ENGINEERING PRACTICE STUDY

PROJECT NUMBER: 5961–2016–030

TITLE:

MIL–STD–750–1, TEST METHOD 1041: PROPOSED REVISION TO CHANGE THE pH MEASUREMENT TEMPERATURE, AND REFORMAT CONTENT

04 NOVEMBER 2016

FINAL REPORT

STUDY CONDUCTED BY

DLA LAND AND MARITIME – VAC

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ATTCH 3

I. OBJECTIVES: The objective of this Engineering Practice (EP) Study was to obtain input from the military services, device manufacturers, laboratories, and user community concerning a proposed revision to test method 1041 of MIL–STD–750–1 (see attachments 1 and 2).

II. BACKGROUND: At the 2015 Columbus JEDEC committee meetings, a request was made to modify the temperature requirement when measuring the pH level of salt solutions. After performing a review of the content of the current revision, 1041.4 (see attachment 3), additional consistency, editorial, and organization issues were noted and were included in the proposal.

The initial draft of this EPS was coordinated to the FSC 5961 community and DLA Land and Maritime had requested that those activities that have an interest in test method 1041 to review the proposed revision.

III. RESULTS: Essential, technical and suggested comments were received regarding the proposed revision. The preparing activity reviewed and assigned a recommeded disposition to all comments. That recommenred disposition was coordinated to all commenters and there were no objections to the assigned disposition.

IV. CONCLUSIONS: The format and technical changes to test method 1041, as documented in attachment 1, should be proposed for incorporation into the next standardization document change action to MIL–STD–750–1. Based on the essential comment received regarding the pH measurment temperature, no changes will be made to that temperature until there is an agreement to use the same temperature in both test method 1041 of MIL–STD–750 and test method 1009 of MIL–STD–883.

V. RECOMMENDATIONS: Prepare an initial draft of MIL–STD–750–1 with the results of this study used for test method 1041.5. Also, work with the project officer for MIL–STD–883 to determine if a modification to the pH measurment temperature can be standardized between the two military standards.

The point of contact for this final report is Mr. David Corbett, DLA Land and Maritime – VAC, P. O. Box 3990, Columbus, OH 43218-3990. Mr. Corbett can also be reached at 614-692-0526 / DSN 850-0526, or by facsimile at 614-693-642 / DSN 850-1642, or by e-mail to: 750.TestMethods@dla.mil.

This draft, dated 04 November, prepared by DLA-CC, has not been approved and is subject to modification.
DO NOT USE PRIOR TO APPROVAL. Text that is highlighted in yellow has been added.

METHOD 1041.5

SALT ATMOSPHERE (CORROSION)

1. Purpose. This test method is an accelerated laboratory corrosion test simulating the effects of seacoast atmospheres on semiconductor devices.

2. Overview. The salt atmosphere test is classified as a destructive test by 4.7 of MIL–STD–750.

3. Definitions. The definitions for all terms used herein shall be as specified in MIL–PRF–19500 and those contained herein. The following definitions shall apply for this test method.

a. Blister: A localized swelling and separation between the coating (s) and base metal.

b. Corrosion: The deterioration of coating or base metal or both by chemical or electrochemical action.

c. Corrosion product (deposit): The effect of corrosion (i.e., rust or iron oxide, nickel oxide, tin oxide, etc. The product of corrosion may be at the corrosion site, or may flow or run (due to action of liquid carrier of salt) so at to cover non-corroded areas.

d. Corrosion site: The site at which the coating or base metal or both is corroded. The location of corrosion.

e. Corrosion stain: A semitransparent deposit due to corrosion products.

f. Flaking: The separation of small pieces of coating that exposes the base metal.

g. Pinhole: A small hole occurring in the coating as an imperfection which penetrates entirely through the coating.

h. Pitting: The localized corrosion of coating or base metal or both, confined to a point or small area that takes the form of a cavity.

