LAMPIRAN 1

 $\begin{tabular}{ll} ASTM Standard G 1-03, \\ Standard Practice for Preparing, Cleaning, and Evaluating \\ Corrosion Test Specimen \\ \end{tabular}$





Designation: G 1 - 03

Standard Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens¹

This standard is issued under the fixed designation G 1; 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 suggested procedures for preparing bare, solid metal specimens for tests, for removing corrosion products after the test has been completed, and for evaluating the corrosion damage that has occurred. Emphasis is placed on procedures related to the evaluation of corrosion by mass loss and pitting measurements. (Warning—In many cases the corrosion product on the reactive metals titanium and zirconium is a hard and tightly bonded oxide that defies removal by chemical or ordinary mechanical means. In many such cases, corrosion rates are established by mass gain rather than mass loss.)
- 1.2 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 specific precautionary statements, see 1 and 7.2.

2. Referenced Documents

- 2.1 ASTM Standards:
- A 262 Practices for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels²
- D 1193 Specification for Reagent Water3
- D 1384 Test Method for Corrosion Test for Engine Coolants in Glassware⁴
- D 2776 Test Methods for Corrosivity of Water in the Absence of Heat Transfer (Electrical Methods)⁵
- G 15 Terminology Relating to Corrosion and Corrosion Testing⁶
- G 16 Guide for Applying Statistics to Analysis of Corrosion Data⁶
- ¹ This practice is under the jurisdiction of ASTM Committee G01 on Corrosion of Metals and is the direct responsibility of Subcommittee G01.05 on Laboratory Corrosion Tests.
- Current edition approved October 1, 2003. Published October 2003. Originally approved in 1967. Last previous edition approved in 1999 as $G 1 90 (1999)^{e1}$.
 - ² Annual Book of ASTM Standards, Vol 01.03.
 - 3 Annual Book of ASTM Standards, Vol 11.01.
 - ⁴ Annual Book of ASTM Standards, Vol 15.05.
- ⁵ Discontinued, replaced by Guide G 96. See 1990 Annual Book of ASTM Standards, Vol 03.02.
 - ⁶ Annual Book of ASTM Standards, Vol 03.02.

- G 31 Practice for Laboratory Immersion Corrosion Testing of Metals⁶
- G 33 Practice for Recording Data from Atmospheric Corrosion Tests of Metallic-Coated Steel Specimens⁶
- G 46 Guide for Examination and Evaluation of Pitting Corrosion⁶
- G 50 Practice for Conducting Atmospheric Corrosion Tests on Metals⁶
- G 78 Guide for Crevice Corrosion Testing of Iron Base and Nickel-Base Stainless Alloys in Seawater and Other Chloride-Containing Aqueous Environments⁶

3. Terminology

3.1 See Terminology G 15 for terms used in this practice.

4. Significance and Use

- 4.1 The procedures given are designed to remove corrosion products without significant removal of base metal. This allows an accurate determination of the mass loss of the metal or alloy that occurred during exposure to the corrosive environment.
- 4.2 These procedures, in some cases, may apply to metal coatings. However, possible effects from the substrate must be considered.

5. Reagents and Materials

- 5.1 Purity of Reagents—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.
- 5.2 Purity of Water—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type IV of Specification D 1193.

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⁷ Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see Analar Standards for Laboratory Chemicals, BDH Ltd., Poole, Dorset, U.K., and the United States Pharmacopeia and National Formulary, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.



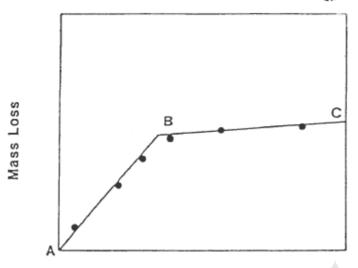
6. Methods for Preparing Specimens for Test

- 6.1 For laboratory corrosion tests that simulate exposure to service environments, a commercial surface, closely resembling the one that would be used in service, will yield the most meaningful results.
- 6.2 It is desirable to mark specimens used in corrosion tests with a unique designation during preparation. Several techniques may be used depending on the type of specimen and test
- 6.2.1 Stencil or Stamp—Most metallic specimens may be marked by stenciling, that is, imprinting the designation code into the metal surface using hardened steel stencil stamps hit with a hammer. The resulting imprint will be visible even after substantial corrosion has occurred. However, this procedure introduces localized strained regions and the possibility of superficial iron contamination in the marked area.
- 6.2.2 Electric engraving by means of a vibratory marking tool may be used when the extent of corrosion damage is known to be small. However, this approach to marking is much more susceptible to having the marks lost as a result of corrosion damage during testing.
- 6.2.3 Edge notching is especially applicable when extensive corrosion and accumulation of corrosion products is anticipated. Long term atmospheric tests and sea water immersion tests on steel alloys are examples where this approach is applicable. It is necessary to develop a code system when using edge notches.
- 6.2.4 Drilled holes may also be used to identify specimens when extensive metal loss, accumulation of corrosion products, or heavy scaling is anticipated. Drilled holes may be simpler and less costly than edge notching. A code system must be developed when using drilled holes. Punched holes should not be used as they introduce residual strain.
- 6.2.5 When it is undesirable to deform the surface of specimens after preparation procedures, for example, when testing coated surfaces, tags may be used for specimen identification. A metal or plastic wire can be used to attach the tag to the specimen and the specimen identification can be stamped on the tag. It is important to ensure that neither the tag nor the wire will corrode or degrade in the test environment. It is also important to be sure that there are no galvanic interactions between the tag, wire, and specimen.
- 6.3 For more searching tests of either the metal or the environment, standard surface finishes may be preferred. A suitable procedure might be:
- 6.3.1 Degrease in an organic solvent or hot alkaline cleaner. (See also Practice G 31.)
- Note $\,1$ —Hot alkalies and chlorinated solvents may attack some metals.
- Note 2—Ultrasonic cleaning may be beneficial in both pre-test and post-test cleaning procedures.
- 6.3.2 Pickle in an appropriate solution if oxides or tarnish are present. In some cases the chemical cleaners described in Section 6 will suffice.
 - Note 3-Pickling may cause localized corrosion on some materials.
- 6.3.3 Abrade with a slurry of an appropriate abrasive or with an abrasive paper (see Practices A 262 and Test Method

- D 1384). The edges as well as the faces of the specimens should be abraded to remove burrs.
 - 6.3.4 Rinse thoroughly, hot air dry, and store in desiccator.
- 6.4 When specimen preparation changes the metallurgical condition of the metal, other methods should be chosen or the metallurgical condition must be corrected by subsequent treatment. For example, shearing a specimen to size will cold work and may possibly fracture the edges. Edges should be machined.
- 6.5 The clean, dry specimens should be measured and weighed. Dimensions determined to the third significant figure and mass determined to the fifth significant figure are suggested. When more significant figures are available on the measuring instruments, they should be recorded.

7. Methods for Cleaning After Testing

- 7.1 Corrosion product removal procedures can be divided into three general categories: mechanical, chemical, and electrolytic.
- 7.1.1 An ideal procedure should remove only corrosion products and not result in removal of any base metal. To determine the mass loss of the base metal when removing corrosion products, replicate uncorroded control specimens should be cleaned by the same procedure being used on the test specimen. By weighing the control specimen before and after cleaning, the extent of metal loss resulting from cleaning can be utilized to correct the corrosion mass loss.
- Note 4—It is desirable to scrape samples of corrosion products before using any chemical techniques to remove them. These scrapings can then be subjected to various forms of analyses, including perhaps X-ray diffraction to determine crystal forms as well as chemical analyses to look for specific corrodants, such as chlorides. All of the chemical techniques that are discussed in Section 7 tend to destroy the corrosion products and thereby lose the information contained in these corrosion products. Care may be required so that uncorroded metal is not removed with the corrosion products.
- 7.1.2 The procedure given in 7.1.1 may not be reliable when heavily corroded specimens are to be cleaned. The application of replicate cleaning procedures to specimens with corroded surfaces will often, even in the absence of corrosion products, result in continuing mass losses. This is because a corroded surface, particularly of a multiphase alloy, is often more susceptible than a freshly machined or polished surface to corrosion by the cleaning procedure. In such cases, the following method of determining the mass loss due to the cleaning procedure is preferred.
- 7.1.2.1 The cleaning procedure should be repeated on specimens several times. The mass loss should be determined after each cleaning by weighing the specimen.
- 7.1.2.2 The mass loss should be graphed as a function of the number of equal cleaning cycles as shown in Fig. 1. Two lines will be obtained: AB and BC. The latter will correspond to corrosion of the metal after removal of corrosion products. The mass loss due to corrosion will correspond approximately to point B.
- 7.1.2.3 To minimize uncertainty associated with corrosion of the metal by the cleaning method, a method should be chosen to provide the lowest slope (near to horizontal) of line BC.



