas requiring protection against corrosion; however, the current density appearing in this test is much greater than that usually required for cathodic protection in natural, inland soil environments.

4.6 Test voltages higher than those recommended may result in the formation of chlorine gas. The subsequent chemical effects on the coating could cast doubt on the interpretation of the test results. Filter tube with fritted disk (see Test Method G 95) or layer of sand (40 mesh) put on the coated surface may reduce this effect.

5. Apparatus

5.1 *Test Vessel for Pipe Specimen (Fig. 4)*—A suitable nonreactive vessel shall be used, capable of withstanding internal heating at test temperature and suitable to accommodate a test specimen, an anode. Heating the test sample can be provided by internally heating. The pipe sample may be filled with a suitable heat transfer material (oil, steel, shot, sand, copper chips, etc.) A thermocouple or thermometer and heater can be immersed in the heat transfer medium to effectively control the temperature of the sample. Dimensions of the vessel shall permit the following requirements:

5.1.1 Test specimen shall be suspended vertically in the vessel with at least 25 mm (1 in.) clearance from the bottom.

5.1.2 Test specimen shall be separated by not less than 38 mm (1½ in.), and a vertically suspended anode can be placed at an equal distance from each specimen not less than the separation distance.

5.1.3 Test specimen shall be separated from any wall of the vessel by not less than 13 mm (½in.).

5.1.4 Depth of electrolyte shall permit the test length of the specimen to be immersed as required in 7.4.

5.1.5 The reference electrode may be placed anywhere in the vessel, provided it is separated from the specimen and from the anode by not less than 38 mm (1½ in.).

5.2 *Test Vessel for Flat or Cut From Pipe Specimens (Fig. 3)*—A transparent plastic of glass tube that is centered over the intentional holiday and sealed to the test sample surface with a waterproof sealing material. The cylinder is to be 101.6 mm (4.0 in. nominal diameter) and of sufficient height to contain 127.0 mm (5.0 in.) of electrolyte.

5.3 *Impressed-Current Anode*—Anode shall be of the platinum wire type, 0.51 mm (0.020 in.)–24 gage diameter. It shall be of sufficient length to extend outside the confines of the test cell and shall be connected to the wire from the power source with a bolted or compressed fitting.

5.4 *Anode Assembly*—Anode shall be suspended inside the test vessel so that the tip of the anode assembly closest to the holiday is 25.4 mm (1 in.) above, and the edge of the anode assembly is 12.7 mm (½ in.) above, and the edge of the anode assembly is 12.7 mm (½in.) offset from the holiday.

5.5 *Reference Electrode*—Saturated Cu-$CuSO_4$ of conventional glass or plastic tube with porous plug construction, preferably not over 19.05 mm (0.750) in.) in diameter, having