4. General requirements.

4.1 Apparatus. Apparatus used in the salt-atmosphere test shall include the following:

a. Salt-atmosphere exposure chamber with racks or fixtures for supporting devices.

b. Salt solution reservoir with means for monitoring an adequate level of solution.

c. Means for atomizing the salt solution, including suitable nozzles and compressed-air supply.

d. Means for heating and controlling the temperature of the chamber.

e. Means for humidifying the air at a temperature above the chamber temperature.

f. Magnifier(s) 10X to 20X.

g. Soft–hair brush or plastic bristle brush.

4.2 Materials. The materials used in the salt-atmosphere test shall include the following:

a. Deionized or distilled water.

b. Salt (NaCl). The salt used shall be sodium chloride containing on the dry basis not more than 0.1 percent by weight of sodium iodide and not more than 0.3 percent by weight total impurities.

c. Solutions for adjusting pH level. Only CP grade (dilute solution) hydrochloric acid or sodium hydroxide shall be used to adjust the pH of the salt solution.

5. Detailed requirements.

5.1 Salt-atmosphere exposure chamber.

5.1.1 Salt solution. The salt concentration shall be 0.5 to 3.0 percent by weight in deionized or distilled water as required to achieve the deposition rates required by 5.2. The salt used shall be in accordance with 4.2.b. The pH of the salt solution shall be maintained between 6.5 and 7.2 when measured at 95°F ±5°F (35°C ±3°C). The solutions for adjusting pH level shall be in accordance with 4.2.c.

5.1.2 Maintenance and conditioning of the chamber. The purpose of the cleaning cycle is to assure that all materials which could adversely affect the results of the subsequent tests are removed from the chamber.

5.1.2.1 Cleaning. The chamber shall be cleaned by operating it at with deionized or distilled water. The chamber shall be cleaned each time the salt solution in the reservoir has been exhausted. After the cleaning cycle, on restarting the chamber, the reservoir shall be filled with salt solution and the chamber shall be stabilized by operating it until the temperature reaches equilibrium.

5.1.2.2 Intermittent or lulls in operation. If operation of the chamber is discontinued for more than one week, the remaining salt solution, if any, shall be discarded. Cleaning shall be performed prior to restarting the chamber.

5.2 Procedure for device exposure. The device shall be placed within the chamber. Unless otherwise specified, a salt atmosphere fog having a temperature of 95°F ±5°F (35°C ±3°C) shall be passed through the chamber for a period of 24 +2, –0 hours. The salt atmosphere fog concentration and velocity shall be adjusted so that the rate of salt deposit in the test area is between 10 and 50 g/m^2 for each 24 hours. Salt deposition rate shall be determined and verified by either volumetric or gravimetric techniques.

5.3 Examinations.

5.3.1 Preparation of specimens for examination. Unless otherwise specified, upon completion of the test, and to aid in the examinations, devices shall be prepared in the following manner: Salt deposits shall be removed by a gentle wash or dip in running water not warmer than 98.6°F (+37°C) and a light brushing, using a soft–hair brush or plastic bristle brush.

5.3.2 Inspection magnification. All inspections shall be performed at a magnification of 10X to 20X.

5.4 Failure criteria. A device with illegible markings, leads missing, broken, or partially separated, evidence of flaking or pitting of the finish or corrosion exceeding five percent of the package area or five percent of the lead shall be considered a failure. Discoloration of the plating or lead finish shall not be considered a failure. The marking legibility requirement shall not apply to characters with a height of less than .030 inches (0.76 mm).

6. Summary. The following conditions shall be specified in the applicable performance specification sheet or acquisition document:

a. Time of exposure, if other than that specified (see 5.2).

b. Measurements and examinations after test (see 5.3).

Initial draft changes: Text highlighted in yellow was added. Red text highlighted in blue with ~~strike-out~~ to be deleted.

Final report changes: Text highlighted in grey has been restored (was proposed for deletion in the initial draft). Text highlighted in green has been added. White text highlighted in ~~dark purple with strike-out~~ is to be deleted.

