



Standard Test Method for Ammonia Vapor Test for Determining Susceptibility to Stress Corrosion Cracking in Copper Alloys¹

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1. Scope

1.1 This test method describes a procedure to determine the presence of residual stresses in wrought copper alloy products that may lead to stress corrosion cracking. An ammonia vapor atmosphere is used as an accelerated test.

1.2 This test method is only suitable for products fabricated from copper alloys that are known to be susceptible to stress corrosion cracking in ammonia vapor atmospheres. It is intended to create an environmental condition of reproducible severity.

NOTE 1—It is well known that the critical step in the cracking mechanism is the development of an environment in the condensate film that occurs on the surface of the test specimen, and is rich in copper complex ions.

1.3 The severity of this test method depends upon the pH of the corrosive solution. In [Annex A1](#) are given four different atmospheres to which the product may be exposed, and the appropriate pH of the solution to be used for the test, depending on the risk level associated with the intended application.

1.3.1 The appropriate pH value for the test shall be specified in the product specification, or as per established agreement between the supplier and purchaser, with respect to the alloy and its intended application.

1.4 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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.*

¹ This test method is under the jurisdiction of ASTM Committee B05 on Copper and Copper Alloys and is the direct responsibility of Subcommittee B05.06 on Methods of Test.

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2. Referenced Documents

2.1 *ASTM Standards*:²

[B154 Test Method for Mercurous Nitrate Test for Copper Alloys](#)

[B846 Terminology for Copper and Copper Alloys](#)

[D1193 Specification for Reagent Water](#)

3. Terminology

3.1 For definitions of terms related to copper and copper alloys, refer to Terminology [B846](#).

3.2 *Definitions*:

3.2.1 *applied stress, n*—stress in a body as a result of application of an external load.

4. Summary of Test Method

4.1 The prepared test specimen is placed in a closed container and exposed to ammonia vapor with a specific pH at ambient temperature for 24 h. Upon removal from the test atmosphere, the test specimen is examined for the presence of cracks.

5. Significance and Use

5.1 This test method is an accelerated test to determine if a copper alloy product will be susceptible to stress-corrosion cracking when exposed to a particular atmospheric condition during service with the appropriate risk level—see [Annex A1](#).

5.1.1 This test method is generally intended to determine if a copper alloy product will crack because of internal stresses when subjected to the test, and is not intended for testing assemblies under applied stress. If used for this purpose, it shall be for information only and not a cause for rejection of the assembly, its component parts, or the original mill product.

6. Apparatus

6.1 *pH meter*.

6.2 *Closed vessel*, such as a desiccator.

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

*A Summary of Changes section appears at the end of this standard

6.3 Equipment for examining test pieces at 10× to 15× magnification.

7. Reagents and Materials

7.1 *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.³ 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.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type IV or better, of Specification **D1193**.

7.3 *Ammonium Chloride Solution (4N)*—Dissolve 107 g of ammonium chloride (NH_4Cl) in water and dilute to 500 mL. Store the solution in a closed vessel.

7.4 *Hydrogen Peroxide (H_2O_2)*, 30 to 35 % technical grade. (**Warning**—Hydrogen peroxide in high concentrations can cause severe skin burns. Use of proper safety equipment is advised.)

7.5 *Sodium Hydroxide Solution (300 to 500 g/L)*—Dissolve 300 to 500 g of sodium hydroxide (NaOH) into water and dilute to 1 L. (**Warning**—Sodium hydroxide can cause chemical burns to the skin and eyes. Use of proper safety equipment is advised.)

7.6 A clean organic solvent or hot alkaline solution that contains no ammoniacal-type groups or substituents.

7.7 *Sulfuric Acid Solution (50 mL/L)*—Slowly add 50 mL of concentrated sulfuric acid (H_2SO_4) into water and dilute to 1 L.

8. Test Media

8.1 Slowly add sodium hydroxide solution to ammonium chloride solution to give a test solution with a pH value appropriate to the intended application (see **Annex A1**). Maintain the solution at ambient temperature and dilute with DI water up to a volume of 1 L. Check the pH value with the pH meter after dilution. Prepare the solution preferably in a fume hood in a laboratory using appropriate safe laboratory procedures and store it in a closed vessel. Before use, check the pH value again, and adjust if necessary.

8.1.1 In the closed test vessel, the ratio of surface area of test solution to air volume of the vessel above the solution shall be a minimum of 20 cm^2/L .

8.2 The pickling solution for cleaning test pieces before and after testing shall be 5 % sulfuric acid. If necessary, for cleaning test pieces after testing, a small amount of hydrogen peroxide solution may be added to the pickling solution (for example, 20 to 30 mL of hydrogen peroxide solution per litre of pickling solution).

³ *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.

