METRIC

MIL–STD–753C
29 JUNE 1990

SUPERSEDING
MIL–STD–753B
24 MAY 1985

MILITARY STANDARD

CORROSION–RESISTANT STEEL PARTS: SAMPLING, INSPECTION AND TESTING FOR SURFACE PASSIVATION

AMSC N/A

FSC MFFP

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FOREWORD

1. This Military Standard is approved for use by all Departments and Agencies of the Department of Defense.

2. Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Commanding Officer, Naval Air Engineering Center, Systems Engineering and Standardization Department (Code 53), Lakehurst, NJ 08733-5100, by using the self-addressed standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

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TEST METHODS

1. SCOPE

1.1 Scope. This standard describes general and detailed methods of sampling and testing for surface passivity of corrosion-resistant steel parts. These tests may also be useful to determine if there is a need for passivation.

2. APPLICABLE DOCUMENTS

2.1 Government documents.

2.1.1 Standards. The following standard forms a part of this document to the extent specified herein. Unless otherwise specified, the issue of this document is the one listed in the issue of the Department of Defense Index of Specifications and Standards (DODISS) and supplement thereto, cited in the solicitation.

STANDARDS

MILITARY

MIL-STD-105	Sampling Procedures and Tables for Inspection by Attributes

(Unless otherwise indicated, copies of military standards are available from the Naval Publications and Forms Center (Attn: NPODS), 5801 Tabor Avenue, Philadelphia, PA 19120-5099.)

2.2 Non-Government publications. The following document forms a part of this document to the extent specified herein. Unless otherwise specified, the issue of the document shall be the one listed in the issue of the DODISS cited in the solicitation.

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

ASTM B 117 - Salt Spray(Fog) Testing

(Applications for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.)

2.3 Order of precedence. In the event of a conflict between the text of this document and the references cited herein, the text of this document takes precedence. Nothing in this document, however, supersedes applicable laws and regulations unless a specific exemption has been obtained.

3. DEFINITIONS

3.1 Passivation. Passivation is a final treatment/cleaning process used to remove free iron or other anodic contaminants from the surfaces of corrosion resistant steel parts such that uniform formation of a passive surface is obtained. This treatment induces a more noble (cathodic) potential onto the part thus enhancing corrosion resistance.

3.2 Corrosion-resistant steel. The term "corrosion-resistant steel" as used herein refers to those alloyed steels containing chromium in excess of 10.5%.

4. GENERAL REQUIREMENTS

4.1 Sampling and testing.

4.1.1 Sampling for tests. Unless otherwise specified, samples used for the tests specified in 4.1.2 shall be chosen in accordance with MIL-STD-105, Inspection Level S-4 with an Acceptable Quality Level (AQL) of 1.0 percent defective.

4.1.2 Test methods.

4.1.2.1 Visual examination for surface contamination. Samples chosen in accordance with 4.1.1 shall be visually inspected to determine that none of the surfaces show evidence of contamination such as the presence of metal particles, paint, oil, grease, flux and other contaminants.

4.1.2.2 Surface contaminant tests (free iron or other contaminants). Unless a particular test is specified, samples chosen in accordance with 4.1.1 shall be subjected to one of the test methods specified in 5.1 to determine the presence of free iron or other anodic surface contaminants.

4.1.2.2.1 Test chemicals. All required test chemicals shall be American Chemical Society reagent grade purity.

4.2 Failure. Any lot failing to comply with the requirements specified shall be rejected. A lot having failed the specified requirements may be resubmitted for inspection provided that the contractor has removed all defective material or has reworked the lot. Resubmitted lots shall be subjected to tightened inspection in accordance with MIL-STD-105.

5. DETAILED REQUIREMENTS

5.1 Test methods. All detailed test requirements for the determination of free iron or other anodic surface contaminants shall be as specified in the following test methods:

Test Method	Title
100	Water Immersion Test
101	High Humidity Test
102	Copper Sulfate Test
103	Potassium Ferricyanide-Nitric Acid Solution Test
104	Salt Spray Test

6. NOTES

(This section contains information of a general or explanatory nature that may be helpful, but is not mandatory.)

6.1 Intended use. This standard is intended to provide methods for testing the effectiveness of passivation. This standard can also be used to determine if there is a need for passivation.

6.2 Copper sulfate test. The purpose of the copper sulfate test is to determine the presence of free iron which is usually induced onto the surface of a part during fabrication with steel components. The principle of the test is based on an oxidation-reduction reaction which causes the aqueous copper ions to deposit or plate out onto the free iron particles. This method is not recommended for use on martensitic 400 series alloys or lower chromium grades (less than 16%) of ferritic 400 series alloys because the copper may plate out onto the parts even though all the free iron has been removed from the surface during passivation. This is due to the higher amount of iron used in the composition of these alloys which makes it difficult to differentiate between a copper deposit that is the result of free iron and one that is the result of the nature of the alloy.

6.3 Potassium ferricyanide test. This test is more sensitive to the presence of metallic iron than the copper sulfate test, see 6.2. Because of its sensitivity, it is recommended for use only on austenitic AISI 200 and 300 series alloys and only when the determination of trace amounts of free iron is required. Due to the higher amounts of iron used in the composition of the martensitic and ferritic 400 series alloys, this test may show a positive reaction on these alloys even though all the free iron has been removed from the surface during passivation.

6.4 Subject term (keyword) listing.

Corrosion Resistant Steel
Passivation
Stainless Steel

6.5 Changes from previous issue. Marginal notations are not used in this revision to identify changes with respect to the previous issue due to the extensiveness of the changes.