METHOD 1041.~~4~~5

SALT ATMOSPHERE (CORROSION)

1. Purpose. This test method is an accelerated laboratory corrosion test simulating the effects of seacoast atmospheres on semiconductor devices. ~~The salt atmosphere test is classified as a destructive test.~~

2. Overview. ~~The salt atmosphere test, in which devices are subjected to a fine mist of salt solution, has several useful purposes when utilized with full recognition of its deficiencies and limitations. Originally proposed as an accelerated laboratory corrosion test simulating the effects of seacoast atmospheres on metals, with or without protective coatings, this test has been erroneously considered by many as an all-purpose accelerated corrosion test, which if "withstood successfully" will guarantee that metals or protective coatings will prove satisfactory under any corrosive condition. Experience has since shown that there is seldom a direct relationship between resistance to salt atmosphere corrosion and resistance to corrosion in other media, even in so-called "marine" atmospheres and seawater. However, some idea of the relative service life and behavior of different samples of the same (or closely related) metals or of protective coating-base metal combinations in marine and exposed seacoast locations can be gained by means of the salt atmosphere test, provided accumulated data from correlated field service tests and laboratory salt atmosphere tests show that such a relationship does exist, as in the case of aluminum alloys. (Such correlation tests are also necessary to show the degree of acceleration, if any, produced by the laboratory test). The salt atmosphere test is generally considered unreliable for comparing the general corrosion resistance of different kinds of metals or coating-metal combinations, or for predicting their comparative service life. The salt atmosphere test has received its widest acceptance as a test for evaluating the uniformity (specifically, thickness and degree of porosity) of protective coatings, metallic and nonmetallic, and has served this purpose with varying amounts of success. In this connection, the test is useful for evaluating different lots of the same product, once some standard level of performance has been established. The salt atmosphere test is especially helpful as a screening test for revealing particularly inferior coatings. When used to check the porosity of metallic coatings, the test is more dependable when applied to coatings that are cathodic rather than anodic toward the basic metal. This test can also be used to detect the presence of free iron contaminating the surface of another metal, by inspection of the corrosion products.~~ The salt atmosphere test is classified as a destructive test by 4.7 of MIL–STD–750.

3. ~~1.1~~ Definitions. The definitions for all terms used herein shall be as specified in MIL–PRF–19500 and those contained herein. The following definitions shall apply for this test method.

a. Blister: A localized swelling and separation between the coating (s) and base metal.

b. Corrosion: The deterioration of coating or base metal or both by chemical or electrochemical action.

c. Corrosion product (deposit): The effect of corrosion (i.e., rust or iron oxide, nickel oxide, tin oxide, etc. The product of corrosion may be at the corrosion site, or may flow or run (due to action of liquid carrier of salt) so at to cover non-corroded areas.

d. Corrosion site: The site at which the coating or base metal or both is corroded. The location of corrosion.

e. Corrosion stain: A semitransparent deposit due to corrosion products.

f. Flaking: The separation of small pieces of coating that exposes the base metal.

g. Pinhole: A small hole occurring in the coating as an imperfection which penetrates entirely through the coating.

h. Pitting: The localized corrosion of coating or base metal or both, confined to a point or small area that takes the form of a cavity.

4. General requirements.

4.1 ~~2.~~ Apparatus. Apparatus used in the salt-atmosphere test shall include the following:

a. Salt-atmosphere exposure chamber with racks or fixtures for supporting devices.

b. Salt solution reservoir with means for monitoring an adequate level of solution.

c. Means for atomizing the salt solution, including suitable nozzles and compressed-air supply.

d. Means for heating and controlling the temperature of the chamber ~~heating means and controls~~.

e. Means for humidifying the air at a temperature above the chamber temperature.

f. Magnifier(s) ~~1X to 3X,~~ 10X to 20X ~~and 30X to 60X~~.

g. Soft–hair brush or plastic bristle brush.