Number of Cleaning Cycles FIG. 1 Mass Loss of Corroded Specimens Resulting from Repetitive Cleaning Cycles

- 7.1.3 Repeated treatment may be required for complete removal of corrosion products. Removal can often be confirmed by examination with a low power microscope (for example, 7× to 30×). This is particularly useful with pitted surfaces when corrosion products may accumulate in pits. This repeated treatment may also be necessary because of the requirements of 7.1.2.1. Following the final treatment, the specimens should be thoroughly rinsed and immediately dried.
- 7.1.4 All cleaning solutions shall be prepared with water and reagent grade chemicals.
- 7.2 Chemical procedures involve immersion of the corrosion test specimen in a specific solution that is designed to remove the corrosion products with minimal dissolution of any base metal. Several procedures are listed in Table A1.1. The choice of chemical procedure to be used is partly a matter of trial and error to establish the most effective method for a specific metal and type of corrosion product scale. (Warning—These methods may be hazardous to personnel).
- 7.2.1 Chemical cleaning is often preceded by light brushing (non metallic bristle) or ultrasonic cleaning of the test specimen to remove loose, bulky corrosion products.
- 7.2.2 Intermittent removal of specimens from the cleaning solution for light brushing or ultrasonic cleaning can often facilitate the removal of tightly adherent corrosion products.
- 7.2.3 Chemical cleaning is often followed by light brushing or ultrasonic cleaning in reagent water to remove loose products.
- 7.3 Electrolytic cleaning can also be utilized for removal of corrosion products. Several useful methods for corrosion test specimens of iron, cast iron, or steel are given in Table A2.1.
- 7.3.1 Electrolytic cleaning should be preceded by brushing or ultrasonic cleaning of the test specimen to remove loose, bulky corrosion products. Brushing or ultrasonic cleaning should also follow the electrolytic cleaning to remove any loose slime or deposits. This will help to minimize any redeposition of metal from reducible corrosion products that would reduce the apparent mass loss.

- 7.4 Mechanical procedures can include scraping, scrubbing, brushing, ultrasonic cleaning, mechanical shocking, and impact blasting (for example, grit blasting, water-jet blasting, and so forth). These methods are often utilized to remove heavily encrusted corrosion products. Scrubbing with a nonmetallic bristle brush and a mild abrasive-distilled water slurry can also be used to remove corrosion products.
- 7.4.1 Vigorous mechanical cleaning may result in the removal of some base metal; therefore, care should be exercised. These should be used only when other methods fail to provide adequate removal of corrosion products. As with other methods, correction for metal loss due to the cleaning method is recommended. The mechanical forces used in cleaning should be held as nearly constant as possible.

8. Assessment of Corrosion Damage

8.1 The initial total surface area of the specimen (making corrections for the areas associated with mounting holes) and the mass lost during the test are determined. The average corrosion rate may then be obtained as follows:

Corrosion Rate =
$$(K \times W)/(A \times T \times D)$$
 (1)

where:

K = a constant (see 8.1.2).

T = time of exposure in hours,

 $A = area in cm^2$

W = mass loss in grams, and

 $D = density in g/cm^3$ (see Appendix X1).

- 8.1.1 Corrosion rates are not necessarily constant with time of exposure. See Practice G 31 for further guidance.
- 8.1.2 Many different units are used to express corrosion rates. Using the units in 7.1 for T, A, W, and D, the corrosion rate can be calculated in a variety of units with the following appropriate value of K:

| | Constant (K) in Corrosion |
|--|-------------------------------|
| Corrosion Rate Units Desired | Rate Equation |
| mils per year (mpy) | 3.45×10^{6} |
| inches per year (ipy) | 3.45×10^{3} |
| inches per month (ipm) | 2.87×10^{2} |
| millimetres per year (mm/y) | 8.76×10^{4} |
| micrometres per year (um/y) | 8.76×10^{7} |
| picometres per second (pm/s) | 2.78×10^{6} |
| grams per square meter per hour (g/m2·h) | $1.00 \times 10^{4} \times D$ |
| milligrams per square decimeter per day (mdd) | $2.40 \times 10^{6} \times D$ |
| micrograms per square meter per second (µg/m²-s) | $2.78 \times 10^{6} \times D$ |

Note 5—If desired, these constants may also be used to convert corrosion rates from one set of units to another. To convert a corrosion rate in units X to a rate in units Y, multiply by K_{ν}/K_{X} ; for example:

$$15 \text{ mpy} = 15 \times (2.78 \times 10^6)/(3.45 \times 10^6) \text{ pm/s}$$
 (2)

8.1.3 In the case of sacrificial alloy coatings for which there is preferential corrosion of a component whose density differs from that of the alloy, it is preferable to use the density of the corroded component (instead of the initial alloy density) for calculating average thickness loss rate by use of Eq 1. This is done as follows: (1) cleaning to remove corrosion products only and determine the mass loss of the corroded component; (2) stripping the remaining coating to determine the mass of the uncorroded component; (3) chemical analysis of the stripping solution to determine the composition of the uncorroded



component; (4) performing a mass balance to calculate the composition of the corroded component; (5) using the mass and density of the corroded component to calculate the average thickness loss rate by use of Eq 1. An example of this procedure is given in Appendix X2.

The procedure described above gives an average penetration rate of the coating, but the maximum penetration for a multiphase alloy may be larger when the corroded phase is not uniformly distributed across the surface. In such cases, it is generally considered good practice to obtain a cross section through the corroded surface for microscopic examination. This examination will reveal the extent of selective corrosion of particular phases in the coating, and help in understanding the mechanism of attack.

8.2 Corrosion rates calculated from mass losses can be misleading when deterioration is highly localized, as in pitting or crevice corrosion. If corrosion is in the form of pitting, it may be measured with a depth gage or micrometer calipers with pointed anvils (see Guide G 46). Microscopical methods will determine pit depth by focusing from top to bottom of the pit when it is viewed from above (using a calibrated focusing knob) or by examining a section that has been mounted and metallographically polished. The pitting factor is the ratio of the deepest metal penetration to the average metal penetration (as measured by mass loss).

Note 6—See Guide G 46 for guidance in evaluating depths of pitting.

Note 7—See Guide G 78 for guidance in evaluating crevice corrosion.

- 8.3 Other methods of assessing corrosion damage are:
- 8.3.1 Appearance—The degradation of appearance by rusting, tarnishing, or oxidation. (See Practice G 33.)
- 8.3.2 Mechanical Properties—An apparent loss in tensile strength will result if the cross-sectional area of the specimen (measured before exposure to the corrosive environment) is reduced by corrosion. (See Practice G 50.) Loss in tensile strength will result if a compositional change, such as dealloying taking place. Loss in tensile strength and elongation will result from localized attack, such as cracking or intergranular corrosion.
- 8.3.3 Electrical Properties—Loss in electrical conductivity can be measured when metal loss results from uniform corrosion. (See Test Methods D 2776.)
- 8.3.4 Microscopical Examination—Dealloying, exfoliation, cracking, or intergranular attack may be detected by metallographic examination of suitably prepared sections.

9. Report

9.1 The report should include the compositions and sizes of specimens, their metallurgical conditions, surface preparations, and cleaning methods as well as measures of corrosion damage, such as corrosion rates (calculated from mass losses), maximum depths of pitting, or losses in mechanical properties.

10. Precision and Bias

- 10.1 The factors that can produce errors in mass loss measurement include improper balance calibration and standardization. Generally, modern analytical balances can determine mass values to ± 0.2 mg with ease and balances are available that can obtain mass values to ± 0.02 mg. In general, mass measurements are not the limiting factor. However, inadequate corrosion product removal or overcleaning will affect precision.
- 10.2 The determination of specimen area is usually the least precise step in corrosion rate determinations. The precision of calipers and other length measuring devices can vary widely. However, it generally is not necessary to achieve better than ± 1 % for area measurements for corrosion rate purposes.
- 10.3 The exposure time can usually be controlled to better than ± 1 % in most laboratory procedures. However, in field exposures, corrosive conditions can vary significantly and the estimation of how long corrosive conditions existed can present significant opportunities for error. Furthermore, corrosion processes are not necessarily linear with time, so that rate values may not be predictive of the future deterioration, but only are indications of the past exposure.
- 10.4 Regression analysis on results, as are shown in Fig. 1, can be used to obtain specific information on precision. See Guide G 16 for more information on statistical analysis.
- 10.5 Bias can result from inadequate corrosion product removal or metal removal caused by overcleaning. The use of repetitive cleaning steps, as shown in Fig. 1, can minimize both of these errors.
- 10.5.1 Corrosion penetration estimations based on mass loss can seriously underestimate the corrosion penetration caused by localized processes, such as pitting, cracking, crevice corrosion, and so forth.