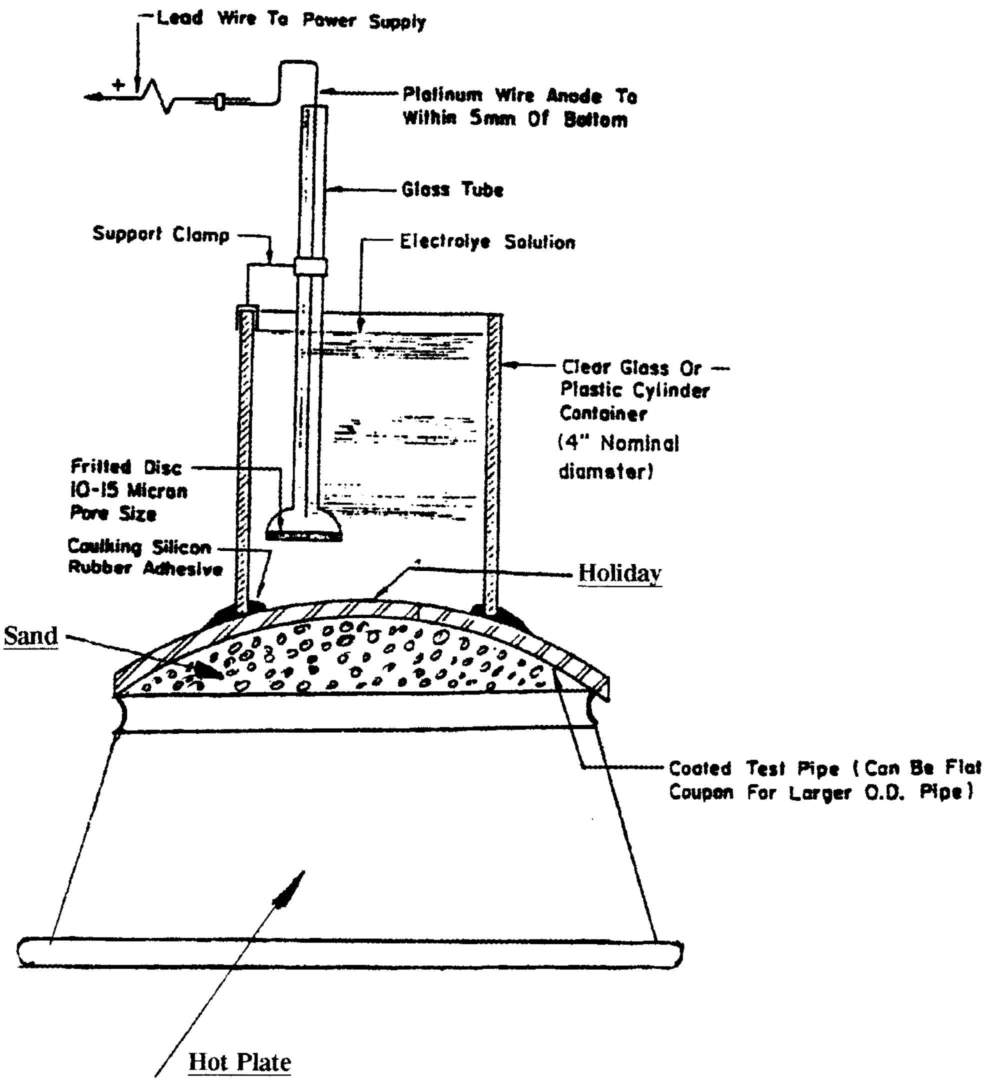

FIG. 3 Test Set-Up for Cathodic Disbonding Test with Coated Pipe Coupon (or Flat Coupon) Heated Over Hot Plate

a potential of -0.316 V with respect to the standard hydrogen electrode. A saturated calomel electrode may be used, but measurements made with it shall be converted to the Cu-$CuSO_4$ reference for reporting by adding -0.072 V to the observed reading.

5.6 *Reference Electrode Placement*—Submerge the tip of the reference electrode 25.4 mm (1 in.) into the electrolyte.

5.7 *High-Impedance Multimeter*—For making direct current and voltage measurements. Multimeter must have an internal resistance of not less than 10 MΩ and be capable of measuring as low as 10 μ V potential drop across a shunt in the test cell circuit, and voltage up to 10 V.

5.8 *Direct-Current Power Supply*—Capable of supplying low-ripple voltage at 1.5, ± 0.01, V, as measured between the test specimen and reference electrode.

5.9 *Precision Wire-Wound Resistor*—1 Ω ± 1 %, 1-W (minimum) to be used in the test cell circuit as a shunt for measuring current.

5.10 *Thickness Gage*—for measuring coating thickness in accordance with Test Method G 12.

5.11 *Holiday Detector*—for locating holidays in the coating of the test specimen in accordance with Test Methods G 62.

5.12 *Connections*—Wiring from current source to the specimen shall be by either soldering, brazing, or bolting to the non-immersed area of the specimen. A junction in the connection wire is not desirable but, if necessary, may be made by means of a bolted pair of terminal lugs, soldering or mechanically crimping to clean wire ends.

5.13 *Additional Connecting Wires*—If additional wiring is necessary, it shall be stranded, insulated copper and not less than 1.75 mm (0.069 in.–15 gage) diameter.

