



# Standard Test Method for Repassivation Potential of Aluminum and Its Alloys by Galvanostatic Measurement<sup>1</sup>

This standard is issued under the fixed designation D6208; 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 A procedure to determine the repassivation potential of aluminum alloy 3003-H14 (UNS A93003) (**1**)<sup>2</sup> as a measure of relative susceptibility to pitting corrosion by conducting a galvanostatic polarization is described. A procedure that can be used to check experimental technique and instrumentation is described, as well.

1.2 The test method serves as a guide for similar measurement on other aluminum alloys and metals (**2-5**).

1.3 The values stated in SI units are to be regarded as the standard. 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*:<sup>3</sup>

**D1193** Specification for Reagent Water

**D3585** Specification for ASTM Reference Fluid for Coolant Tests

**G3** Practice for Conventions Applicable to Electrochemical Measurements in Corrosion Testing

**G15** Terminology Relating to Corrosion and Corrosion Testing (Withdrawn 2010)<sup>4</sup>

**G46** Guide for Examination and Evaluation of Pitting Corrosion

**G107** Guide for Formats for Collection and Compilation of Corrosion Data for Metals for Computerized Database Input

## 3. Terminology

3.1 *Definitions*: Terms used in this test method can be found in Practice **G3** and Terminology **G15**.

3.2 *Symbols*:

3.2.1  $E_B$ —break potential, potential at which the passive aluminum oxide layer breaks down.

3.2.2  $E_G$ —protection potential as measured in this galvanostatic method, potential at which oxide layer repassivates.

3.2.3  $J$ —current density, in A/m<sup>2</sup>

## 4. Summary of Test Method

4.1 The test method described is an adaptation of the method described in FORD Motor Company standards (**6**).

4.2 An aluminum alloy specimen is polarized at fixed current density for 20 min. in a solution of coolant and corrosive water containing chloride. The potential as a function of time is recorded.

4.3 The maximum potential,  $E_B$  reached upon polarization is determined, as is the minimum potential following the maximum potential,  $E_G$ .

4.4 Visual examination of the specimen may be made using Guide **G46** as a guide after disassembly and rinsing.

## 5. Significance and Use

5.1 This test method is designed to measure the relative effectiveness of inhibitors to mitigate pitting corrosion of aluminum and its alloys, in particular AA3003-H14, rapidly and reproducibly. The measurements are not intended to correlate quantitatively with other test method values or with susceptibility to localized corrosion of aluminum observed in service. Qualitative correlation of the measurements and susceptibility in service has been established (**1**).

5.2 The maximum potential reached upon initial polarization,  $E_B$ , is a measure of the resistance to breakdown of the aluminum oxide film. Lower susceptibility to initiation of pitting corrosion is indicated by a more noble potential (See

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D15 on Engine Coolants and Related Fluids and is the direct responsibility of Subcommittee D15.06 on Glassware Performance Tests.

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<sup>2</sup> The boldface numbers in parentheses refer to the list of references at the end of this standard.

<sup>3</sup> 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.

<sup>4</sup> The last approved version of this historical standard is referenced on www.astm.org.

Practice G3 and Terminology G15.) This potential, as measured in this test method, is not very sensitive to the inhibitors present.

5.3 The minimum potential,  $E_G$ , following the maximum potential is a measure of the protection against continued pitting corrosion by the inhibitors. Again, a more noble potential indicates better protection. This potential is sensitive to the inhibitors present.

5.4 Visual examination of the specimens can provide information about subtleties of the pitting and inhibition mechanisms. Number of pits, pit depth, amount of deposit, and surface discoloration are some examples of recordable observations, which can assist evaluation of inhibitor effectiveness.

5.5 The presence of chloride in the test solution is critical to observation of pitting corrosion. Also, a coolant/corrosive water solution in which gas bubbles evolve spontaneously on the aluminum (indicating general corrosion) is unlikely to have a significant amount of observable pitting corrosion.

## 6. Apparatus

6.1 *General Description*—The apparatus for the electrochemical test consists of a cell, current supply, recorder, and three electrodes. Fig. 1 is a generalized schematic of the arrangement. More specific requirements for each component are given below.

6.2 *Cell*—The cell consists of a No.25 O-ring borosilicate glass joint held vertically using standard laboratory clamps and ring stand. The working electrode will be clamped to the bottom using the matching O-ring clamp and viton or silicone rubber gasket.

