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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Method for Mercurous Nitrate Test for Copper and Copper Alloys[1]

This standard is issued under the fixed designation B 154; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method describes the technique for conducting the mercurous nitrate test for residual stress in wrought copper and cast or wrought copper-base alloy products. This test method is intended to cover the mercurous nitrate test of certain copper-alloy products supplied in accordance with ASTM specifications prepared by Committee B-5. This test method is not intended to be used on assembled parts.

NOTE 1—For any particular copper alloy, reference should be made to the material specification.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 *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 and hazard statements see Note 2, Note 3, Note 4, Note 5, and Note 6 and Sections 1, 4, 5, and 7.

NOTE 2—**Warning:** Mercury is a definite health hazard in use and disposal.

2. Terminology

2.1 *Definitions:*

2.1.1 *stress corrosion crack*—spontaneous failure of metals by cracking under combined action of corrosion and stress, residual or applied.

2.1.2 *residual stress*—stresses that remain within a body as the result of plastic deformation or casting.

3. Significance and Use

3.1 This test method is an accelerated test for detecting the presence of residual (internal) stresses which might result in failure of individual parts in storage or in service due to stress corrosion cracking. This test method is not intended for assemblies under applied stress. If used for that purpose it shall be for information only and not a cause for rejection of the assembly, its component parts, or the original mill product.

4. Mercurous Nitrate Solution

4.1 *Concentration*—The solution shall be an aqueous mercurous nitrate solution containing 10 g of $HgNO_3$ and 10 mL of HNO_3 (sp gr 1.42) per litre of solution.

4.2 *Preparation*—The aqueous mercurous nitrate solution shall be prepared by either of the following procedures, A or B. Used solutions may be replenished as described in 4.3.

4.2.1 *Procedure A*—Dissolve 11.4 g of $HgNO_3 \cdot 2H_2O$ or 10.7 g of $HgNO_3 \cdot H_2O$ in approximately 40 mL of distilled water acidified with 10 mL of HNO_3 (sp gr 1.42). After the crystals are completely dissolved, dilute the solution with distilled water to 1000 mL.

NOTE 3—**Warning:** The mercurous nitrate crystals are obtainable in both the monohydrate and dihydrate form and should be handled with caution because of their highly toxic effects.

NOTE 4—**Caution:** When weighing crystals, the weight of the water of crystallization should be taken into consideration. The mercurous nitrate crystals are photosensitive and when they have turned yellow are difficult to dissolve.

NOTE 5—**Precaution:** Care should be exercised when mixing chemicals. Mixing should only be done by qualified personnel using appropriate chemical laboratory techniques.

4.2.2 *Procedure B*—Dissolve 76 g of mercury in 114 mL of diluted HNO_3 (1 part water to 1 part HNO_3) (sp gr 1.42). Carefully dilute with distilled water to 1000 mL. This provides a concentration of 100 g of $HgNO_3$ after a slight loss due to heating. Add the water in small portions while stirring to prevent local overdilution. This gradual dilution, together with the excess acid, will prevent precipitation of basic salts of mercury. Dilute 100 mL of this solution (10 %) with 7 mL of HNO_3 (sp gr 1.42) and 893 mL of water.

NOTE 6—**Warning:** Mercury is a definite health hazard and therefore equipment for the detection and removal of mercury vapor produced in volatilization is recommended. The use of rubber gloves in testing is advisable.

4.3 *Replenishment of Solution*—The spent solution may be reclaimed by replenishing the mercurous nitrate solution, to a 1 % concentration, as follows:

4.3.1 Measure as accurately as possible in a small graduated cylinder 50 mL of the spent $HgNO_3$ solution.

4.3.2 Transfer to an Erlenmeyer flask, and add 10 mL of HNO_3 (1 + 1).

[1] This test method is under the jurisdiction of ASTM Committee B-5 on Copper and Copper Alloys and is the direct responsibility of Subcommittee B05.06 on Methods of Test.

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4.3.3 Add slowly $KMnO_4$ solution (1 %) from a buret with a constant shaking until there is an excess as indicated by the pink color, which persists for several minutes.

4.3.4 Add $FeSO_4$ crystals until the solution, when shaken, becomes clear. Then titrate the solution with 0.1 *N* KCNS solution to the appearance of a reddish brown color. Repeat this procedure with 50 mL of a standard $HgNO_3$ solution (1 %).