5.14 *Holiday Tools*—A drill and a suitable drill bit that will accomplish drilling of test hole, as described in 9.2.

5.15 *Heaters*:

5.15.1 *Pipe Specimens—Liquid Heat Exchange Medium*—Circulating bath with built-in heater shall be used for heating the heat transfer medium (silicone oil or other) to produce and control temperature of 80° ± 1°C (or other temperature as specified) inside of the coated pipe samples connected with the bath by the hoses.

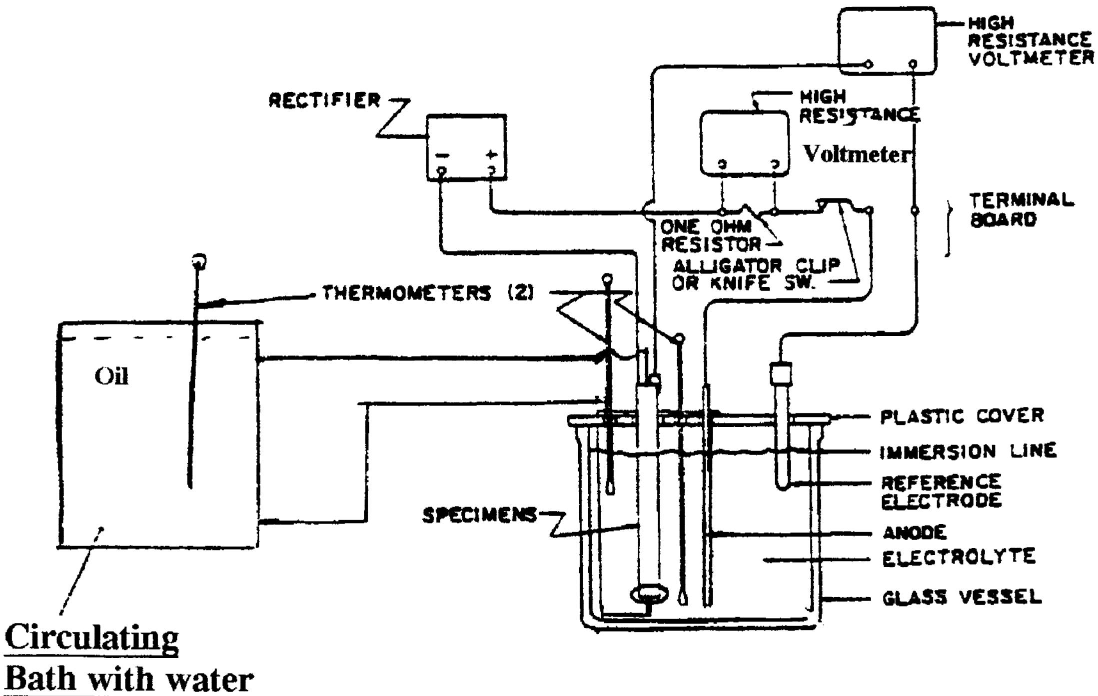

FIG. 4 Test Set-Up for Cathodic Disbonding Test at Elevated Temperature

5.15.2 *Pipe Specimens—Solid Heat Exchange Medium*—Heaters suitable for controlling temperature at 80° ± 1°C or as otherwise specified shall be employed inside the coated pipe.

5.15.3 *Flat Plates*—Heater plate, or equivalent, shall be used for heating coated samples. The heater shall be adjustable to produce and control a temperature of 80° ± 1°C or other specified temperature on the uncoated surface of testing panel.

5.16 *Thermometers or Thermocouples,* to measure temperature of heat transfer medium or on the uncoated surface of panel over hot plate and in electrolyte.

5.17 *Sharp-Pointed Knife*, with a safe handle is required for use in making physical examination.

6. Reagents and Materials

6.1 *Potable Tap Water or Higher Purity Water* (distilled or demineralized water is satisfactory) with 1 weight percent of each of the following technical-grade salts, calculated on an anhydrous basis: sodium chloride, sodium sulfate, and sodium carbonate.