6.3 *Current Supply and Recorder*—A constant current supply capable of generating 872  $\mu\text{A}$  continuously is required. The recorder must have a high input impedance ( $> 10^{12}$  Ohms), be capable of recording potentials of  $\pm 2$  V with mV accuracy, and have a low gain. These capabilities are typical of commercial potentiostat/galvanostat instruments connected to either a strip chart recorder or computer, for experimental control and data acquisition. The schematic in Fig. 1 shows connections using a

current supply and mV strip chart recorder, and Fig. X2.1 shows a schematic for using a computer and potentiostat/galvanostat.

### 6.4 Electrodes:

6.4.1 *Working Electrode (WE)*—The working electrode, aluminum test coupon, is cut as 51 × 51 mm (2 in. × 2 in.) squares from aluminum sheet 2 to 6 mm (1/16 in. to 1/4 in.) thick. The standard material is AA3003-H14 (UNS A93003), used to develop the precision and bias statements. The coupon is rinsed thoroughly (both sides) with methanol and placed in a low temperature drying oven. No additional surface preparation is desirable. Prior to testing, a coupon is allowed to cool to room temperature. Then it is clamped to the bottom of the O-ring joint using the matching O-ring (viton or silicone rubber) and clamp. The clamping screw may be tightened to finger tightness, if desired. Excessive tightening must be avoided. This gives an area of 8.72 cm<sup>2</sup> aluminum exposed to the solution.

6.4.2 *Auxiliary Electrode (AE)*—Ultrafine grade graphite rod, 6-8 mm (1/4 in.) in diameter and at least 20 cm (8 in.) long. Avoid coarse grades as they can adsorb inhibitors.

6.4.3 *Reference Electrode (RE)*—The reference electrode can be of any convenient type, for example saturated calomel (Hg/HgCl) or silver chloride (Ag/AgCl). The electrode must be in good working order and stable in the solution to be measured. The reference electrode is placed in Luggin probe to avoid solution impedance bias. Appendix X2 contains two suggestions for easily constructed Luggin probes.

6.5 *Timer*—Timer with 1 s resolution out to 30 min.

## 7. Preparation of Apparatus

7.1 *Assembly*—Prior to running tests, assemble the cell and electrodes, using an unprepared Al specimen as the “working” electrode using appropriate clamping. The auxiliary electrode is positioned so that the tip is from 5 to 10 mm from the working electrode surface. The Luggin probe is positioned so that the tip is from 1 to 3 mm from the working electrode surface. It is most convenient if the clamping arrangement is such that this electrode configuration is maintained easily. The cell is then removed and Al specimen unclamped.

## 8. Procedure

8.1 A corrosive water containing chloride, sulfate, and bicarbonate is prepared by dissolving the following amounts of anhydrous salts in distilled or deionized water, ASTM Type II (see Specification D1193):

Sodium sulfate	592 mg
Sodium chloride	660 mg
Sodium bicarbonate	552 mg

The solution is made up to a total weight of 1 kg with distilled or deionized water at 20°C. A 4-kg batch size is convenient if many tests are to be run, multiply amounts above by four. This will give a solution, which is 400 ppm in chloride, sulfate, and bicarbonate.

8.2 Rinse cell, O-ring, Luggin probe (inside and out), auxiliary electrode, and reference electrode thoroughly with Type II water.

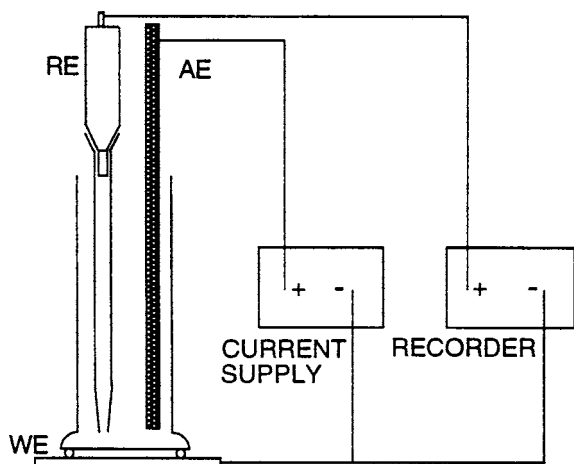


FIG. 1 Generalized Experimental Set-up