This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

Designation: B368 – 21

Standard Test Method for Copper-Accelerated Acetic Acid-Salt Spray (Fog) Testing (CASS Test) [1]

This standard is issued under the fixed designation B368; 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 prescribes the conditions required in copper-accelerated acetic acid-salt spray (CASS) testing for specification purposes. The standard does not specify the type of test specimen or exposure periods to be used for a specific product, nor the interpretation to be given to the results.

1.2 This test method is applicable to evaluating the corrosive performance of decorative copper/nickel/chromium or nickel/chromium coatings on steel, zinc alloys, aluminum alloys, and plastics designed for severe service. It is also applicable to the testing of anodized aluminum. The suitability of this test and correlation of results with service experience should be determined before it is specified for coating systems or materials other than those mentioned in this paragraph.

NOTE 1—The following standards are not requirements. They are referenced for information only: Practices B537 and E50, Specifications B456 and B604, and Test Method B602.

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

1.4 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For more specific safety precautionary information see 8.1.

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

B117 Practice for Operating Salt Spray (Fog) Apparatus
B162 Specification for Nickel Plate, Sheet, and Strip
B456 Specification for Electrodeposited Coatings of Copper Plus Nickel Plus Chromium and Nickel Plus Chromium
B537 Practice for Rating of Electroplated Panels Subjected to Atmospheric Exposure
B602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings
B604 Specification for Decorative Electroplated Coatings of Copper Plus Nickel Plus Chromium on Plastics
D1193 Specification for Reagent Water
E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials
E70 Test Method for pH of Aqueous Solutions With the Glass Electrode

3. Significance and Use

3.1 The CASS test is widely employed and is useful for specification acceptance, simulated service evaluation, manufacturing control, and research and development. It was developed specifically for use with decorative, electrodeposited nickel/chromium and copper/nickel/chromium coatings. Use of the test has improved the quality of electroplated parts and led to the development of new and superior electroplating processes.

4. Apparatus

4.1 The apparatus required for the CASS test consists of a fog chamber, a salt-solution reservoir, a supply of compressed air, one or more atomizing nozzles, specimen supports, provision for heating the chamber, and necessary means of control.

4.2 The size and detailed construction of the apparatus are optional, provided the conditions meet the requirements of this

[1] This test method is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.10 on Test Methods.

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The CASS test was developed by the initiative of the Research Board of the American Electroplaters Society under AES Project 15.

[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.

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test method. The construction of the apparatus is described in the appendix of Test Method B117. For the CASS test, however, the requirements for air pressure and temperature are typically 0.08 to 0.12 MPa and 60 to 65 °C, respectively. The actual air pressure will be that required to produce the proper collection rate (see 8.3.1).

4.3 The apparatus shall be constructed so that drops of solution that accumulate on the ceiling or cover of the chamber do not fall on the specimens being tested. Drops of solution that fall from the specimens shall not be returned to the solution reservoir for respraying.

4.4 Materials of construction shall not affect the corrosiveness of the fog, nor be themselves corroded by the fog.

5. Test Solution

5.1 Prepare the salt solution by dissolving 5 parts by weight of salt in 95 parts of water conforming to Specification D1193, Type IV. The salt shall be sodium chloride (NaCl), ACS reagent grade, or equivalent. The pH of this solution shall be between 6.0 and 7.0. Impurities or contamination of either the salt or the water, or both, shall be suspected if the pH is outside of this range (Note 2). No adjustment should be made.

5.2 Add 0.25 g of reagent grade copper chloride ($CuCl_2 \cdot 2H_2O$) to each liter of the salt solution; dissolve and mix thoroughly.

5.3 The pH of the salt-copper solution shall be adjusted to the range of 3.1 to 3.3, as measured on a sample of the collected spray, by the addition of glacial acetic acid, ACS reagent grade, or equivalent (Note 3). The pH measurement shall be made electrometrically at 25 °C. Before the solution is atomized, it shall be free of suspended solids (Note 4).

NOTE 2—Reference Practice B117 and Test Method E70 for requirements on salt. The made up CASS solution should be adjusted to a specific gravity of 1.030 to 1.040, when measured at a temperature of 25 °C, will meet the concentration requirement. It is suggested that a daily check be made.

NOTE 3—The initial solution may be adjusted to a pH that gives the proper pH range in the collected solution. Adjustment of the initial pH for makeup solution is based upon the requirements to maintain the required pH of the collected samples. If less than 1.3 or more than 1.6 mL of the glacial acetic acid are required per litre of sodium chloride and copper solution to attain the specified pH, some discrepancy in the system may be suspected (the purity of the water or salt, or both; the accuracy of the pH meter; the general cleanliness of the system; etc.).