11. Keywords

11.1 cleaning; corrosion product removal; evaluation; mass loss; metals; preparation; specimens



ANNEXES

(Mandatory Information)

A1. CHEMICAL CLEANING PROCEDURES

TABLE A1.1 CHEMICAL CLEANING PROCEDURES FOR REMOVAL OF CORROSION PRODUCTS

| Designation | Material | Solution | Time | Temperature | Remarks |
|-------------|-------------------------------------|---|--------------|-----------------|---|
| C.1.1 | Aluminum and Alu- minum Alloys | 50 mL phosphoric acid (H ₃ PO ₄ , sp gr 1.69) 20 g chromium trioxide (CrO ₃) Reagent water to make 1000 mL | 5 to 10 min | 90°C to Boiling | If corrosion product films remain, rinse, then follow with nitric acid procedure (C.1.2). |
| C.1.2 | | Nitric acid (HNO ₃ , sp gr 1.42) | 1 to 5 min | 20 to 25°C | Remove extraneous deposits and bulky corrosion products to avoid reactions that may result in excessive removal of base metal. |
| C.2.1 | Copper and Copper Alloys | 500 mL hydrochloric acid (HCl, sp gr 1.19) Reagent water to make 1000 mL | 1 to 3 min | 20 to 25°C | Deaeration of solution with purified nitrogen will minimize base metal removal. |
| C.2.2 | | 4.9 g sodium cyanide (NaCN) Reagent water to make 1000 mL | 1 to 3 min | 20 to 25°C | Removes copper sulfide corrosion products that may not be removed by hydrochloric acid treatment (C.2.1). |
| C.2.3 | | 100 mL sulfuric acid (H ₂ SO ₄ , sp gr 1.84) Reagent water to make 1000 mL | 1 to 3 min | 20 to 25°C | Remove bulky corrosion products before treatment to minimize copper redeposition on specimen surface. |
| C.2.4 | | 120 mL sulfuric acid (H ₂ SO ₄ , sp gr 1.84) 30 g sodium dichromate (Na ₂ Cr ₂ O ₇ ·2H ₂ O) Reagent water to make 1000 mL | 5 to 10 s | 20 to 25°C | Removes redeposited copper resulting from sulfuric acid treatment. |
| C.2.5 | | 54 mL sulfuric acid (H ₂ SO ₄ , sp gr 1.84) Reagent water to make 1000 mL | 30 to 60 min | 40 to 50°C | Deaerate solution with nitrogen. Brushing of test specimens to remove corrosion products followed by re-immersion for 3 to |
| | | | | | 4 s is recommended. |
| C.3.1 | Iron and Steel | 1000 mL hydrochloric acid (HCl, sp gr 1.19) 20 g antimony trioxide (Sb ₂ O ₃) 50 g stannous chloride (SnCl ₂) | 1 to 25 min | 20 to 25°C | Solution should be vigorously stirred or specimen should be brushed. Longer times may be required in certain instances. |
| C.3.2 | | 50 g sodium hydroxide (NaOH) 200 g granulated zinc or zinc chips | 30 to 40 min | 80 to 90°C | Caution should be exercised in the use of any zinc dust since spontaneous ignition |
| C.3.3 | | Reagent water to make 1000 mL | 30 to 40 min | 80 to 90°C | upon exposure to air can occur. Caution should be exercised in the use of |
| 0.3.3 | | 200 g sodium hydroxide (NaOH) 20 g granulated zinc or zinc chips Reagent water to make 1000 mL | 30 to 40 min | 90 to 80 C | any zinc dust since spontaneous ignition upon exposure to air can occur. |
| C.3.4 | | 200 g diammonium citrate | 20 min | 75 to 90°C | Depending upon the composition of the |
| | | ((NH ₄) ₂ HC ₆ H ₅ O ₇) Reagent water to make 1000 mL | | | corrosion product, attack of base metal may occur. |
| C.3.5 | | 500 mL hydrochloric acid (HCl, sp gr 1.19) 3.5 g hexamethylene tetramine Reagent water to make 1000 mL | 10 min | 20 to 25°C | Longer times may be required in certain instances. |
| C.3.6 | | Molten caustic soda (NaOH) with | 1 to 20 min | 370°C | For details refer to Technical Information |
| | | 1.5–2.0 % sodium hydride (NaH) | | | Bulletin SP29-370, "DuPont Sodium Hydride Descaling Process Operating Instructions." |
| C.4.1 | Lead and Lead Alloys | 10 mL acetic acid (CH ₃ COOH) | 5 min | Boiling | |
| C.4.2 | | Reagent water to make 1000 mL 50 g ammonium acetate (CH ₃ COONH ₄) | 10 min | 60 to 70°C | |
| C.4.3 | | Reagent water to make 1000 mL 250 g ammonium acetate (CH ₃ COONH ₄) Reagent water to make 1000 mL | 5 min | 60 to 70°C | |
| | | Reagent water to make 1000 mc | | | |
| C.5.1 | Magnesium and Mag- nesium Alloys | 150 g chromium trioxide (CrO ₃) 10 g silver chromate (Ag ₂ CrO ₄) Reagent water to make 1000 mL | 1 min | Boiling | The silver salt is present to precipitate chloride. |
| C.5.2 | | 200 g chromium trioxide (CrO ₃) 10 g silver nitrate (AgNO ₃) 20 g barium nitrate (Ba(NO ₃) ₂) Reagent water to make 1000 mL | 1 min | 20 to 25°C | The barium salt is present to precipitate sulfate. |
| C.6.1 | Nickel and Nickel Alloys | 150 mL hydrochloric acid (HCl, sp gr 1.19) Reagent water to make 1000 mL | 1 to 3 min | 20 to 25°C | *** |
| C.6.2 | | 100 mL sulfuric acid (H ₂ SO ₄ , sp gr 1.84) Reagent water to make 1000 mL | 1 to 3 min | 20 to 25°C | |
| C.7.1 | Stainless Steels | 100 mL nitric acid (HNO ₃ , sp gr 1.42) Reagent water to make 1000 mL | 20 min | 60°C | |

TABLE A1.1 Continued

| Designation | Material | Solution | Time | Temperature | Remarks |
|-------------|---------------------------|--|--------------|-------------|--|
| C.7.2 | | 150 g diammonium citrate | 10 to 60 min | 70°C | |
| | | ((NH ₄) ₂ HC ₆ H ₅ O ₇) | | | |
| 0.7.0 | | Reagent water to make 1000 mL | E | 2010 | |
| C.7.3 | | 100 g citric acid (C ₆ H ₈ O ₇) | 5 min | 60°C | *** |
| | | 50 mL sulfuric acid (H ₂ SO ₄ , sp gr 1.84) | | | |
| | | 2 g inhibitor (diorthotolyl thiourea or | | | |
| | | quinoline ethyliodide or betanaphthol quinoline) | | | |
| | | Reagent water to make 1000 mL | | | |
| C.7.4 | | 200 g sodium hydroxide (NaOH) | 5 min | Boiling | |
| 0.7 | | 30 g potassium permanganate (KMnO ₄) | 0 111111 | Doming | *** |
| | | Reagent water to make 1000 mL | | | |
| | | followed by | | | |
| | | 100 g diammonium citrate | | | |
| | | ((NH ₄) ₂ HC ₆ H ₅ O ₇) | | | |
| | | Reagent water to make 1000 mL | | | |
| C.7.5 | | 100 mL nitric acid (HNO ₃ , sp gr 1.42) | 5 to 20 min | 20 to 25°C | *** |
| | | 20 mL hydrofluoric acid (HF, sp gr | | | |
| | | 1.198-48 %) | | | |
| | | Reagent water to make 1000 mL | | | |
| C.7.6 | | 200 g sodium hydroxide (NaOH) | 20 min | Boiling | Caution should be exercised in the use of |
| | | 50 g zinc powder | | | any zinc dust since spontaneous ignition |
| | T 1T 41 | Reagent water to make 1000 mL | 40 . | 5 7 | upon exposure to air can occur. |
| C.8.1 | Tin and Tin Alloys | 150 g trisodium phosphate | 10 min | Boiling | *** |
| | | (Na ₃ PO ₄ ·12H ₂ O) | | | |
| C.8.2 | | Reagent water to make 1000 mL 50 mL hydrochloric acid (HCl, sp gr 1.19) | 10 min | 20°C | |
| 0.0.2 | | Reagent water to make 1000 mL | 10 111111 | 20 0 | |
| C.9.1 | Zinc and Zinc Alloys | 150 mL ammonium hydroxide (NH ₄ OH, | 5 min | 20 to 25°C | |
| 0.0.1 | Ellio dila Ellio / liloys | sp gr 0.90) | O IIIIII | 20 10 20 0 | |
| | | Reagent water to make 1000 mL | | | |
| | | followed by | | | |
| | | 50 g chromium trioxide (CrO ₃) | 15 to 20 s | Boiling | The silver nitrate should be dissolved in water |
| | | 10 g silver nitrate (AgNO ₃) | | | and added to the boiling chromic acid to |
| | | Reagent water to make 1000 mL | | | prevent excessive crystallization of silver |
| | | | | | chromate. The chromic acid must be |
| | | | | | sulfate free to avoid attack of the zinc base |
| | | | | | metal. |
| C.9.2 | | 100 g ammonium chloride (NH ₄ Cl) | 2 to 5 min | 70°C | |
| | | Reagent water to make 1000 mL | | 0000 | Objected and an invitation of the observation and |
| C.9.3 | | 200 g chromium trioxide (CrO ₃) | 1 min | 80°C | Chloride contamination of the chromic acid from corrosion products formed in salt |
| | | Reagent water to make 1000 mL | | | environments should be avoided to prevent |
| | | | | | attack of the zinc base metal. |
| C.9.4 | | 85 mL hydriodic acid (HI, sp gr 1.5) | 15 s | 20 to 25°C | Some zinc base metal may be removed. A |
| | | Reagent water to make 1000 mL | | 20 10 20 0 | control specimen (3.1.1) should be |
| | | | | | employed. |
| C.9.5 | | 100 g ammonium persulfate ((NH ₄) ₂ S ₂ O ₈) | 5 min | 20 to 25°C | Particularly recommended for galvanized |
| | | Reagent water to make 1000 mL | | | steel. |
| C.9.6 | | 100 g ammonium acetate (CH ₃ COONH ₄) | 2 to 5 min | 70°C | *** |
| | | Reagent water to make 1000 mL | | | |

