



Designation: G 37 – 98

## Standard Practice for Use of Mattsson's Solution of pH 7.2 to Evaluate the Stress-Corrosion Cracking Susceptibility of Copper-Zinc Alloys<sup>1</sup>

This standard is issued under the fixed designation G 37; 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 This practice covers the preparation and use of Mattsson's solution of pH 7.2 as an accelerated stress-corrosion cracking test environment for brasses (copper-zinc base alloys). The variables (to the extent that these are known at present) that require control are described together with possible means for controlling and standardizing these variables.

1.2 This practice is recommended only for brasses (copper-zinc base alloys). The use of this test environment is not recommended for other copper alloys since the results may be erroneous, providing completely misleading rankings. This is particularly true of alloys containing aluminum or nickel as deliberate alloying additions.

1.3 This practice is intended primarily where the test objective is to determine the relative stress-corrosion cracking susceptibility of different brasses under the same or different stress conditions or to determine the *absolute* degree of stress corrosion cracking susceptibility, if any, of a particular brass or brass component under one or more specific stress conditions. Other legitimate test objectives for which this test solution may be used do, of course, exist. The tensile stresses present may be known or unknown, applied or residual. The practice may be applied to wrought brass products or components, brass castings, brass weldments, and so forth, and to all brasses. Strict environmental test conditions are stipulated for maximum assurance that apparent variations in stress-corrosion susceptibility are attributable to real variations in the material being tested or in the tensile stress level and not to environmental variations.

1.4 This practice relates solely to the preparation and control of the test environment. No attempt is made to recommend surface preparation or finish, or both, as this may vary with the test objectives. Similarly, no attempt is made to recommend particular stress-corrosion test specimen configurations

or methods of applying the stress. Test specimen configurations that may be used are referenced in Practice G 30 and STP 425.<sup>2</sup>

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* (For more specific safety hazard statements see Section 8.)

### 2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water<sup>3</sup>

G 30 Practice for Making and Using the U-Bend Stress-Corrosion Specimens<sup>4</sup>

### 3. Summary of Practice

3.1 The practice consists of completely and continuously immersing a stressed test specimen in an aqueous solution containing 0.05 g-atom/L of  $\text{Cu}^{++}$  and 1 g-mol/L of  $\text{NH}_4^+$  and of pH 7.2. The copper is added as  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  and the  $\text{NH}_4^+$  as a mixture of  $\text{NH}_4\text{OH}$  and  $(\text{NH}_4)_2\text{SO}_4$ . The ratio of these latter two compounds is adjusted to give the desired pH. Exposure time, criterion of failure, and so forth, are variable and not specifically recommended.

### 4. Significance and Use

4.1 This test environment is believed to give an accelerated ranking of the relative or *absolute* degree of stress-corrosion cracking susceptibility for different brasses. It has been found to correlate well with the corresponding service ranking in environments that cause stress-corrosion cracking, which is thought to be due to the combined presence of traces of moisture and ammonia vapor. The extent to which the accelerated ranking correlates with the ranking obtained after

<sup>1</sup> 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 Environmentally Assisted Cracking.

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<sup>2</sup> *Stress Corrosion Testing, ASTM STP 425*, ASTM (Although currently out of print, copies may be obtained from University Microfilms, Inc., 300 North Zeeb Rd., Ann Arbor, MI 48106).

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 03.02.

long-term exposure to environments containing corrosives other than ammonia is not at present known. Examples of such environments may be severe marine atmospheres ( $\text{Cl}^-$ ), severe industrial atmospheres (predominantly  $\text{SO}_2$ ), and super-heated ammonia-free steam.

4.2 It is not possible at present to specify any particular time to failure (defined on the basis of any particular failure criteria) in pH 7.2 Mattsson's solution of pH 7.2 that corresponds to a distinction between acceptable and unacceptable stress-corrosion behavior in brass alloys. Such particular correlations must be determined individually.

4.3 Mattsson's solution of pH 7.2 may also cause stress independent general and intergranular corrosion of brasses to some extent. This leads to the possibility of confusing stress-corrosion failures with mechanical failures induced by corrosion-reduced net cross sections. This danger is particularly great with small cross section specimens, high applied stress levels, long exposure periods, and stress-corrosion resistant alloys. Careful metallographic examination is recommended for correct diagnosis of the cause of failure. Alternatively, unstressed control specimens may be exposed to evaluate the extent to which stress independent corrosion degrades mechanical properties.