4.2 Materials. The materials used in the salt-atmosphere test shall include the following:

a. Deionized or distilled water.

b. Salt (NaCl). The salt used shall be sodium chloride containing on the dry basis not more than 0.1 percent by weight of sodium iodide and not more than 0.3 percent by weight total impurities.

c. Solutions for adjusting pH level. Only CP grade (dilute solution) hydrochloric acid or sodium hydroxide shall be used to adjust the pH of the salt solution.

5. Detailed requirements.

5.1 Salt-atmosphere exposure chamber.

5.1.1 Salt solution. The salt concentration shall be 0.5 to 3.0 percent by weight in deionized or distilled water as required to achieve the deposition rates required by ~~3~~ 5.2. The salt used shall be in accordance with 4.2.b. ~~sodium chloride containing on the dry basis not more than 0.1 percent by weight of sodium iodide and not more than 0.3 percent by weight total impurities.~~ The pH of the salt solution shall be maintained between 6.5 and 7.2 when measured at ~~74.5°F to 100.5°F (23°C to 38°C)~~ 95°F ~~+/– 3~~ ±5°F (35°C ~~+/–~~ ±3°C). ~~Only CP grade (dilute solution) hydrochloric acid or sodium hydroxide shall be used to adjust the pH.~~ The solutions for adjusting pH level shall be in accordance with 4.2.c.

5.1.2 ~~3.1~~ Maintenance and conditioning of ~~test~~ the chamber. The purpose of the cleaning cycle is to assure that all materials which could adversely affect the results of the subsequent tests are removed from the chamber.

5.1.2.1 Cleaning. The chamber shall be cleaned by operating it at 95°F ±5°F (35°C ±3°C) with deionized or distilled water. The chamber shall be cleaned each time the salt solution in the reservoir has been exhausted. After the cleaning cycle, on restarting the chamber, the reservoir shall be filled with salt solution and the chamber shall be stabilized by operating it until the temperature reaches equilibrium.

5.1.2.2 Intermittent or lulls in operation. ~~Intermittent operation of the chamber is acceptable provided salt concentration and the pH of the salt solution and are kept within limits of 5.1.1.~~ If operation of the chamber is discontinued for more than one week, the remaining salt solution, if any, shall be discarded. Cleaning shall be performed prior to restarting the ~~test~~ chamber.

5.2 ~~3.~~ Procedure for device exposure. The device shall be placed within the ~~test~~ chamber. Unless otherwise specified, a salt atmosphere fog having a temperature of 95°F ±5°F (35°C ±3°C) ~~minimum~~ shall be passed through the chamber for a period of 24 +2, –0 hours. The salt atmosphere fog concentration and velocity shall be adjusted so that the rate of salt deposit in the test area is between 10 and 50 g/m^2 ~~per~~ for each 24 hours. Salt deposition rate shall be determined and verified by either volumetric or gravimetric techniques.

5.3 ~~4.~~ Examinations.

5.3.1 Preparation of specimens for examination. Unless otherwise specified, upon completion of the test, and to aid in the examinations, devices shall be prepared in the following manner: Salt deposits shall be removed by a gentle wash or dip in running water not warmer than 98.6°F (+37°C) and a light brushing, using a soft–hair brush or plastic bristle brush.

5.3.2 Inspection magnification. ~~Unless otherwise specified,~~ All inspections shall be performed at a magnification of 10X to 20X.

5.4 Failure criteria. A device with illegible markings, leads missing, broken, or partially separated, evidence ~~(when examined with 10X – 20X magnification)~~ of flaking or pitting of the finish or corrosion exceeding five percent of the package area or five percent of the lead shall be considered a failure. Discoloration of the plating or lead finish shall not be considered a failure. The marking legibility requirement shall not apply to characters with a height of less than .030 inches (0.76 mm).

6. ~~5.~~ Summary. The following conditions shall be specified in the applicable performance specification sheet or acquisition document:

a. Time of exposure, if other than that specified (see ~~3~~ 5.2).

b. Measurements and examinations after test (see ~~4~~ 5.3).