NOTE 4—The freshly prepared salt solution may be filtered or decanted before it is placed in the reservoir, or the end of the tube leading from the solution to the atomizer may be covered with a double layer of cheese cloth to prevent plugging of the nozzle.

6. Air Supply

6.1 The compressed air supply to the nozzle or nozzles for atomizing the test solution shall be free of oil and dirt (Note 5). Pressure shall be adequate to provide a specified condensate rate.

NOTE 5—The air supply can be freed of oil and dirt by passing it through a water scrubber or at least 60 cm of suitable cleaning material, such as sheep's wool, excelsior, slag wool, or activated alumina. Commercial filters for compressed air may be used.

NOTE 6—Atomizing nozzles may have a critical pressure, at which an abnormal increase in the corrosiveness of the salt fog occurs. If the critical pressure of a nozzle has not been determined with certainty, control of fluctuation in the air pressure within ±0.0007 MPa by installing a pressure regulator valve minimizes the possibility that the nozzle will be operated at its critical pressure. Pressure of 0.10 ± 0.02 MPa has been found satisfactory.

7. Test Specimens

7.1 The type and number of test specimens to be used, as well as the criteria for the evaluation of the test results, shall be defined in the specifications covering the material or product being tested or shall be mutually agreed upon between the purchaser and the supplier.

8. Procedure

8.1 *Preparation of Test Specimens*— Clean metallic and metallic coated specimens. Unless otherwise agreed upon, clean decorative copper/nickel/chromium or nickel/chromium coatings immediately before testing by wiping significant surfaces with a cotton pad saturated with a slurry containing 10 g of pure magnesium oxide powder (ACS reagent grade) in 100 mL of distilled water. Upon rinsing in warm running water, be sure that the clean surface is free of water break. Anodized aluminum parts may be cleaned with inhibited 1,1,1-trichloroethane or other suitable organic solvent (see Warning). Do not clean organic and other nonmetallic coated specimens. Other methods of cleaning, such as the use of a nitric-acid solution for the chemical cleaning or passivation of stainless steel specimens, are permissible when agreed upon between the purchaser and the supplier. Take care that the specimens after cleaning are not recontaminated by excessive or careless handling. Protect the cut edges of plated, coated, or multilayered materials and areas containing identification marks or in contact with the racks or supports with a coating that is stable under the conditions of the test, such as wax, stop-off lacquer, or pressure-sensitive tape. (**Warning—**1,1,1-Trichloroethane should be used in a well-ventilated area away from open flames.)

8.2 *Positioning of Specimens*—Position the specimens in the CASS test chamber during the test so that the following conditions are met:

8.2.1 Support or suspend the specimens 15 – 30° from the vertical and preferably parallel to the principal direction of horizontal flow of fog through the chamber, based upon the dominant surface being tested, avoiding pooling of solution on the sample. See Note 8 on automobile parts.

8.2.2 The top of the specimen shall not be higher than the top of the collection tubes.

8.2.3 Make sure the specimens do not come in contact with each other or any other metallic material or any material capable of acting as a wick.

8.2.4 Place each specimen so as to permit free settling of fog on all specimens.

8.2.5 Make sure the salt solution from one specimen does not drip on any other specimen.

8.2.6 Place the specimens in the chamber just prior to bringing the test chamber to the required temperature and turning on the air, since storage in an idle chamber overnight, or for other significant length of time, can affect test results.

NOTE 7—Suitable materials for the construction or coating of racks and

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supports are glass, rubber, plastic, or suitably coated wood. Bare metal should not be used. Specimens are preferably supported from the bottom or the side. Slotted wooden strips are suitable for the support of flat panels. Suspension from glass hooks or waxed string may be used as long as the specified position of the specimens is obtained. If necessary, such suspension may be made by means of secondary support at the bottom of the specimens.

NOTE 8—Support suspended automobile parts, however, so as to expose all significant surfaces at the general level of the condensate collectors. If the position on the automobile is vertical, place the part in an incline position 15-30° from vertical to allow surface wetting by the condensate. If the position on the automobile is facing down, rotate the part approximately 180° to test the significant surface. If there are several significant surfaces at different angles, expose each surface of one or more specimens.

8.3 *Conditions in the Salt-Spray Chamber*—Maintain the exposure zone of the CASS test chamber at a temperature of 49± 1 °C during the exposure period (Note 9).The test duration shall commence when the temperature is 49 ± 1 °C and fog is present in the chamber. Heat the air supply by passing fine bubbles through heated distilled or deionized water (see Specification D1193, Type IV) so that the temperature of the air after expansion at the nozzle is 49 ± 1 °C. Record the temperature within the exposure zone of the closed cabinet (Note 10) twice a day at least 7 h apart (except Saturdays, Sundays, and holidays, when the salt-spray test is not interrupted for exposing, rearranging, or removing test specimens or to check and replenish the solution in the reservoir).