## 5. Apparatus

5.1 Any suitable inert container may be used to hold the test solution and test specimens during exposure. Glass or plastic is highly recommended. The container should be fitted with a removable top to reduce evaporation during test, thus preventing dust and other particulate matter from entering the environment, and facilitating periodic inspection of the specimens. The top should not, however, be airtight, but instead should permit reasonably free access of laboratory air to the surface of the environment. The container and top should not be transparent and should be fully opaque to light.

5.2 *Specimen holders* should be designed to insulate electrically the test specimens from each other and from any other bare metal. They should also be completely inert to the test environment so that leaching of soluble components or direct reaction with the test environment will not interfere with the test. The shape and form of the specimen holders and supports should be such that they do not interfere with free access to the test environment to the test specimen and its stressed surfaces.

## 6. Reagents and Materials

6.1 Reagent grade copper sulfate crystals ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ), ammonium sulfate crystals ( $(\text{NH}_4)_2\text{SO}_4$ ), and concentrated ammonium hydroxide solution ( $\text{NH}_4\text{OH}$ ) (28 to 30 %  $\text{NH}_3$  assayed and 0.90 sp gr) conforming to the specifications of the Committee on Analytical Reagents of the American Chemical Society shall be used.<sup>5</sup>

<sup>5</sup> *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.

6.2 *Purity of Water*— Reagent water Type IV (Specification D 1193) shall be used to prepare the test solution.

## 7. Test Solution

7.1 The concentration of the test solution shall be 0.05 g-atom/L with respect to  $\text{Cu}^{++}$  and 1.0 g-mol/L with respect to  $\text{NH}_4^+$ . The pH of the test solution shall be  $7.2 \pm 0.3$ ,  $-0.1$  pH.

7.2 The test solution shall be prepared by completely dissolving  $590.0 \pm 1.0$  g of  $(\text{NH}_4)_2\text{SO}_4$  in 4 L of water and by completely dissolving  $125.0 \pm 0.5$  g of  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  in 1 L of water. These two solutions should then be thoroughly mixed and  $71.0 \pm 0.2$  mL of  $\text{NH}_4\text{OH}$  solution added, preferably with a buret. Finally, the whole should be diluted to  $10.0 \pm 0.1$  L and allowed to age for 48 to 96 h in the test container prior to use. It is not recommended that the solution be stored for extended periods or used without the specified aging. Smaller or larger volumes of solution can be prepared using lesser amounts of reagents in the same proportions.

7.3 After aging, the pH of the test solution should be measured. If outside the range specified above, the pH may be adjusted to within the range 7.1 to 7.5 by the addition of fresh pH 4 or pH 10 Mattsson's solution. Addition of  $\text{NH}_4\text{OH}$  or  $\text{H}_2\text{SO}_4$  to adjust pH is not recommended since the concentration of the various soluble ions can be considerably altered.

7.4 Temperature control of the test solution is not recommended. Instead, the room air temperature should be controlled at  $21 \pm 3^\circ\text{C}$  ( $70 \pm 5^\circ\text{F}$ ) and the test solution allowed to reach its equilibrium temperature with the air. No room air relative humidity control is recommended, and temperature recording is not mandatory.

7.5 The volume of test solution used should be sufficiently large to avoid any appreciable change in corrosivity due to either exhaustion of corrosive constituents or accumulation of corrosion products. An arbitrary minimum ratio between the volume of test solution and the exposed specimen surface area of  $3 \text{ L}/\text{dm}^2$  ( $30 \text{ mL}/\text{cm}^2$ ) is recommended.

7.6 The test solution should be changed after every seven days of specimen exposure time by transferring the test specimens to a new batch of 48-h aged test solution. Evaporation losses should be replenished with reagent water Type IV (Specification D 1193) as frequently as deemed necessary. As far as possible, the air above the solution should be static to minimize water and ammonia evaporation. Forced ventilation should be avoided. It has not been found necessary to check or adjust the pH of the test solution during test after the initial 48-h aging period.

## 8. Hazards

8.1 The test solution presents no undue safety hazard. It is recommended, however, that rubber gloves and shatterproof goggles be worn during inspection.