NOTE 1—The resulting solution has a pH of 10 or higher and a resistivity of 25 to 50 Ω·CM at room temperature.

6.2 *Materials*, bituminous products, wax, epoxy, or other materials, including molded elastomeric or plastic end caps, capable of withstanding the test temperature, for sealing the ends of coated pipe specimens.

6.3 *Plywood*, has been found suitable for the construction of nonconductive test vessel covers and for the support through apertures of test specimens and electrodes. Wood dowels introduced through holes in the top ends of test specimens have been found suitable for suspending test specimens from the vessel cover.

6.4 *Hoses*, capable of withstanding the test temperature of 80° ± 1°C for connection of the plastic end caps in pipe specimens with circulating bath.

6.5 *Sand*, (40 mesh), to be used over hot plate for more consistent better uniform heating of flat panels.

7. Test Specimen (Coated Pipes)

7.1 The test specimen shall be a representative piece of coated pipe. Each end shall be plugged, sealed, or capped with inlet and outlet nozzles connected with circulating bath.

7.2 One holiday shall be made in the middle of the immersed length by drilling a radial hole through the coating so that the angular cone point of the drill will fully enter the steel where the cylindrical portion of the drill meets the steel surface. The drill diameter shall be not less than three times the coating thickness, but it shall never be less than 6 mm (¼ in.) in diameter. The steel wall of the pipe shall not be perforated. With small diameter pipes, where there is danger of perforating the pipe, the holiday shall be started with a standard 60° cone point and finished with a drill that has had a substantial portion of the cone point ground away.

NOTE 2—Before making the holiday, see 7.5.

7.3 The end of the pipe, which will protrude above the immersion line, shall be provided with suitable supporting means and a separate wire connection for electrical purposes, soldered, or brazed to the pipe. The protruding end, including hanger and wire connections, shall be protected and sealed with an insulating coating material.

7.4 The specimen test area shall consist of the area between the edge of the bottom end seal and the immersion line. The bottom end seal area shall not be considered part of the area tested. Coated specimens of any suitable diameter and length of pipe may be used, but the immersed area shall not be less than 23 200 mm^2 (36 in.^{2}). An area of 92 900 mm^2 (1 ft^2) has been found preferable when convenient.

7.5 The continuity of the coating and efficiency of the end seal shall be tested before making artificial holidays (see 9.1).

8. Test Specimen (for Flat Plates)

8.1 During the coating operation, the applicator may be directed to cut with torch or saw, a section of coated pipe of sufficient length for laboratory evaluation of the coating. Precautions are to be taken when cutting the pipe so that spatter will not harm the coating where testing will be done. Wet rags shall be placed on each side of the torch-cut area to minimize thermal changes and spatter damage to the coating. Test sample shall be taken at least 76.2 mm (3 in.) from any torch-cut edge. Special precautions are to be taken so that coating is not damaged in handling, transporting, or further cutting.

8.2 Flat plate 152 by 152 by 6 mm (6 by 6 by ¼ in.) may be used as a test specimen to simulate pipe with large outside diameter or for screening testing.

8.3 One intentional holiday shall be made in each specimen to be tested. The holiday shall be drilled so that the angular cone point of the drill will fully enter the steel where the cylindrical portion of the drill meets the steel surface. The hole shall be 6 mm (¼ in.) in diameter.

9. Procedure

9.1 Verify the coating integrity in the area to be tested in accordance with Test Methods G 62. Discard specimen found to contain holidays. Measure and record the maximum and minimum coating thickness measured in the area subjected to test in accordance with Test Method G 12.

9.2 Make electrical connections to the test specimen (Fig. 5).

9.3 Assemble testing vessel and specimen.

9.3.1 *For Pipe Specimens*—connect coated pipes with the hoses to the circulating bath and install pipe in the vessel.