MIL–STD–750–1A

METHOD 1041.4

SALT ATMOSPHERE (CORROSION)

1. Purpose. This test method is an accelerated laboratory corrosion test simulating the effects of seacoast atmospheres on semiconductor devices.

1.1 Definitions. The definitions for all terms used herein shall be as specified in MIL–PRF–19500 and those contained herein.

a. Blister: A localized swelling and separation between the coating (s) and base metal.

b. Corrosion: The deterioration of coating or base metal or both by chemical or electrochemical action.

c. Corrosion product (deposit): The effect of corrosion (i.e., rust or iron oxide, nickel oxide, tin oxide, etc. The product of corrosion may be at the corrosion site, or may flow or run (due to action of liquid carrier of salt) so at to cover noncorroded areas.

d. Corrosion site: The site at which the coating or base metal or both is corroded. The location of corrosion.

e. Corrosion stain: A semitransparent deposit due to corrosion products.

f. Flaking: The separation of small pieces of coating that exposes the base metal.

g. Pinhole: A small hole occurring in the coating as an imperfection which penetrates entirely through the coating.

h. Pitting: The localized corrosion of coating or base metal or both, confined to a point or small area that takes the form of a cavity.

2. Apparatus. Apparatus used in the salt-atmosphere test shall include the following:

a. Exposure chamber with racks for supporting devices.

b. Salt-solution reservoir.

c. Means for atomizing the salt solution, including suitable nozzles and compressed-air supply.

d. Chamber-heating means and controls.

e. Means for humidifying the air at a temperature above the chamber temperature.

3. Procedure. The device shall be placed within the test chamber. Unless otherwise specified, a salt atmosphere fog having a temperature of +35°C shall be passed through the chamber for a period of 24 +2, –0 hours. The fog concentration and velocity shall be adjusted so that the rate of salt deposit in the test area is between 10 and 50 g/m^2 per 24 hours. Salt deposition rate shall be determined and verified by either volumetric or gravimetric techniques.

3.1 Maintenance and conditioning of test chamber. The purpose of the cleaning cycle is to assure that all materials which could adversely affect the results of the subsequent tests are removed from the chamber. The chamber shall be cleaned by operating it at 95°F ±5°F (35°C ±3°C) with deionized or distilled water. The chamber shall be cleaned each time the salt solution in the reservoir has been exhausted. After the cleaning cycle, on restarting the chamber, the reservoir shall be filled with salt solution and the chamber shall be stabilized by operating it until the temperature reaches equilibrium. If operation of the chamber is discontinued for more than one week, the remaining salt solution, if any, shall be discarded. Cleaning shall be performed prior to restarting the test chamber.

3.2 Salt solution. The salt concentration shall be 0.5 to 3.0 percent by weight in deionized or distilled water as required to achieve the deposition rates required by 3. The salt used shall be sodium chloride containing on the dry basis not more than 0.1 percent by weight of sodium iodide and not more than 0.3 percent by weight total impurities. The pH of the salt solution shall be maintained between 6.5 and 7.2 when measured at 95°F, +/– 3°F (35°C, +/– 3°C). Only CP grade (dilute solution) hydrochloric acid or sodium hydroxide shall be used to adjust the pH.

4. Examinations. Unless otherwise specified, upon completion of the test, and to aid in the examinations, devices shall be prepared in the following manner: Salt deposits shall be removed by a gentle wash or dip in running water not warmer than +37°C and a light brushing, using a soft–hair brush or plastic bristle brush. A device with illegible markings, leads missing, broken, or partially separated, evidence (when examined with 10X – 20X magnification) of flaking or pitting of the finish or corrosion exceeding five percent of the package area or five percent of the lead shall be considered a failure. Discoloration of the plating or lead finish shall not be considered a failure. The marking legibility requirement shall not apply to characters with a height of less than .030 inches (0.76 mm).

5. Summary. The following conditions shall be specified in the applicable performance specification sheet or acquisition document:

a. Time of exposure, if other than that specified (see 3).

b. Measurements and examinations after test (see 4).