Designation: B154 – 16

Standard Test Method for Mercurous Nitrate Test for Copper Alloys[1]

This standard is issued under the fixed designation B154; 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 U.S. Department of Defense.

1. Scope*

1.1 This test method describes the technique for conducting the mercurous nitrate test for residual stresses in wrought copper alloy mill products.

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

NOTE 2—Test Method B858 may be considered as a possible alternative test method which does not involve the use of mercury.

NOTE 3—This test method is considered historically reliable for determining the potential state of residual stress in copper alloys, but not promoted for use due to the hazards relating to mercury use and environmentally appropriate disposal.

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

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 determines the applicability of regulatory limitations prior to use.* For specific precautionary and hazard statements see Sections 1, 6, and 7. (**Warning—**Mercury has been designated by many regulatory agencies as a hazardous substance that can cause serious medical issues. Mercury, or its vapor, has been demonstrated to be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury containing products. See the applicable product Safety Data Sheet (SDS) for additional information. Users should be aware that selling mercury and/or mercury containing products into your state or country may be prohibited by law.*)*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

B846 Terminology for Copper and Copper Alloys

B858 Test Method for Ammonia Vapor Test for Determining Susceptibility to Stress Corrosion Cracking in Copper Alloys

D1193 Specification for Reagent Water

3. Terminology

3.1 For terms related to copper and copper alloys, refer to Terminology B846.

4. Summary of Test Method

4.1 The prepared test specimen is completely immersed in the mercurous nitrate test solution for 30 min at ambient temperature. Upon removal from the solution, the test specimen is wiped and immediately examined visually for cracks. Test specimen and test supplies are discarded in accordance with all federal, state, and local requirements.

5. Significance and Use

5.1 This test method is an accelerated test for detecting the presence of residual (internal) stresses that might result in failure of individual parts in storage or in service due to stress corrosion cracking.

5.2 This test method is not intended for use on assemblies or parts under applied stress. If used for that purpose, the results shall be for information only and not a cause for rejection of the assembly, its component parts, or the original mill product.

6. Reagents and Materials

6.1 *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 Reagent 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.

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[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] 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.

[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. (USP), Rockville, MD.

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

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

6.3 *Mercurous Nitrate Solution*—The solution shall be an aqueous mercurous nitrate solution containing 10 g of mercurous nitrate solution ($HgNO_3$) and 10 mL of nitric acid (HNO_3) (sp gr 1.42) per litre of solution.

6.4 *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 6.5.

6.4.1 *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 water to 1000 mL. (**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.) (**Warning—**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.) (**Warning—**Care should be exercised when handling and mixing chemicals. Qualified personnel using appropriate chemical-laboratory techniques should only do the handling and mixing.)

6.4.2 *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. (**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.)

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

6.5.1 Measure 50 mL of the spent $HgNO_3$ solution in a graduated cylinder.

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

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

6.5.4 Add iron (II) sulfate ($FeSO_4$) crystals until the solution, when shaken, becomes clear. Then titrate the solution with 0.1 *N* potassium thiocyanate (KCNS) solution to the appearance of a reddish brown color. Repeat this procedure with 50 mL of a standard 1 % weight per volume of $HgNO_3$ solution.

6.5.5 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 volume percent $HgNO_3$ in 3 volume percent 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.

7. Hazards

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

7.2 *Suggested Mercurous Nitrate Disposal:*

7.2.1 To mercurous nitrate solutions add sodium hydroxide (NaOH) to pH 10 to 11.

7.2.2 Filter precipitated mercury and other heavy metals.

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

7.2.4 To each litre of filtrate, add two drops (0.1 cm^3) of 24 volume percent ammonium sulfide $(NH_4)_2S$.

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

NOTE 4—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.

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

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 10 volume percent mercurous nitrate solution 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		

7.2.6 The precipitates should be collected and stored with the mercury-contaminated test samples and disposed of or treated by a licensed mercury disposal-service.

8. Sampling and Test Specimen Preparation

8.1 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 150 mm shall be tested.

8.2 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.

8.2.1 The burrs shall be removed by fine file or abrasive paper to facilitate this test.

NOTE 5—The presence of burrs on the test specimen may contribute to acceleration of liquid-metal embrittlement of the mercurous nitrate solution, if not removed prior to performance of the test.

9. Test Procedure

9.1 Degrease the specimen in a suitable alkaline degreasing solution or organic solvent. If necessary, totally immerse the specimen in an aqueous solution of 15 volume percent sulfuric acid (H_2SO_4) or 40 volume percent nitric acid 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, at room temperature, immerse it totally in the mercurous nitrate solution prepared in accordance with 6.4. Use at least 15 mL of mercurous nitrate solution per 1000 mm^2 of exposed surface of the test specimen.

9.2 After 30 min remove the specimen from the mercurous nitrate solution. Wipe off any excess mercury from the surface of the specimen with damp cloth or paper towels. Immediately examine it visually for cracks unless a time limitation is provided in the product specification.

9.2.1 Tested specimens and test supplies (cloths, towels, etc.) could be contaminated with mercury and must be disposed of in accordance with all applicable environmental regulations. They may not be included with returns for remelting or machined into product.

9.2.2 Make sure all material including test specimens, wiping material, and mercury solutions are monitored, that they meet appropriate health safety handling standards, and disposition of all related material is in accordance with all applicable environmental regulations.

9.3 Do not reuse the solution unless it is replenished to 1 % in accordance with the procedure in 6.5.

10. Test Report

10.1 When testing is completed by an independent third-party laboratory, the test report shall include the following information. In-house captive laboratories shall only report 10.1.1, 10.1.2, and 10.1.5.

10.1.1 The date of test,

10.1.2 Sample identification,

10.1.3 Reference to the test method used,

10.1.4 The number of replicate test pieces tested,

10.1.5 The test results: Pass or Fail (relative to cracks) as required in the appropriate product specification, and

10.1.6 Any other features of the material noted during the determination.

11. Precision and Bias

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

12. Keywords

12.1 mercurous nitrate; residual stresses; residual stress test; stress corrosion

APPENDIX

(Nonmandatory Information)

X1. RATIONALE COMMENTARY

X1.1 This test method does not attempt to compare the effectiveness of the test to other test methods, including the Ammonia Vapor Test, Test Method B858, 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.

SUMMARY OF CHANGES

Committee B05 has identified the location of selected changes to this standard since the last issue (B154 – 12$^{\varepsilon 1}$) that may impact the use of this standard. (Approved Oct. 1, 2016.)

(1) In 1.3, "hazardous material" was changed to "hazardous substance" in the first sentence of the mercury caveat.
(2) In 4.1, a third sentence was added.
(3) In 6.2 and 6.3, typos were corrected.
(4) In 6.5.3 and 6.5.4, "1 weight per volume percent" was changed to "1 % weight per volume."
(5) In 7.2.6, "sold" was changed to "disposed of or treated by."
(6) In 9.2.1, "cloth/toweling" was changed to "test supplies (cloths, towels, etc.)."