9.3.2 *For Flat or Cut from Pipe Specimens*—Attach the test cylinder to the test specimen. Take care to ensure that the cylinder satisfactorily fits the curvature of the pipe sample and that it is centered over the intentional holiday.

9.3.3 Add the electrolyte solution to the specified level in the test vessel. Record the pH of the solution. Check the level of the electrolyte each day and maintain by adding preheated distilled or demineralized water as required. Do not add more of the original solution with salts because this will increase significantly the electrolyte salt concentration.

9.4 Insert the platinum anode into the vessel.

9.5 Attach the positive lead of the current source to the platinum anode and the negative lead to the cathode specimen. Adjust the rectifier or voltage divider so that the potential between specimen and reference cell is -1.50 ± 0.01 V (see Fig. 5). Position the holiday to face toward the anode. Space the anode with respect to test specimens as required in 5.1.

9.6 *Heating*:

9.6.1 *For Pipe Specimens*—Maintain specified temperature inside the pipe by circulating liquid heat transfer medium from circulating heating bath or by using solid heat exchange medium and internal heaters.

9.6.2 *For Flat or Cut from Pipe Specimens*—Install specimens over hot plate or another heating element covered with sand and heated to specified temperature; measure temperature under the uncoated side of specimen.

9.6.3 The testing temperature shall be not less than 80° ± 1°C) unless otherwise specified.

9.7 Conduct the test at a specified temperature (not less than 80° ± 1°C) for a period of 30 days unless otherwise specified.

9.8 *Electrical Monitoring Schedule*:

9.8.1 Electrical measurements shall be made on the start-up day and on each normal working day thereafter for the duration of the test. A maximum of three consecutive nonworking days shall be proceeded by at least two working days: one nonworking day or two consecutive nonworking days shall be preceded by at least one working day, except at start-up and termination when three and two working days are required, respectively.

9.8.2 Electrical measurements characterizing the start of the test are defined as the average of measurements taken on the second and third days after immersion.

9.8.3 Electrical measurements characterizing intermediate and terminal time spans shall be taken on two successive days prior to and including the target date.

9.9 *Electrical Measurements and Adjustments made Each Normal Working Day*:

9.9.1 Measure E_2, the stress potential in volts between the test specimen and the reference electrode, without disconnecting the energized anode or specimen from the circuit. Use a high resistance voltmeter described in 5.7. The stress potential, E_2, should measure -1.50 ± 0.01 V; if it does not, adjust the rectifier or voltage divider accordingly.

9.9.2 After E_2 has been measured and adjusted, if necessary, measure I_1, the current demand in amperes, by determining the potential drop across the 1-Ω precision resistor permanently installed in the test cell circuit with the high resistance voltmeter described in 5.7. The voltage reading will be numerically equal to amperes.

NOTE 3—An alternative method of measuring current demand uses a zero-resistance ammeter. In this method, the wire connection between the test specimen and anode is temporarily broken and a zero-resistance ammeter temporarily interposed between the specimen and the anode. Reconnect the specimen to the anode with the connector wire as soon as this measurement is completed.

9.9.3 Measure E_1, the polarized potential, in volts, using the high-resistance voltmeter described in 5.7 connected between the test specimen and the reference electrode as follows:

9.9.3.1 Disconnect the anode from the test specimen while closely observing the high-resistance voltmeter. As the instrument pointer falls, it will dwell significantly at the polarized potential before recording further. The dwell point is E_1.

10. Examination

10.1 At the end of the test period, disassemble the cell and rinse the test area with warm tap water. Adjust the temperature of tap water so that it approximates that of the room temperature. Immediately wipe the sample dry and visually examine the entire test area for any evidence of unintentional holidays and loosening of coating at the edge of all holidays, including