4.3.5 The ratio, *R*. of the number of millilitres of KCNS solution required to titrate the spent solution, to the number of millilitres required to titrate the standard solution, determines the number of millilitres, *X*, of 10 % $HgNO_3$ in 3 % HNO_3 solution required to replenish 1 L of spent solution. Values of *R* and *X* for a litre volume are given in Table 1.

5. Hazards

5.1 **Warning**—Mercury is a definite health hazard in use and disposal.

5.2 *Suggested Mercurous Nitrate Disposal*:

5.2.1 To mercurous nitrate solutions add sodium hydroxide to pH 10 to 11.

5.2.2 Filter precipitated mercury and other heavy metals.

5.2.3 Though the filtrate is low in free mercurous or mercuric ions, it must be further treated before disposal.

5.2.4 To each litre of filtrate, add two drops (0.1 cm^3) of 24 % ammonium sulfide.

5.2.5 After the second filtering, the filtrate may be discarded.

TABLE 1 Replenishment of Spent Mercurous Nitrate Solution to 1 % Concentration

NOTE 1—$X = 111.1\ (1 - R)$

where:

R = fraction of mercury remaining in solution (determined by titration), and

X = number of millilitres of mercurous nitrate solution (10 %) to be added to 1 L of spent mercurous nitrate solution to raise the concentration of mercurous nitrate to 1 %.

R	*X*	*R*	*X*
0.10	100.0	0.56	48.9
0.12	97.8	0.58	46.7
0.14	95.5	0.60	44.4
0.16	93.3	0.62	42.2
0.18	91.1	0.64	40.0
0.20	88.9	0.66	37.8
0.22	86.7	0.68	35.6
0.24	84.4	0.70	33.3
0.26	82.2	0.72	31.1
0.28	80.0	0.74	28.9
0.30	77.8	0.76	26.7
0.32	75.5	0.78	24.4
0.34	73.3	0.80	22.2
0.36	71.1	0.82	20.0
0.38	68.9	0.84	17.8
0.40	66.7	0.86	15.6
0.42	64.4	0.88	13.3
0.44	62.2	0.90	11.1
0.46	60.0	0.92	8.9
0.48	57.8	0.94	6.7
0.50	55.6	0.96	4.4
0.52	53.3	0.98	2.2
0.54	51.1		

NOTE 7—If heating is used in either of the previous procedures, the container should be covered with a watch glass to prevent loss of HNO_3 and water to the atmosphere. After solution is complete, use a small volume of retained dilution water to rinse the watch glass into the container.

5.2.5.1 Monitor the filtrate to assure it meets appropriate health safety standards, or is disposed of properly.

5.2.6 The precipitates should be collected and stored with the mercury contaminated test samples and sold to a licensed mercury disposal service.

6. Test Specimen

6.1 The test specimen shall be prescribed in the specification for the material 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 length of 6 in. (152 mm) shall be tested.

6.2 The presence of burrs on the test specimen may contribute to acceleration of stress corrosion cracking if not removed prior to the mercurous nitrate test. The burrs shall be removed by fine file or abrasive paper to facilitate this test.

7. Procedure

7.1 Degrease the specimen in a suitable alkaline degreasing solution or organic solvent. If necessary, totally immerse the specimen in an aqueous solution of sulfuric acid (15 volume %) or nitric acid (40 volume %) until all oxides are completely removed from its surface or pickle in such solutions as may be prescribed in the specification for the material being tested. Remove the specimen from the pickling solution and wash it immediately in running water. Drain the specimen free of excess water and totally immerse it at room temperature in the mercurous nitrate solution prepared in accordance with 4.2. Use at least 10 mL of mercurous nitrate solution per square inch of exposed surface of the test specimen.

7.2 After 30 min remove the specimen from the mercurous nitrate solution and wash it in running water. Wipe off any excess mercury from the surface of the specimen. Immediately examine it visually for cracks unless a time limitation is provided in the product specification.

7.2.1 Tested specimens are mercury contaminated and must be disposed of properly. They may not be included with returns for remelting or machined into product.

7.2.2 Make sure that rinse water and wiping material are monitored to ensure that they meet appropriate health safety standards, or are disposed of properly.

7.3 Do not reuse the solution unless it is replenished to 1 % in accordance with the procedure in 4.3.

8. Precision and Bias

8.1 No statement is made about the precision or bias of this test method since the procedure is directed at a subjective visual interpretation of condition of the specimen and its relation to an applicable product specification.

9. Keywords

9.1 mercurous nitrate; residual stress; stress corrosion