9. Test Specimen Preparation

9.1 The test specimen size shall be prescribed in the specification of the product or part being tested. In the event that a test specimen size is not prescribed in a given rod, wire, or tube specification, a full cross section having a minimum length of 152 mm shall be tested.

9.2 The presence of burrs on the test specimen may contribute to acceleration of stress corrosion cracking if not removed before the ammonia vapor test. The burrs shall be removed by fine file or abrasive paper to facilitate this test.

9.3 Degrease the test specimen using the clean organic solvent or alkaline solution.

9.4 After degreasing, clean the test piece in the pickling solution and immediately thereafter thoroughly rinse it, first in cold running water, then in hot water, and finally, completely dry it in a stream of warm air.

10. Test Procedure

10.1 Allow the dry test piece to reach the exposure temperature specified below, and transfer it immediately to the closed vessel (see 6.2) at the same temperature, and containing the freshly prepared test solution at the specified pH value (see 8.1).

10.1.1 Suspend (or place) the test piece such that the ammonia vapor has free access to all surfaces, not less than 50 mm above the test solution surface.

10.1.1.1 When placed on a porcelain support within the test vessel, the region of the test piece within 5 mm of the support shall be disregarded when inspecting for cracks.

10.1.1.2 The test piece shall be placed no less than 10 mm from the inside walls of the test vessel.

10.1.1.3 When more than one test piece is placed in the vessel, the test pieces shall be no less than 10 mm apart.

10.2 The volume of the test solution shall be at least 200 mL per square decimeter of test piece surface. The exposure temperature shall be between 20 and 30°C and shall be kept constant to within $\pm 1^\circ\text{C}$ during the test. In case of dispute, the exposure temperature shall be $25^\circ\text{C} \pm 1^\circ\text{C}$.

10.3 The exposure time shall be 24 h.

10.4 After exposure, remove the test piece from the closed vessel and immediately clean it in pickling solution for a few minutes at ambient temperature (below 40°C), or until the surfaces of the test piece are sufficiently clean from corrosion products to allow observation of possible cracks. After rinsing in water and drying in warm air, examine the surface of the test piece for cracks at a magnification of 10 to 15×.

10.5 Before inspection, deform the test piece slightly by bending or flattening to open up fine cracks to make them more easily observable.

10.6 Metallographic examination may be used to evaluate the nature of any observed cracks.

11. Test Report

11.1 The test report shall contain the following information:

11.1.1 Sample identification,

- 11.1.2 Reference to the test method used,
- 11.1.3 The pH value used for the solution producing the ammonia vapor atmosphere,
- 11.1.4 The number of replicate test pieces tested,
- 11.1.5 The test results: cracks or no cracks (as required in the appropriate product specification),
- 11.1.6 Any other features of the material noted during the determination, and
- 11.1.7 The date of the test.

12. Precision and Bias

12.1 No information is presented about either the precision or bias of this test method for determining susceptibility to

stress corrosion cracking in copper alloys since the procedure is directed at a subjective nonquantitative visual interpretation of condition of the specimen and its relation to an applicable product specification.

13. Keywords

13.1 ammonia test; copper alloys; residual stress; stress corrosion ; stress corrosion cracking

ANNEX

(Mandatory Information)

A1. REPRESENTATIVE pH VALUES

A1.1 On the basis of the known correlation between the behavior of test pieces in the ammonia vapor test and the behavior of copper alloy products under service conditions, the following pH values are considered as being representative of atmospheres of different corrosiveness and corresponding to different safety requirements.

A1.1.1 An appropriate risk level (pH value) shall be specified in the product specification.

Low	Indoor atmosphere conditions	8.3	9.5
Moderate	Indoor atmosphere with risk of formation of condensation	9.5	10.0
	Outdoor atmosphere, temperate climate	9.8	10.0
High	Atmosphere with ammoniacal pollution, for example in stables	10.0	10.5

Corrosiveness of Atmosphere	pH Value	Safety Requirement
	Low	High

APPENDIX

(Nonmandatory Information)

X1. RATIONALE (COMMENTARY)

X1.1 This test method was developed to address the demand for a test method for determination of the presence of residual stresses in copper alloy products which may lead to stress corrosion cracking other than the mercurous nitrate test, Test Method **B154**. Research work performed by Mattsson, et al⁴

validates the technical integrity of the test method.

X1.2 This test method does not attempt to compare the effectiveness of the test to other test methods, including the mercurous nitrate test, Test Method **B154**, nor does it attempt to quantify its relative effectiveness on various copper alloy products. These issues must be addressed on a case by case basis, since such products and tests are specific for their respective requirements and applications.

⁴ Einar Mattsson, Rolf Holm, and Lars Hassel, "Ammonia Test for Stress Corrosion of Copper Alloys."

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