NOTE 9—This can be best accomplished by preheating the chamber to 49 °C before starting solution atomization.

NOTE 10—A suitable method to record the temperature is by a thermometer that can be read from outside the closed cabinet. The recorded temperature must be obtained with the salt-spray chamber closed to avoid a false low reading, because of wet-bulb effect when the chamber is open. Automatic control of temperature in the chamber and a continuous record of temperature are desirable.

8.3.1 Place at least two or more (depending on the size of the cabinet) clean fog collectors within the exposure zone so that no drops of solution will be collected from the test specimens or any other source. Position the collectors in each corner. The arrangement is shown in Fig. 1. Make sure that the fog is such that for each 80 cm^2 of horizontal collecting area each collector collects from 1.0 to 2.0 mL/h of solution, based on a typical run of at least 22 h (Note 11). Maintain the sodium chloride concentration of the collected solution between 4.5 and 5.5 mass % (Note 12 and Note 13). Dilution and evaporation of condensate should be avoided (Note 14).

NOTE 11—Suitable collecting devices are glass funnels with the stems inserted through stoppers into graduated cylinders. Funnels with a diameter of 10 cm (area of about 80 cm^2) and 50 mL cylinders are preferred.

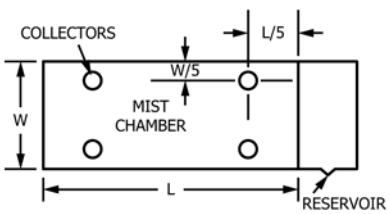

FIG. 1 Arrangement of Fog Collectors

NOTE 12—A solution having a specific gravity of from 1.030 to 1.040, when measured at 25 °C, will meet the concentration requirement. The concentration may also be determined as follows: Dilute 5 mL of the collected solution to 100 mL with distilled water and mix thoroughly; pipet a 10 mL aliquot into an evaporating dish or casserole; add 40 mL of distilled water and 1 mL of 1% potassium chromate (K_2CrO_4) (ACS reagent grade with less than 0.005 % chloride) and titrate with 0.1 *N* silver nitrate ($AgNO_3$) (ACS reagent grade) solution to the first appearance of a persistent red coloration. A test solution that requires between 3.9 and 4.9 mL of 0.1 N Ag NO_3 solution will meet the concentration requirements.

NOTE 13—It has not been found necessary to check copper concentration of the collected solution.

NOTE 14—Factors that may contribute to dilution and evaporation of condensate and make it difficult to control the concentration are lower temperature, inadequate cover insulation, and prolonged storage of test solution at above room temperature.

8.3.2 Direct or baffle the nozzle or nozzles so that none of the spray can impinge directly on the test specimens.

8.4 *Continuity of Test*—Unless otherwise specified in the specification covering the material or product being tested, the test shall be continuous for the duration of the entire test period. Continuous operation implies that the chamber be closed and the spray operating continuously, except for the short daily interruptions necessary to inspect, rearrange, or remove test specimens, to check and replenish the solution in the reservoir, and to make necessary recordings as described in 8.3. Schedule operations so that these interruptions are held to a minimum.

8.5 *Period of Test*—Designate the period of test by the specifications covering the material or product being tested or as mutually agreed upon between the purchaser and the supplier.

NOTE 15—Typical exposure periods are: 6, 16, 22, 44, 110, 192, 240, 504, and 720 h. Parts may be rinsed prior to daily inspection.

8.6 *Cleaning Tested Specimens*—Unless specified in the specifications covering the material or product being tested, carefully remove specimens at the end of the test, rinse gently in clean, running, warm water (38 °C, max) to remove salt deposits from their surfaces, and then dry. To dry the specimens use a stream of clean, compressed air at 0.25 to 0.30 MPa max-gauge pressure or air dry.

NOTE 16—If steel parts are rinsed immediately after removal from the test chamber, small rust spots or flakes of rust may lift off and be rinsed away. It is then difficult to tell where the spots had been. If, after removal from the test chamber, the part is allowed to dry 30 to 60 min before rinsing, all corrosion spots are easily visible. This not does not apply to zinc-die castings.

8.7 *Verification of Corrosive Conditions*—This procedure for verification and standardization of the corrosive conditions within the cabinet involves exposure of nickel test panels and determination of their mass loss in a specified period of time. This may be done monthly or more frequently to make sure the cabinet is operating within the limits specified in 8.7.4. When performing benchmarking, co-relation studies or round robins, nickel coupons shall be run and all data recorded.