A2. ELECTROLYTIC CLEANING PROCEDURES

TABLE A2.1 ELECTROLYTIC CLEANING PROCEDURES FOR REMOVAL OF CORROSION PRODUCTS

| Designation | Material | Solution | Time | Temperature | Remarks |
|-------------|------------------------|---|--------------|-------------|---|
| E.1.1 | Iron, Cast Iron, Steel | 75 g sodium hydroxide (NaOH) 25 g sodium sulfate (Na ₂ SO ₄) 75 g sodium carbonate (Na ₂ CO ₃) Reagent water to make 1000 mL | 20 to 40 min | 20 to 25°C | Cathodic treatment with 100 to 200 A/m ² cur- rent density. Use carbon, platinum or stainless steel anode. |



TABLE A2.1 Continued

| Designation | Material | Solution | Time | Temperature | Remarks |
|-------------|--|--|-------------|-------------|--|
| E.1.2 | | 28 mL sulfuric acid (H ₂ SO ₄ , sp gr 1.84) 0.5 g inhibitor (diorthotolyl thiourea or quinoline ethyliodide or betanaphthol quinoline) | 3 min | 75°C | Cathodic treatment with 2000 A/m² current density. Use carbon, platinum or lead anode. |
| E.1.3 | | Reagent water to make 1000 mL 100 g diammonium citrate ((NH ₄) ₂ HC ₆ H ₅ O ₇) Reagent water to make 1000 mL | 5 min | 20 to 25°C | Cathodic treatment with 100 A/m² current density. Use carbon or platinum anode. |
| E.2.1 | Lead and Lead Alloys | 28 mL sulfuric acid (H ₂ SO ₄ , sp gr 1.84) 0.5 g inhibitor (diorthotolyl thiourea or quinoline ethyliodide or betanaphthol quinoline) | 3 min | 75°C | Cathodic treatment with 2000 A/m² current density. Use carbon, platinum or lead anode. |
| E.3.1 | Copper and Copper Alloys | Reagent water to make 1000 mL 7.5 g potassium chloride (KCI) Reagent water to make 1000 mL | 1 to 3 | 20 to 25°C | Cathodic treatment with 100 A/m ² current den- sity. Use carbon or platinum anode. |
| E.4.1 | Zinc and Cadmium | 50 g dibasic sodium phosphate (Na ₂ HPO ₄) Reagent water to make 1000 mL | 5 min | 70°C | Cathodic treatment with 110 A/m ² current den- sity. Specimen must be energized prior to im- mersion. Use carbon, platinum or stainless steel anode. |
| E.4.2 | | 100 g sodium hydroxide (NaOH) Reagent water to make 1000 mL | 1 to 2 min | 20 to 25°C | Cathodic treatment with 100 A/m ² current den- sity. Specimen must be energized prior to im- mersion. Use carbon, platinum or stainless steel anode. |
| E.5.1 | General (excluding Alu- minum, Magnesium and Tin Alloys) | 20 g sodium hydroxide (NaOH) Reagent water to make 1000 mL | 5 to 10 min | 20 to 25°C | Cathodic treatment with 300 A/m ² current den- sity. A S31600 stainless steel anode may be used. |

APPENDIXES

(Nonmandatory Information)

X1. DENSITIES FOR A VARIETY OF METALS AND ALLOYS

TABLE X1.1 DENSITIES FOR A VARIETY OF METALS AND ALLOYS

Note 1-All UNS numbers that include the letter X indicate a series of numbers under one category.

Note 2—An asterisk indicates that a UNS number not available.

| LINION | | p 3 |
|------------|------------------|---------------------------|
| UNS Number | Alloy | Density g/cm ³ |
| A91100 | 1100 | 2.71 |
| A91199 | 1199 | 2.70 |
| A92024 | 2024 | 2.78 |
| A92219 | 2219 | 2.84 |
| A93003 | 3003 | 2.73 |
| A93004 | 3004 | 2.72 |
| A95005 | 5005 | 2.70 |
| A95050 | 5050 | 2.69 |
| A95052 | 5052 | 2.68 |
| A95083 | 5083 | 2.66 |
| A95086 | 5086 | 2.66 |
| A95154 | 5154 | 2.66 |
| A95357 | 5357 | 2.69 |
| A95454 | 5454 | 2.69 |
| A95456 | 5456 | 2.66 |
| A96061 | 6061 | 2.70 |
| × | 6062 | 2.70 |
| A98070 | 6070 | 2.71 |
| A96101 | 6101 | 2.70 |
| A97075 | 7075 | 2.81 |
| A97079 | 7079 | 2.75 |
| A97178 | 7178 | 2.83 |
| | Stainless Steels | |
| S20100 | Type 201 | 7.94 |
| S20200 | Type 202 | 7.94 |
| S30200 | Type 302 | 7.94 |
| S30400 | Type 304 | 7.94 |



TABLE X1.1 Continued

| | TABLE X1.1 Continued | |
|------------------|--|---------------------------|
| | Aluminum Alloys | |
| UNS Numbe | Alloy | Density g/cm ³ |
| S30403 | Type 304L | 7.94 |
| \$30900 | Type 309 | 7.98 |
| S31000 | Type 310 | 7.98 |
| S31100 | Type 311 | 7.98 |
| S31600 | Type 318 | 7.98 |
| S31603 | ** | 7.98 |
| | Type 316L | |
| S31700 | Type 317 | 7.98 |
| S32100 | Type 321 | 7.94 |
| S32900 | Type 329 | 7.98 |
| N08330 | Type 330 | 7.98 |
| S34700 | Type 347 | 8.03 |
| S41000 | Type 410 | 7.70 |
| S43000 | Type 430 | 7.72 |
| \$44600 | Type 448 | 7.65 |
| \$50200 | Type 502 | 7.82 |
| 330200 | ** | 7.02 |
| FAVOOR | Other Ferrous Metals | 7.00 |
| F1XXXX | Gray cast iron | 7.20 |
| GXXXXX—KXXXX | Carbon steel | 7.86 |
| * | Silicon iron | 7.00 |
| KXXXXX | Low alloy steels | 7.85 |
| | Copper Alloys | |
| C38600 | Copper | 8.94 |
| C23000 | Red brass 230 | 8.75 |
| C26000 | Cartridge brass 260 | 8.52 |
| | | |
| C28000 | Muntz metal 280 | 8.39 |
| * | Admiralty 442 | 8.52 |
| C44300 | Admiralty 443 | 8.52 |
| C44400 | Admiralty 444 | 8.52 |
| C44500 | Admiralty 445 | 8.52 |
| C68700 | Aluminum brass 687 | 8.33 |
| C22000 | Commercial bronze 220 | 8.80 |
| C80800 | Aluminum bronze, 5 % 608 | 8.16 |
| C00000 | | |
| _ | Aluminum bronze, 8 % 612 | 7.78 |
| | Composition M | 8.45 |
| * | Composition G | 8.77 |
| C51000 | Phosphar bronze, 5 % 510 | 8.86 |
| C52400 | Phosphor bronze, 10 % 524 | 8.77 |
| × | 85-5-5-5 | 8.80 |
| C65500 | Silicon bronze 655 | 8.52 |
| C70600 | Copper nickel 708 | 8.94 |
| C71000 | | 8.94 |
| | Copper nickel 710 | |
| C71500 | Copper nickel 715 | 8.94 |
| C75200 | Nickel silver 752 | 8.75 |
| | Lead | |
| L53305-53405 | Antimonial | 10.80 |
| L5XXXX | Chemical | 11.33 |
| Nickel Alloys | | |
| N02200 | Nickel 200 | 8.89 |
| N04400 | Nickel copper 400 | 8.84 |
| N06600 | Nickel chromium iron alloy 600 | 8.51 |
| | | |
| N06625 | Nickel chromium molybdenum alloy 625 | 8.44 |
| N08825 | Iron nickel chromium alloy 825 | 8.14 |
| N08020 | Iron nickel chromium alloy 20 Cb-3 | 8.08 |
| × | Iron nickel chromium cast alloy 20 | 8.02 |
| N10665 | Nickel molybdenum alloy B2 | 9.2 |
| N10276 | Nickel chromium molybdenum alloy | 8.8 |
| | C-276 | 0.0 |
| NIDRODE | | 0.0 |
| N06985 | Nickel chromium molybdenum alloy G-3 Other Metals | 8.3 |
| M1XXXX | Magnesium | 1.74 |
| R03600 | Molybdenum | 10.22 |
| | | |
| P04980 | Platinum | 21.45 |
| P07016 | Silver | 10.49 |
| | Tantalum | 16.60 |
| R05200 | Tin | 7.30 |
| R05200 L13002 | | |
| L13002 | Titanium | |
| | Titanium Zinc | 4.54 7.13 |

X2. CALCULATION OF AVERAGE THICKNESS LOSS RATE OF AN ALLOY WHEN THE DENSITY OF THE CORRODING METAL DIFFERS FROM THAT OF THE BULK ALLOY