Custodians:
Army - MR
Navy - AS
Air Force - 11

Review Activity:
Army - MI

Preparing Activity:
Navy - AS
(Project No. MFFP-0454)

METHOD 100

WATER IMMERSION TEST

1. SCOPE

1.1 This method is used for the detection of any anodic surface contaminants on corrosion resistant steel, including free iron.

2. APPARATUS AND MATERIALS

2.1 A non-rusting tank.

2.2 Distilled water.

3. PROCEDURE

3.1 Parts shall be alternately immersed in distilled water for one hour and allowed to dry for one hour for a minimum of twenty-four hours.

3.2 After completion of the test, parts shall show no evidence of rust or corrosion.

METHOD 101

HIGH HUMIDITY TEST

1. SCOPE

1.1 This method is used for the detection of any anodic surface contaminant of corrosion resistant steel, including free iron.

2. APPARATUS AND MATERIALS

2.1 A humidity cabinet capable of maintaining the test conditions specified herein.

3. PROCEDURE

3.1 Parts shall be placed in a humidity cabinet and subjected to 97 $\pm$ 3 percent humidity at a temperature of 100 $\pm$ 5°F. The parts shall be exposed to these conditions for a minimum of 24 hours.

3.2 After completion of the test, parts shall show no evidence of rust or corrosion.

METHOD 102

COPPER SULFATE TEST

1. SCOPE

1.1 This method is recommended to detect the presense of free iron on the surface of austenitic chromium-nickel steels of the AISI Type 200 and 300 series alloys, precipitation hardened types, and ferritic AISI 400 series alloys having a minimum of 16% chromium. It is not recommended for use on martensitic AISI 400 series alloys or ferritic AISI 400 series alloys with less than 16% chromium since the test will show a positive reaction on these materials. This test is sensitive and should be used and interpreted only by personnel familiar with its limitations.

WARNING

DO NOT USE THIS TEST ON PARTS TO BE USED IN FOOD PROCESSING.

2. APPARATUS

2.1 10 ml Graduate
500 ml Graduate
1000 ml Beaker
Swab
Balance

3. MATERIAL

3.1 Copper Sulfate ($CuSO_4:5H_2O$)
Distilled Water
Sulfuric Acid (sp gr 1.84)

4. PROCEDURE

4.1 Prepare the test solution as follows: Dissolve 8g of copper sulfate in 500 ml of distilled water to which 2 to 3 ml of sulfuric acid has been added.

NOTE: Aqueous copper sulfate solutions more than two weeks old shall not be used for the purposes of this test.

4.2 Swab the surface to be inspected with test solution, applying additional solution, if needed, to keep the surface wet for a period of 6 minutes.

4.3 At the end of this 6-minute period, carefully rinse and dry the surface such that no copper deposits are removed.

4.4 A copper deposit indicates the presence of metallic iron.

METHOD 103

POTASSIUM FERRICYANIDE-NITRIC ACID SOLUTION TEST

1. SCOPE

1.1 This method is recommended when the detection of very small amounts of free iron is required. It can be used on austenitic chromium-nickel steels of the AISI Type 200 and 300 series but it is not recommended for ferritic or martensitic types of the AISI 400 series since the test will show a positive reaction on these materials. This test is hypersensitive and should be used and interpreted only by personnel familiar with its limitations.

WARNING

DO NOT USE THIS TEST ON PARTS TO BE USED FOR FOOD PROCESSING.

2. APPARATUS

2.1 50 ml Graduate
500 ml Graduate
1000 ml Beaker
Swab
Stirring rod, flat ended
Balance

3. MATERIAL

3.1 Potassium ferricyanide C.P.
Distilled water
Nitric acid (70 percent) C.P.
Acetic acid (10 percent)
Oxalic acid (8 percent)

4. PROCEDURE

4.1 Prepare the test solution as follows: Transfer 10g of chemically pure potassium ferricyanide to a 1000 ml beaker containing 500 ml distilled water, add 30 ml of (70 percent) nitric acid and agitate until all of the ferricyanide is in solution. Dilute to 1000 ml with distilled water.

NOTE: The test solution shall be mixed fresh the day tests are made since it changes color on standing.

4.2 Swab the surface to be inspected with test solution. The formation of a dark blue color within 30 seconds denotes the presence of metallic iron.

METHOD 103 (continued)

POTASSIUM FERRICYANIDE-NITRIC ACID SOLUTION TEST

WARNING

WHEN THE TEST IS NEGATIVE, THE SURFACE SHALL BE THOROUGHLY WASHED WITH WARM WATER TO REMOVE THE LAST TRACES OF TEST REAGENT. WHEN THE TEST IS POSITIVE, THE DARK BLUE STAIN SHALL BE REMOVED WITH A 10 PERCENT ACETIC ACID, 8 PERCENT OXALIC ACID SOLUTION, FOLLOWED BY A THOROUGH HOT WATER RINSE.

METHOD 104

SALT SPRAY TEST

1. SCOPE

1.1 This method is used for the detection of any anodic surface contaminants on corrosion resistant steel, including free iron.

2. APPARATUS AND MATERIALS

2.1 As specified in ASTM B117.

3. PROCEDURE

3.1 Parts shall be subjected to a 5% salt spray test as specified in ASTM B117 for a minimum of two hours.

3.2 After completion of the test, parts shall show no evidence of rust or corrosion.