8.7.1 Use nickel panels that are 7.5 by 10 cm, approximately 0.09 cm thick, and 77.5 cm^2 (main face and 3 edges) and made from the higher carbon grade (0.15% max) given in Specification B162.

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8.7.2 Place one weighed nickel test panel in each corner of the cabinet with the 10-cm length inclined 30° to the vertical using plastic supports that are about 1.5 by 4 by 7.5 cm. The four test panels should be on the inboard side of the condensate collectors parallel to the cabinet length. Make sure the top of the panels are at the same level as the top of the condensate collectors. See Note 17.

8.7.3 Expose the test panels to 22 h of CASS testing, recording test operating conditions on the data sheet as usual. Remove the panels from the cabinet and rinse each in reagent grade water (see Specification D1193, Type IV) to remove salt. Immerse each panel in 20 % by volume hydrochloric acid prepared with distilled water for 2 min at 20 to 25 °C using nickel-copper alloy 400 (UNS N04400) tongs. The tongs may be nonmetallic or another material that will not affect the mass loss of the nickel by galvanic action. Rinse each panel in water at room temperature and then in warm (38 °C) flowing water. Dry panels in an oven at 100 °C for 10 min. Allow the panels to reach room temperature and weigh immediately on an analytical balance.

8.7.4 Determine the mass loss of each panel by subtracting its weight after exposure from its initial weight. Determine the corrosive loss by dividing the mass loss by the area 77.5 cm^2 (main face and 3 edges). The corrosive rate shall be 0.45 to 0.85 mg/cm^2 to satisfy the requirements of this test method.

NOTE 17—Condition the Panels – New panels may require "conditioning." This can be achieved by pickling panels per 8.7.3 and then exposing them to CASS until proper weight loss is achieved. (may take 2-4 days) "Inactive Panels" – if panels are allowed to sit for a long period of time they may become passive and therefore will not produce the proper weight loss. They should be treated as if new, pickled and then exposed to CASS again until proper weight loss is achieved. Storing or running panels in the cabinet whenever the CASS cabinet is run is desirable to keep them in good condition.

9. Evaluation of Results

9.1 A careful and immediate examination shall be made for the extent of corrosion of the dry-test specimens; or for other failure, as required by the specifications covering the material or product being tested; or by agreement between the purchaser and the supplier. Areas where solution has pooled or run down contamination is evident shall be disregarded.

NOTE 18—Surfaces that are not exposed to the proper angles may be disregarded. For proper analysis each surface being tested should be placed at the proper angle. Cutting of the sample may be required to do so. Care must be given to ensure that contamination does not occur during the cutting process. Masking of the cut surface may be required.

10. Records and Reports

10.1 The following information shall be recorded, unless otherwise prescribed in the specifications covering the material or product being tested:

10.1.1 The title, designation and year of issue of this test method.

10.1.2 The salt, water, copper chloride, and acetic acid used in preparing the salt solution,

10.1.3 All readings of temperature within the exposure zone of the chamber,

10.1.4 Daily records of data obtained from each fog collecting device, including the following:

10.1.4.1 Volume of salt solution collected in (mL/h)/8 cm^2,

10.1.4.2 Concentration or specific gravity at 25 °C of solution collected, and

10.1.4.3 pH of solution collected,

10.1.5 Nickel panel corrosion rate verification,

10.1.6 Type of specimen and its dimensions, or number, or description of part,

10.1.7 Method of cleaning specimens,

10.1.8 Method of supporting or suspending the article in the salt-spray chamber,

10.1.9 Description of protection used as required in 8.1,

10.1.10 Exposure period,

10.1.11 Interruptions in the test, cause and length of time, and

10.1.12 Results of inspections.

NOTE 19—If any of the atomized salt solution that has not come in contact with the test specimens is returned to the reservoir, it is advisable to record the concentration or specific gravity and the pH of this solution also.

NOTE 20—Round robin testing revealed the inability to exhibit repeatability of the mass loss requirements of 8.7.4. Further testing is ongoing to clarify the root causes.

11. Precision and Bias

11.1 The reliability, reproducibility, and accuracy of the CASS test depends on proper and consistent control of a number of factors, including:

11.1.1 Cleaning of the test samples,

11.1.2 Positioning of the specimens and samples in the test cabinet,

11.1.3 Rate of condensation within the test cabinet,

11.1.4 CASS solution makeup,

11.1.5 Cabinet maintenance, and

11.1.6 Verification of corrosive conditions.

11.2 The precision and bias of the CASS test are being determined and will include study of the reproducibility of the verification procedure described in 8.7.

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