8.2 The solution contains copper and is poisonous and should not be ingested.

8.3 Should any of the solution come into contact with the eyes, qualified medical aid should be obtained immediately.

8.4  $(\text{NH}_4)_2\text{SO}_4$  has been reported to be allergenic. Repeated short-time skin contact with the solution over extended periods of time should be avoided.

8.5 The fumes given off by the test solution contain ammonia. The least detectable ammonia odor corresponds to a concentration of 50 ppm; 100 ppm can be tolerated for several hours without serious disturbance; 700 ppm causes immediate eye irritation; and greater than 5000 ppm can be lethal. The test should therefore be run in a well ventilated area.

## 9. Calibration and Standardization

9.1 When a new test facility is established, calibration tests should be conducted to determine how the results obtained compare with published data.

9.2 A suitable supply of control material which can be exposed periodically to confirm the reproducibility of the test procedure should be available.

## 10. Procedure

10.1 Completely and continuously immerse the test specimens in the test solution with the exception of periodic withdrawals for inspection or measurement. Gently rinse the specimens in distilled water after each withdrawal and when the test solution is being changed.

10.2 The duration of the test will be determined solely by the material being tested. Highly susceptible alloys or highly stressed alloys may fail with extreme rapidity. Moderately stressed alloys may take considerable time to fail. Generally, failures are not observed after periods of greater than 1000 h. It is therefore recommended that this be the longest duration of the test.

## 11. Cleaning

11.1 After completion of the test, rinse the specimens well in distilled water and air dry.

11.2 Any other cleaning may be performed, depending on test objective and method of failure determination.

## 12. Report

12.1 The report shall include the following:

12.1.1 Chemical composition, thermal treatments, mechanical treatments, and mechanical properties of the material,

12.1.2 Microstructure and grain size of the material,

12.1.3 Size, type, and orientation of the test specimens,

12.1.4 Surface treatment and preparation procedures of the test specimens,

12.1.5 In configurations where the stress level is known, the applied tensile stress,

12.1.6 Ratio of test solution volume to exposed specimen surface area,

12.1.7 Total duration of the test and the frequency of inspection periods,

12.1.8 Measured pH of the test solution and the  $\text{NH}_4^+$  content if determined analytically,

12.1.9 Criterion used for definition of failure,

12.1.10 Number of specimens tested,

12.1.11 Details of the solution container, specimen loading jigs, and so forth, where these are in any way exceptional,

12.1.12 Method used to clean the test specimens after exposure, and

12.1.13 Any intentional or inadvertent deviations from the recommended test procedures and any other information that the investigator considers pertinent.

## 13. Keywords

13.1 accelerated stress-corrosion cracking test environment; continuous immersion; copper-ammonium sulfate solution; copper-zinc alloys

## ANNEX

### (Mandatory Information)

#### A1. PREPARATION OF MATTSSON'S SOLUTION OF pH 4 AND pH 10

A1.1 The preparation of Mattsson's solution of pH 4 and pH 10 is provided in this annex to enable the pH of Mattsson's solution, pH 7.2, to be adjusted correctly.

##### A1.2 *Mattsson's Solution of pH 4:*

66 g  $(\text{NH}_4)_2\text{SO}_4$

12.5 g  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  plus reagent water Type IV (Specification D 1193) to make 1 L.

A1.2.1 Proceed as for Mattsson's solution of pH 7.2. The pH is insensitive to composition and adjustment should not be required. The solution should be 1 M with respect to  $\text{NH}_3$  and 0.05 M with respect to  $\text{Cu}^{++}$ .

##### A1.3 *Mattsson's Solution of pH 10:*

15.2 g  $(\text{NH}_4)_2\text{SO}_4$

12.5 g  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$

52 mL of concentrated ammonium hydroxide solution ( $\text{NH}_4\text{OH}$ ) (28 to 30 %  $\text{NH}_3$  assayed and 0.90 sp gr) plus reagent water Type IV (Specification D1193) to make 1L.

A1.3.1 Proceed as for Mattsson's solution of pH 7.2. The pH is insensitive to composition and adjustment should not be required. The solution should be 1 M with respect to  $\text{NH}_3$  and 0.05 M with respect to  $\text{Cu}^{++}$ .

A1.4 Both of the above solutions should be aged for 48 to 96 h before use.



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