X2.1 Example

X2.1.1 55% A1-Zn alloy coating on steel sheet exposed for 20.95 years at Point Reyes, CA. (As reported in H.E. Townsend and H.H.Lawson, "Twenty-One Year Results for Metallic-Coated Sheet in the ASTM 1976 Atmospheric Corrosion Tests").8

X2.2 Measurements

X2.2.1 Initial aluminum content of coating, C₁, as measured by stripping (Table A1.1, C.3.) and chemical analysis of uncorroded specimens.

$$C_1 = 55.0\% \text{ Al}$$
 (X2.1)

X2.2.2 Time of Exposure, T

$$T = 20.95 \text{ years} = 183 648 \text{ hours}$$
 (X2.2)

X2.2.3 Specimen Area, A

$$A = 300 \text{ cm}^2$$
 (X2.3)

X2.2.4 Initial Mass, W1

$$W_1 = 79.3586 \text{ g}$$
 (X2.4)

X2.2.5 Mass after exposure and removal of corrosion products according to Table A1.1, C.9.3, W₂

$$W_2 = 78.7660 \text{ g}$$
 (X2.5)

X2.2.6 Mass after removal of remaining coating according to Table A1.1, C.3.5, W₃

$$W_3 = 75.0810 \text{ g}$$
 (X2.6)

X2.2.7 Aluminum content of remaining uncorroded coating by chemical analysis of the stripping solution, C_u

$$C_u = 57.7\% \text{ Al}$$
 (X2.7)

(X2.9)

X2.3 Calculations

X2.3.1 Mass loss of corroded coating, W

$$W = W_1 - W_2 = 79.3586 - 78.7660 = 0.5926 g$$
 (X2.8)

X2.3.2 Mass of remaining uncorroded coating, Wu

$$W_u = W_2 - W_3 = 78.7660 - 75.0810 = 3.6850 \text{ g}$$

X2.3.3 Total mass of original coating, Wt

$$W_t = W + W_u = 0.5926 + 3.6850 = 4.2776 g$$
 (X2.10)

X2.3.4 Composition of corroded coating, C

$$CW + C_uW_u = C_1W_t$$
 (X2.11)

Rearranging gives

$$C = (C_1W_t - C_uW_u)/W$$
 (X2.12)

$$C = (55.0 \times 4.2776 - 57.7 \times 3.6850)/0.5926$$
 (X2.13)

$$C = 38.2 \% Al$$
 (X2.14)

X2.3.5 The density, D, of a 38.2 % Al-Zn alloy is 4.32 g/cm⁻³. In cases where alloy densities are not known, they can be estimated by linear interpolation of the component densities.

X2.3.6 Calculate the average thickness loss rate, L (corrosion rate per Eq 1).

$$L = (K \times W)/(A \times T \times D) \tag{X2.15}$$

where K is given in 8.1.2 as 8.76×10^7

 $L = (8.76 \times 10^{7} \times 0.5926)/(300 \times 183648 \times 4.32)$

L = 0.218 micrometres per year

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Outdoor Atmospheric Corrosion, STP 1421, H. E. Townsend, Ed., American Society for Testing and MAterials, West Conshohocken, PA, 2002, pp. 284-291.

LAMPIRAN 2

ASTM Standard G 39 – 03,

Standard Practice for Preparation and Use of Bent-Beam

Stress-Corrosion Test Specimens





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Standard Practice for Preparation and Use of Bent-Beam Stress-Corrosion Test Specimens¹

This standard is issued under the fixed designation G 39; 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 designing, preparing, and using bent-beam stress-corrosion specimens.

1.2 Different specimen configurations are given for use with different product forms, such as sheet or plate. This practice applicable to specimens of any metal that are stressed to levels less than the elastic limit of the material, and therefore, the applied stress can be accurately calculated or measured (see Note 1). Stress calculations by this practice are not applicable to plastically stressed specimens.

Note 1—It is the nature of these practices that only the applied stress can be calculated. Since stress-corrosion cracking is a function of the total stress, for critical applications and proper interpretation of results, the residual stress (before applying external stress) or the total elastic stress (after applying external stress) should be determined by appropriate nondestructive methods, such as X ray diffraction (1).²

- 1.3 Test procedures are given for stress-corrosion testing by exposure to gaseous and liquid environments.
- 1.4 The bent-beam test is best suited for flat product forms, such as sheet, strip, and plate. For plate material the bent-beam specimen is more difficult to use because more rugged specimen holders must be built to accommodate the specimens. A double-beam modification of a four-point loaded specimen to utilize heavier materials is described in 10.5.
- 1.5 The exposure of specimens in a corrosive environment is treated only briefly since other practices deal with this aspect, for example, Specification D 1141, and Practices G 30, G 36, G 44, G 50, and G 85. The experimenter is referred to ASTM Special Technical Publication 425 (2).
- 1.6 The bent-beam practice generally constitutes a constant strain (deflection) test. Once cracking has initiated, the state of stress at the tip of the crack as well as in uncracked areas has changed, and therefore, the known or calculated stress or strain values discussed in this practice apply only to the state of stress existing before initiation of cracks.
- 1.7 The values stated in SI units are to be regarded as standard. The inch-pound equivalents in parentheses are pro-

vided for information

1.8 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 more specific safety hazard information see Section 7 and 12.1.)

2. Referenced Documents

- 2.1 ASTM Standards:
- D 1141 Specification for Substitute Ocean Water³
- G 30 Practice for Making and Using U-Bend Stress Corrosion Test Specimens⁴
- G 36 Practice for Performing Stress-Corrosion Cracking Tests in a Boiling Magnesium Chloride Solution⁴
- G 44 Practice for Evaluating Stress Corrosion Cracking Resistance of Metals and Alloys by Alternate Immersion in 3.5 % Sodium Chloride Solution⁴
- G 50 Practice for Conducting Atmospheric Corrosion Tests on Metals⁴
- G 85 Practice for Modified Salt Spray (Fog) Testing⁴
- 2.2 NACE Documents:5
- NACE TM0177-96 Laboratory Testing of Metals for Resistance to Specific Forms of Environmental Cracking in H₂S Environments

3. Terminology

- 3.1 Definitions of Terms Specific to This Standard:
- 3.1.1 stress-corrosion cracking—a cracking process requiring the simultaneous action of a corrodent and sustained tensile stress. This excludes corrosion-reduced sections that fail by fast fracture. It also excludes intercrystalline or transcrystalline corrosion which can disintegrate an alloy without either applied or residual stress.
- 3.1.2 cracking time—the time elapsed from the inception of test until the appearance of cracking.
- 3.1.2.1 Discussion—1 The test begins when the stress is applied and the stressed specimen is exposed to the corrosive environment, whichever occurs later.
 - 3.1.2.2 Discussion-2 The specimen is considered to have

¹ This practice is under the jurisdiction of ASTM Committee G-1 on Corrosion of Metals, and is the direct responsibility of Subcommittee G01.06 on Stress Corrosion Cracking and Corrosion Fatigue.

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² The boldface numbers in parentheses refer to the list of references appended to this practice.

³ Annual Book of ASTM Standards, Vol 11.02.

⁴ Annual Book of ASTM Standards, Vol 03.02.

⁵ Available from National Association of Corrosion Engineers, Int., P. O. Box 218340, Houston, TX 77218–8340.

failed when cracks are detected. Presence of cracks can be determined with or without optical, mechanical, or electronic aids. However, for meaningful interpretation, comparisons should be made only among tests employing crack detection methods of equivalent sensitivity.

4. Summary of Practice

4.1 This practice involves the quantitative stressing of a beam specimen by application of a bending stress. The applied stress is determined from the size of the specimen and the bending deflection. The stressed specimens then are exposed to the test environment and the time required for cracks to develop is determined. This cracking time is used as a measure of the stress-corrosion resistance of the material in the test environment at the stress level utilized.

5. Significance and Use

5.1 The bent-beam specimen is designed for determining the stress-corrosion behavior of alloy sheets and plates in a variety of environments. The bent-beam specimens are designed for testing at stress levels below the elastic limit of the alloy. For testing in the plastic range U-bend specimens should be employed (see Practice G 30). Although it is possible to stress bent-beam specimens into the plastic range, the stress level cannot be calculated for plastically stressed three- and four-point loaded specimens as well as the double-beam specimens. Therefore, the use of bent-beam specimens in the plastic range is not recommended for general use.

6. Apparatus

6.1 Specimen Holders—Bent-beam specimens require a specimen holder for each specimen, designed to retain the applied stress on the specimen. Typical specimen holder configurations are shown schematically in Fig. 1.

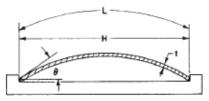
Note 2—The double-beam specimen, more fully described in 10.5, is self-contained and does not require a holder.

Note 3—Specimen holders can be modified from the constant deformation type shown in Fig. 1 to give a constant-load type of stressing. For instance, the loading bolt can be supplanted by a spring or dead-weight arrangement to change the mode of loading.

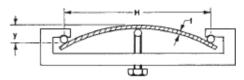
6.1.1 The holder shall be made of a material that would withstand the influence of the environment without deterioration or change in shape.

Note 4—It should be recognized that many plastics tend to creep when subjected to sustained loads. If specimen holders or insulators are made of such materials, the applied stress on the specimen may change appreciably with time. By proper choice of holder and insulator materials, however, many plastics can be used, especially in short-time tests.