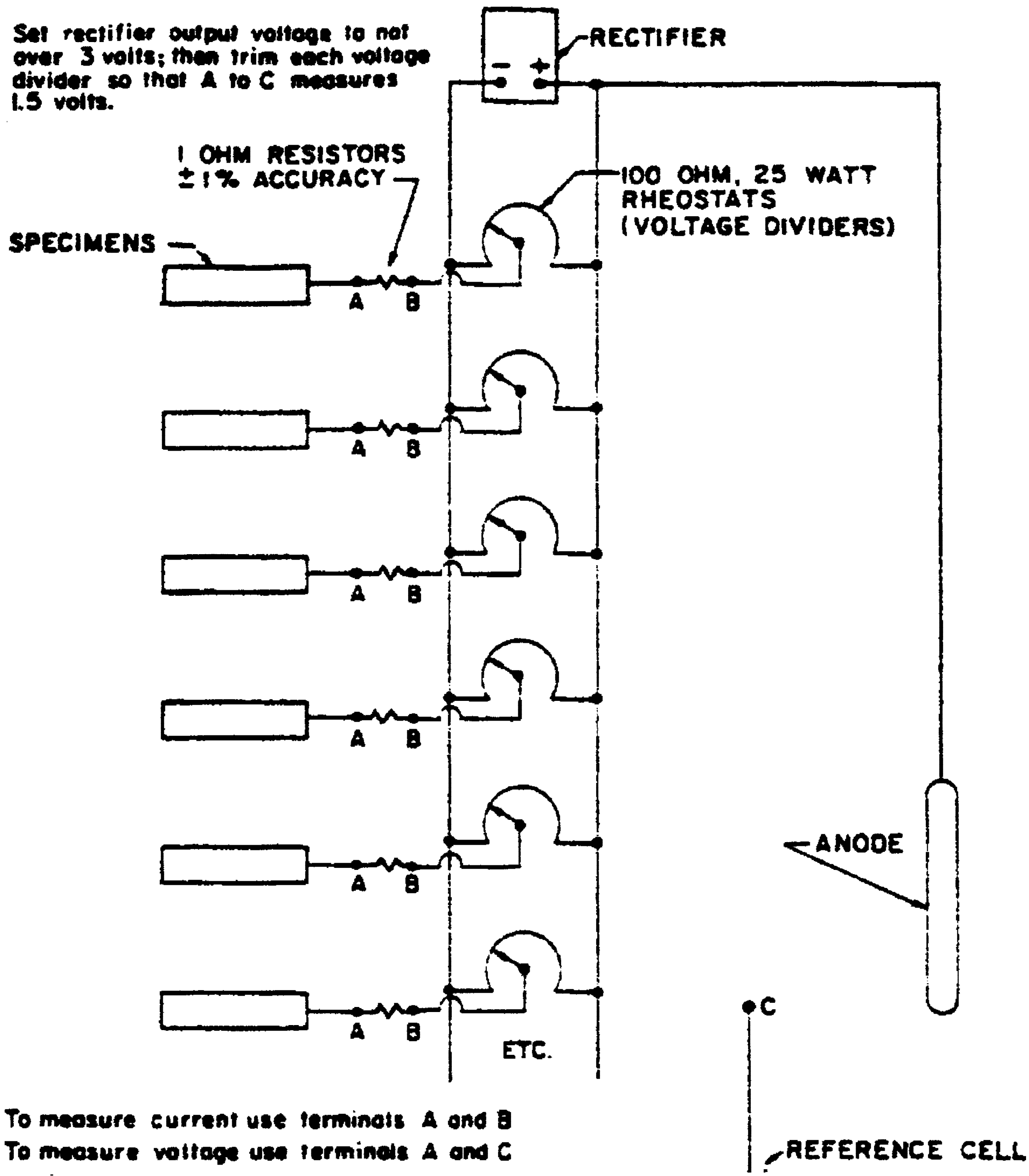

FIG. 5 Circuit Diagram for Cathodic Disbonding Test

the intentional holiday, and record coating condition, for example, color, blisters, cracking, crazing, adhering deposits, and so forth.

10.2 Drill a new reference holiday in the coating in an area that was not immersed. Follow the same drilling procedure as described in 7.2.