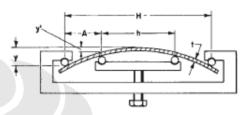
6.1.2 When the stress-corrosion test is conducted by immersion in an electrolyte, galvanic action between specimen and holder (or spacer) shall be prevented (see Note 5). This is accomplished by (1) making the holder of the same material as the individual specimens, (2) inserting electrically insulating materials between specimen and holder at all points of contact (see Note 4), (3) making the entire holder out of a nonmetallic material (see Note 4), or (4) coating the holder with an electrically nonconducting coating that effectively prevents contact between holder and electrolyte.



() TWO-POINT LOADED SPECIMEN



(b) THREE-POINT LOADED SPECIMEN



(c) FOUR-POINT LOADED SPECIMEN

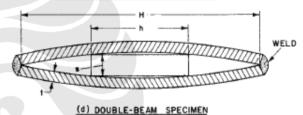


FIG. 1 Schematic Specimen and Holder Configurations

6.1.3 Crevice corrosion may occur in an electrolyte at contact points between specimen and holder (or spacer). In these instances the critical areas should be packed with a hydrophobic filler (such as grease or wax).

Note 5—In atmospheres (gas) galvanic action between specimen and holder either does not exist or is confined to a very small area as experienced in outdoor exposure tests.

6.2 Stressing Jigs—Three-point and four-point loaded specimen holders, Fig. 1 (b and c), contain a stressing feature in the form of a loading screw. To stress two-point loaded specimens (Fig. 1(a)), a separate stressing jig shall be used. A convenient stressing jig is shown in Fig. 2.

Note 6—The double-beam specimen, described in 10.5, requires a mechanical or hydraulic stressing frame (a universal tension testing machine can also be used) as well as welding equipment.

6.3 Deflection Gages—Deflection of specimens is determined by separate gages or by gages incorporated in a loading apparatus as shown in Fig. 3. In designing a deflection gage to suit individual circumstances care must be taken to reference the deflection to the proper support distance as defined in 10.2-10.5.

7. Hazards

7.1 Bent-beam specimens made from high-strength materials may exhibit high rates of crack propagation and a specimen



FIG. 2 Stressing Jig and Two-Point Loaded Specimen with Holder (approximately ¼ actual size)

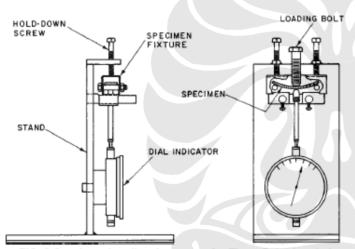


FIG. 3 Specimen Loading Apparatus for Three-Point Loaded Beam Specimens with Integral Deflection Gage

may splinter into several pieces. Due to high stresses in a specimen, these pieces may leave the specimen at high velocity and can be dangerous. Personnel installing and examining specimens should be cognizant of this possibility and be protected against injury.

8. Sampling

- 8.1 Test specimens shall be selected so that they represent the material to be tested. In simulating a service condition, the direction of load application in the specimen shall represent the anticipated loading direction in service with respect to processing conditions, for example, rolling direction.
- 8.2 Paragraphs 7.4 and 7.5 deal specifically with specimen selection as related to the original material surface.

9. Test Specimen

9.1 The bent-beam stress-corrosion specimens shall be flat strips of metal of uniform, rectangular cross section, and uniform thickness.

- 9.2 The identification of individual specimens should be permanently inscribed at each end of the specimen because this is the area of lowest stress and cracking is not expected to be initiated by the identification markings. If stenciling is used for identification, this shall be done only on softened material before any hardening heat treatments to prevent cracking in the stenciled area. Care must be taken to prevent the identification from being obliterated by corrosion.
- 9.3 Mechanical properties should be determined on the same heat-treatment lot from which stress-corrosion specimens are obtained.
- 9.4 The specimens can be cut from sheet or plate in such a fashion that the original material surface is retained. This procedure is recommended when it is desired to include the effect of surface condition in the test.
- 9.5 If, however, it is desired that surface conditions should not influence the test results of several materials with different surface conditions, the surfaces of all specimens must be prepared in the same way. It is recommended that grinding or machining to a surface finish of at least 0.7 µm (30 µin.) and to a depth of at least 0.25 mm (0.01 in.) be utilized for surface preparation. It is desirable to remove the required amount of metal in several steps by alternately grinding opposite surfaces. This practice minimizes warpage due to residual stresses caused by machining. All edges should be similarly ground or machined to remove cold-worked material from previous shearing. Chemical or electrochemical treatments that produce hydrogen on the specimen surface must not be used on materials that may be subject to embrittlement by hydrogen or that react with hydrogen to form a hydride.
- 9.6 Immediately before stressing, the specimens should be degreased and cleaned to remove contamination that occurred during specimen preparation. Only chemicals appropriate for the given metal or alloy should be used. Care must be exercised not to contaminate cleaned specimens. Also, it is suggested that specimens be examined for cracks before exposure to the test environment.

10. Stress Calculations

- 10.1 The equations given in this section are valid only for stresses below the elastic limit of the material. At stresses above the elastic limit but below the engineering yield strength (0.2 % offset) only a small error results from use of the equations (see Note 1). The equations must not be used above the yield strength of the material. The following paragraphs give relationships used to calculate the maximum longitudinal stress in the outer fibers of the specimen convex surface. Calculations for transverse stress or edge-to-edge variation of longitudinal stress are not given; the specimen dimensions are chosen to minimize these stresses consistent with convenient use of the specimens. The specimen dimensions given here can be modified to suit specific needs. However, if this is done the approximate specimen proportions should be preserved to give a similar stress distribution (for instance, if the length is doubled the width should be doubled also).
- 10.1.1 When specimens are tested at elevated temperatures, the possibility of stress relaxation should be investigated. Relaxation can be estimated from known creep data for the specimen, holder, and insulating materials. Differences in

thermal expansion also should be considered.

10.1.2 The applied stress is determined by specimen dimensions and the amount of bending deflection. Thus, the errors in the applied stress are related to those inherent in the use of measuring instruments (micrometers, deflection gages, strain gages, and so forth). For the two-point loaded specimens, most measured values lie within 5 % of the values calculated in accordance with the procedures given in 10.2.1-10.2.3, as reported by Haaijer and Loginow (4). The calculated stress applies only to the state of stress before initiation of cracks. Once cracking is initiated, the stress at the tip of the crack, as well as in uncracked areas, has changed.

10.2 Two-Point Loaded Specimens—This specimen can be used for materials that do not deform plastically when bent to (L-H)/H=0.01 (see section 10.2.5). The specimens shall be approximately 25 by 254-mm (1 by 10-in.) flat strips cut to appropriate lengths to produce the desired stress after bending as shown in Fig. 1(a).

10.2.1 Calculate the elastic stress in the outer fiber at midlength of the two-point loaded specimens from relationships derived from a theoretically exact large-deflection analysis (4), as follows:

$$\epsilon = 4(2E - K) \left[\frac{k}{2} - \frac{2E - K}{12} \left(\frac{t}{H} \right) \right] \frac{t}{H}$$
 (1)

and

$$(L - H)/H = [K/2E - K)] - 1$$
 (2)

where:

L = length of specimen,

H = distance between supports (holder span),

t = thickness of specimen,

ε = maximum tensile strain,

 $K = \int_0^{\pi/2} (1 - k^2 \sin^2 z)^{-1/2} dz$ (complete elliptic integral of the first kind)

the first kind), $E = \int_0^{\pi/2} (1 - k^2 \sin^2 z)^{1/2} dz$ (complete elliptic integral of the second kind),

 $k = \sin \theta/2$,

 maximum slope of the specimen, that is, at the end of the specimen, and

z = integration parameter (4).

10.2.2 The mathematical analysis establishes that Eq 1 and Eq 2 define the relationship between the strain ϵ and (L-H)/H in parameter form. The common parameter in these equations is the modulus k of the elliptic integrals. Thus, the following procedure can be used to determine the specimen length L that is required to produce a given maximum stress σ :

10.2.2.1 Divide the stress σ by the modulus of elasticity E_m to determine the strain ϵ .

$$\epsilon = \sigma / E_m$$

10.2.2.2 From Eq 1 determine the value of k corresponding to the required value of ϵ .

10.2.2.3 By using appropriate values of k evaluate Eq 2 for L. To facilitate calculations a computer can be used to generate a table for a range of strain ϵ and H/t with resultant values of (L-H)/H.

10.2.3 Calculate the deflection of the specimen as follows:

$$y/H = k/(2E - K) \tag{3}$$

where:

y = maximum deflection.

The other quantities are given in 10.2.1.

This relationship can be used as a simple check to ensure that the maximum stress does not exceed the proportional limit. If it should exceed the proportional limit, the measured deflection will be greater than that calculated from Eq 3.

10.2.4 As an alternative method the following approximate relationship can be used for calculating specimen length:

$$L = (ktE/\sigma) \sin^{-1}(H\sigma/ktE)$$
 (4)

where:

L = specimen length,

 $\sigma = \text{maximum stress}$

E = modulus of elasticity,

H = holder span,

t = thickness of specimen, and

k = 1.280, an empirical constant.

This equation can be solved by computer, by trial and error, or by using a series expansion of the sine function. Eq 4 shall be used only when the quantity $(H\sigma/ktE)$ is less than 1.0.

10.2.5 Choose specimen thickness and length and holder span to obtain a value for (L-H)/H of between 0.01 and 0.50, thus keeping the error of stress within acceptable limits. A specimen thickness of about 0.8 to 1.8 mm (0.03 to 0.07 in.) and a holder span of 177.8 to 215.9 mm (7.00 to 8.50 in.) has been very convenient when working with very high strength steels and aluminum alloys with applied stresses ranging from about 205 MPa (30 ksi) for aluminum to 1380 MPa (200 ksi) for steel. The specimen dimensions given here can be modified to suit specific needs. However, if this is done, approximate dimensional proportions shall be preserved.

10.2.6 In two-point loaded specimens the maximum stress occurs at midlength of the specimen and decreases to zero at specimen ends.

10.2.7 The two-point loaded specimen is preferred to three-point loaded specimens because in many instances crevice corrosion of the specimen occurs at the central support of the three-point loaded specimen. Since this corrosion site is very close to the point of highest tension stress, it may cathodically protect the specimen and prevent possible crack formation or cause hydrogen embrittlement. Furthermore, the pressure of the central support at the point of highest load introduces biaxial stresses at the area of contact and could introduce tension stresses where normally compression stresses are present.

Note 7—Occasionally two-point loaded specimens having a nonuniform cross section are used for special purposes. A description of such a specimen is given by Wilson and Spier (5).

10.3 Three-Point Loaded Specimens—The specimen shall be a flat strip typically 25 to 51-mm (1 to 2-in.) wide and 127 to 254-mm (5 to 10-in.) long. The thickness of the specimen is usually dictated by the mechanical properties of the material and the product form available. Support the specimen at the ends and bend the specimen by forcing a screw (equipped with a ball or knife-edge tip) against it at the point halfway between the end supports in a fashion shown in Fig. 1(b). The specimen dimensions given here can be modified to suit specific needs.

However, if this is done, approximate dimensional proportions shall be preserved.

10.3.1 Calculate the elastic stress at midspan in the outer fibers of three-point loaded specimens from the relationship:

$$\sigma = 6Ety/H^2$$
(5)

where:

 σ = maximum tensile stress,

E = modulus of elasticity,

t =thickness of specimen,

v = maximum deflection, and

H = distance between outer supports.

10.3.2 The above relationship is based on small deflections (y/H less than 0.1). In sheet gage bent-beam specimens the deflections are usually large, and thus, the relationship is only approximate. To obtain more accurate stress values, use for calibration a prototype specimen equipped with strain gages. This prototype should have the same dimensions as the test specimens and should be stressed in the same way.

10.3.3 In three-point loaded specimens the maximum stress occurs at midlength of the specimen and decreases linearly to zero at the outer supports.

10.3.4 For limitation in the use of three-point loaded specimens see 10.2.7.

10.4 Four-Point Loaded Specimens—The specimen shall be a flat strip typically 25 to 51-mm (1 to 2-in.) wide and 127 to 254-mm (5 to 10-in.) long. The thickness of the specimen is usually dictated by the mechanical properties of the material and the product form available. Support the specimen at the ends and bend the specimen by forcing two inner supports against it in a fashion shown in Fig. 1(c). The two inner supports shall be located symmetrically around the midpoint between the outer supports. The specimen dimensions given here can be modified to suit specific needs. However, if this is done, approximate dimensional proportions shall be preserved.

10.4.1 Calculate the elastic stress for the midportion of the specimen (between contact points of the inner support) in the outer fibers of four-point loaded specimens from the following relationship:

$$\sigma = 12Ety/(3H^2 - 4A^2)$$
 (6)

where:

 $\sigma = \text{maximum tensile stress},$

E = modulus of elasticity,

t = thickness of specimen,

y = maximum deflection (between outer supports),

H = distance between outer supports, and

A = distance between inner and outer supports.

The dimensions are often chosen so that A = H/4.

10.4.2 An alternative method of calculating the elastic stress between the inner supports is as follows:

$$\sigma = 4Ety'/h^2$$
(7)

where:

h = distance between inner supports, and

y' = deflection between inner supports.

(This equation is a special case of 10.4.1 when A = 0.)

10.4.3 The above relationships are based on small deflections (y/H less than 0.1). In sheet-gage bent-beam specimens

the deflections are usually large, and thus, the relationships are only approximate. To obtain more accurate stress values, use for calibration a prototype specimen equipped with strain gages. This prototype specimen should have the same dimensions as the test specimens and should be stressed in the same way.

10.4.4 In four-point loaded specimens the maximum stress occurs between the contact points with the inner supports; in this area the stress is constant. From the inner supports the stress decreases linearly toward zero at the outer supports.

10.5 Double-Beam Specimen—The specimen shall consist of two flat strips 25 to 51-mm (1 to 2-in.) wide and 127 to 254-mm (5 to 10-in.) long. Bend the strips against each other over a centrally located spacer until both ends of the specimens touch. Hold them in this position by welding the ends together as shown in Fig. 1(d) (see Note 8). An equivalent procedure for bolted specimens is described on pp. 319–321 of Ref (2).

Note 8—If the test is to be conducted in an electrolyte, the spacer shall be made of the same material as the specimen (or of an electrically nonconducting material such as glass, ceramic, and so forth) to prevent galvanic action between specimen and spacer. See also 6.1.2 and Note 4 and Note 5.

10.5.1 Calculate the elastic stress for the midportion of the specimen (between contact points of the spacer) in the outer fibers of the doublebeam specimens from the following relationship:

$$\sigma = \frac{3Ets}{H^{2}[1 - (h/H)][1 + (2h/H)]}$$
(8)

where:

σ = maximum tensile stress,

E = modulus of elasticity,

t = thickness of specimen strip,

s = thickness of spacer,

H = see Fig. 1(d), and

h = length of spacer.

10.5.2 When the length of the spacer h is chosen so that H = 2h the equation in 10.5.1 is simplified to:

$$\sigma = 3Ets/H^2$$

10.5.3 The above relationships are based on small deflections (s/H being less than 0.2). In sheet-gage bent-beam specimens the deflections are usually large, and thus, the relationships are only approximate. To obtain more accurate stress values, use for calibration a prototype specimen equipped with strain gages. The prototype specimen should have the same dimensions as the test specimens and should be stressed in the same way.

10.5.4 In double-beam specimens the maximum stress occurs between the contact points with the spacer; in this area the stress is constant. From the contact with the spacer the stress decreases linearly toward zero at the ends of specimens.

11. Choice of Test Conditions

11.1 The purpose of stress-corrosion testing is to simulate on a small scale the conditions (materials, stress, and environment) that exist in an engineering application. The stresses in an engineering structure can be varied: operational (design) stresses and residual stresses (from heat treatment or fabrication). Residual stresses are frequently the more important, primarily because current design practices and close control of processes have kept operational stresses well below the yield strength of the metal in use. On the other hand, magnitude and direction of residual stresses frequently are difficult to predict and also difficult to measure. Depending on the degree of restraint, residual stresses may even exceed the initial yield strength of the material.

- 11.2 Generally stress-corrosion testing falls into two broad categories: (1) evaluation of materials for a specific application and (2) comparison of the relative behavior of several materials or environments.
- 11.2.1 To evaluate materials for specific applications the testing conditions should be representative of the most severe conditions to which the materials would be subjected in service. Testing at nominal or design conditions could be misleading. An engineering structure, because of residual stresses, is expected to be stressed to its yield strength at some points even if the design stress for that structure is appreciably below yield strength. Thus, the use of the elastically stressed bent-beam specimens for materials evaluation is of limited value.
- 11.2.2 To compare materials or environments for relative stress-corrosion behavior, the test conditions may be only severe enough to produce varying degrees of cracking in the alloys of interest, in mechanical or thermal treatments used, or in sensitivity to specific environments investigated. By testing a set of specimens at a series of stress levels, the stress dependence of alloys can be assessed. The bent-beam specimen is very well suited for establishing the relative merits of several alloys for the relative severity of several environments.
- 11.3 Ideally, the environmental test conditions should be the same that would prevail in the intended use of the alloys. In choosing a set of test conditions, it is important that they (environment and stress) be well defined and reproducible. A detailed discussion is given by Loginow (6).
- 11.4 The presence of a machined notch in the middle of the tension side of a bent beam will induce a severe triaxial stress state at the root of the notch. The actual bending stress there

will be greater by a concentration factor dependent on the notch geometry, than the minimal test stress, and generally, may be expected to be in the range of plastic stain. Advantages of such a notched specimen include the probable localized cracking in the notch and an acceleration of failure. However, unless directly related to practical conditions of usage, misleading failures may ensue.

11.4.1 Another type of stress concentration at the site of two drilled holes located half way between the end supports of a three-point loaded bent beam has been used in the evaluation of metals for oilfield equipment. Details on the preparation and use of this specimen are described in NACE TM0177-96. Laboratory test data for carbon and low-alloy steels have been found to correlate with field data (7).