10.3 Make radial 45° cuts through the coating intersecting at the center of both the intentional holiday and the reference holiday with a sharp, tin-bladed knife. Take care to ensure that the coating is cut completely through to the steel substrate.

10.4 Attempt to lift the coating at both the reference holiday and the intentional holiday with the point of a sharp, tin-bladed knife. Use the bond at the reference holiday as a reference for judging the quality of the bond at the intentional holiday. Measure and record the total area of disbonded coating at the intentional holiday.

10.5 Measure "zero" bond radius and reduced bond radius as compared to an unexposed reference area. Total disbondment radius is a sum of "zero" and "reduced" bond radiuses.

NOTE 4—Disbondment radius may be obtained from the equation for equivalent circle diameter (ECD):

$$ECD = (A/0.785)\ 1/2$$

where:

A = unsealed (disbonded) area developed from artificial holiday and corrected for holiday area, mm^2 (or $in.^2$).

NOTE 5—The use of a transparent film having a grid laid out in small squares such as 2.54 mm (0.1 in.) on a side has been found useful. The film is placed against the unsealed area and the boundary of the unsealed area is traced on the grid. The area is then obtained by counting the squares within the bounded area.

10.6 Determine and record the resistivity and pH of the electrolyte at the end of the test period.

11. Report

11.1 Report the following information (Fig. 6):

11.1.1 Complete identification of the test specimen, including:

11.1.1.1 Name and code number of the coating,

11.1.1.2 Size and wall thickness of pipe,

11.1.1.3 Applicator, production date, and production run number of coating,

11.1.1.4 Minimum-maximum coating thickness, average thickness, and the thickness at the holiday,

11.1.1.5 Size of initial holidays,

11.1.1.6 Dates of starting and termination of test,

11.1.1.7 Cell diameter and depth of electrolyte,

11.1.1.8 Salt composition and concentration,

11.1.1.9 Cell voltage,

11.1.1.10 Length of test period,

11.1.1.11 Temperature of heat transfer medium (circulating inside pipe sample),

11.1.1.12 Temperature under uncoated side of flat or cut from pipe panels,

11.1.1.13 Temperature of the electrolyte,

11.1.1.14 Report the test results by measuring the total area of disbondment by planimeter, square counting, or other precise method. Subtract the initial holiday area and calculate an equivalent circle diameter, "zero" bond radius and "reduced" bond radius,

11.1.1.15 Current demand in microamperes, and

11.1.1.16 Other information that may be pertinent.

12. Precision and Bias

12.1 Precision and bias statements are not available at this time. Round robin tests will be performed later.

13. Keywords

13.1 cathodic disbondment; elevated temperature; pipeline coatings

DATA SHEET AND REPORT

Report No. ________
Page ______ or ______
Initials ________

Cathodic Disbonding of Pipeline Coatings
Subjected to Elevated Temperatures

Specimen ________ Production Date ______ Target, °C Inside the pipe or under flat panel ______
Coating ________ Production Run No. __________
Applied By: ______________________________
Thickness, mm (in.): Max: ________ Min ______ At Holiday (1) ______ (2) ______ (3) ______
Date Started ________ pH @ 25°C ______________ Ohm-cm @ 25°C ________
Date Finished ________ pH @ 25°C Ohm-cm @ 25°C ________
Immersed Area, mm^2 (in.^{2}) __________ Initial Ohms: + __________
Holiday Diameter, mm (in.) ______________________________

Date	Elapsed Days	Temperature, °C		E_1, V	E_2, V	I_1, µA	ΔE (E_2-E_1), V	Average Values on Target Days	
		Under the panel or inside the pipe	Electrolyte					ΔE, V	I_1, µA
Change: Start to Termination:									

Comments

Physical Examination at Termination:

	mm^2 (in.^{2})	Circ. D[a] mm (in.)	Disbonded Radius, mm Zero	Reduced	Total
Area Disbonded					
Less Initial Holiday					
Total Unsealed Area (net)					

[a] Equivalent Circle Diameter

FIG. 6 Suggested Form for Use in Presenting Data