12. Specimen Exposure

- 12.1 Expose the stressed specimens to the environment (gaseous or liquid) of interest. This can be accomplished by mounting the specimen holders on appropriate racks and exposing the entire rack to the environment. A typical atmospheric exposure rack is shown in Fig. 4. As noted in 7.1, bent-beam specimens may break violently and thus cause injury. To protect personnel and to prevent specimen loss, drill holes in specimen ends and holders and secure the specimens by wires to their holders.
- 12.2 Determination of cracking time is a subjective procedure involving visual examination that under some conditions can be very difficult, as noted in Section 13, and depends on the skill and experience of the inspector.
- 12.3 Laboratory Exposure of Bent Beams— In both alternate and sustained immersion of bent beams, avoid galvanic corrosion between fixtures and specimens as discussed in 6.1.2 and Note 4 and Note 5. It should be recognized that, at points of contact between specimen and fixture, crevice corrosion may occur on some materials, which in turn may result in galvanic protection of the stressed area. If this condition occurs, either eliminate the crevice or consider a different kind of specimen. In alternate immersion, expose the specimen to allow complete drainage and drying of the surface. In immersion tests, arrange the specimens so as to prevent contact with

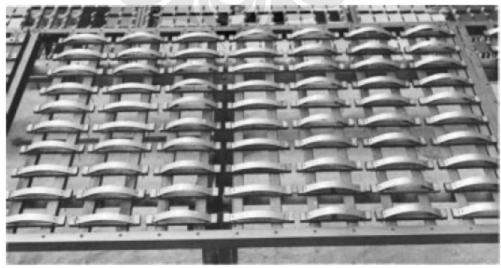


FIG. 4 Bent-Beam Specimens on Atmospheric Exposure Rack



each other. In both sustained and alternate immersion, the solution volume should be large enough to prevent depletion of corrosive agents. In elevated-temperature tests, make arrangements to reflux the solution to maintain a constant concentration.

12.4 Atmospheric Exposure of Bent Beams— Expose the specimens in an area that is representative of the atmospheric conditions of interest.

13. Inspection of Specimens

- 13.1 As continuous observation of specimens is usually impractical, inspect specimens for appearance of cracks at predetermined time intervals. These intervals are usually increased as the test progresses because the logarithms of observed cracking times are often normally distributed as described by Loginow (6) and by Booth et al (8).
- 13.2 Determine presence of cracks by visual observation, usually with the aid of a 5 to 10 power magnifying glass. If the specimen contains only one or a few cracks, the shape of the bend can be considerably changed, predominantly by kinking; this feature helps in identifying cracked specimens. However, if many cracks are present, a change in shape may not be apparent. It should also be noted that presence of voluminous corrosion products may obscure cracks, thus making a careful examination mandatory. In these instances metallographic sectioning of the specimen may be necessary to detect cracks.

14. Report

- 14.1 Results of stress-corrosion tests with bent-beam specimens are expressed as the time to produce failure by cracking or as the fraction of specimens that have cracked in a fixed time. In addition to the cracking time the following data shall be reported:
 - 14.1.1 Specimen identification,
 - 14.1.2 Material name or specification code,
 - 14.1.3 Chemical composition,
 - 14.1.4 Heat treatment,
 - 14.1.5 Mechanical properties,
- 14.1.6 Type and orientation of specimen used and surface condition (hot rolled, cold rolled, machined, surface ground, and so forth),
 - 14.1.7 Applied stress (and residual stress, if known),
- 14.1.8 Details of specimen preparation if different from those specified here (or if not specified),
 - 14.1.9 Detailed description of test environment, and
- 14.1.10 Remarks concerning the size and appearance of cracks may be included.

15. Keywords

15.1 bent-beam; constant deformation; constant load; elastic strain; quantitative stress; stress-corrosion cracking; stress-corrosion test specimen

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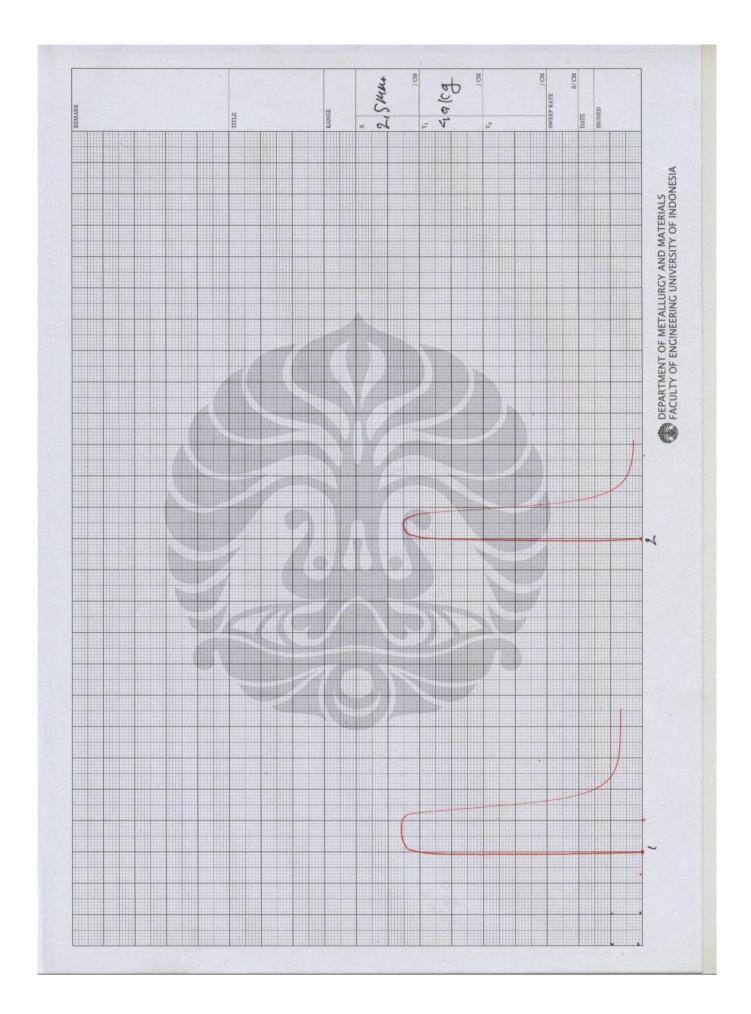
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LAMPIRAN 3

HASIL UJI TARIK MATERIAL UJI ALUMINIUM ALLOY 1xxx





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|-----------------|------------------|--|---|--|--|---------------------------------------|------------------|----------------|-----------------|---|
| Nama customer | | | 1 | No. Kontrak: | | | | | Tanggal Uji: | |
| Mesin Uji | Servopulser Shii | Servopulser Shimadzu Kapasitas 20 ton | 20 ton | Teknisi Penguji | | | | | Paraf Teknisi : | |
| Bahan | AL | | | Standar/Metode Uji | · Ultr | | | | Tanggai bah | Tanggal bahan diterima : . |
| Identitas Bahan | PLAT | | | | | | | | | |
| Kode Sampel | Bentuk | Dimensi Sampel (mm) | Luas perampang (mm²) | Panjang ukur (mm) | Pu (kg) | Py (kg) | AL (mm) | ou (ke/mm²) | cy (ke/mm²) | e (%) Ket. |
| | Rod/Tube | | | | | | | | | |
| AL | P elat | t=0,90 | 23,40 | 05 | 304 | 280 | 4,20 | 13,00 | 12,00 | 8,40 |
| 41 | Rod/Tube | | | | | | ř | | | |
| 110 | The Pelat | t=0,90 | 23.35 | 05 | 300 | 278 | 2,80. | 13,00 | 13,00 12,00 | 5,60 |
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LAMPIRAN 4

HASIL UJI KOMPOSISI MATERIAL UJI ALUMINIUM ALLOY 1xxx





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<u>LAPORAN PENGUJIAN KOMPOSISI KIMIA</u> <u>COMPOSITION TEST REPORT</u>

Hal 1dari 1

| No Laporan | 1177 | Bahan | |
|--------------|------------------------|-------------------|--|
| Report Nr | 11// | Material | AI |
| Pemakai Jasa | ELRIANDRI | Identitas Bahan | ** *********************************** |
| Custumer | ELKIANDKI | Material Identity | Al anode |
| Alamat | | Tanggal Terima | 0.1.5 |
| Address | | Receiving Date | 04 Desember 2008 |
| No Kontrak | 1177/PT.02/FT04/P/2008 | Standar | Lomas Lead |
| Contract Nr. | 11///F1.02/F104/P/2008 | Standard | ASTM A751 |
| Tanggal Uji | 09 Desember 2008 | Mesin Uji | 0 |
| Date of Test | 09 Describer 2008 | Testing machine | Optical Emission Spectrometer |

| Kode Sampel Sample Code | Al (%) | Si (%) | Fe (%) | Cu (%) | Mn (%) | Mg (%) | Zn (%) |
|----------------------------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|
| | 99.1 | 0.278 | 0.327 | 0.180 | < 0.001 | < 0.0001 | < 0.005 |
| Al anode | Cr (%) | Ni (%) | Ti (%) | Be (%) | Ca (%) | In (%) | Pb (%) |
| | < 0.001 | < 0.005 | 0.008 | < 0.0001 | < 0.0005 | < 0.010 | < 0.002 |

Depok, 09 Desember 2008 LABORATORIUM UJI MATERIAL

Manajer Teknis,

(Ahmad Ivan Karayan, ST, M.Eng)

CIMPFA

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