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AEROSPACE MATERIAL SPECIFICATION

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Passivation Treatments for Corrosion-Resistant Steel

NOTICE

This document has been taken directly from U.S. Military Specification QQ-P-35C and contains only minor editorial and format changes required to bring it into conformance with the publishing requirements of SAE technical standards. The initial release of this document is intended to replace QQ-P-35C. Any part numbers established by the original specification remain unchanged.

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1. SCOPE AND CLASSIFICATION:

1.1 Scope:

This specification covers the different types of passivation treatments as well as recommendations, guidance and precautions for cleaning and descaling corrosion-resistant parts, components, equipment and systems.

1.2 Purpose:

This specification covers the method for cleaning and descaling of stainless steel parts and outlines the final cleaning stage or passivation treatments for use on austenitic, ferritic, martensitic and precipitation hardening corrosion resistant steels. Engineering drawings and specifications should specify classification type (passivation treatment) which should be used in accordance with 1.2.1.

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1.2.1 Types: Passivation treatments shall be of the following types, as specified (see 6.2).

Type II - Medium temperature nitric acid solution with sodium dichromate additive
Type VI - Low temperature nitric acid solution
Type VII - Medium temperature nitric acid solution
Type VIII - Medium temperature high concentration nitric acid solution

2. APPLICABLE DOCUMENTS:

The following publications, of the issues in effect on date of invitation for bids or request for proposal, form a part of this specification to the extent specified herein.

2.1 U.S. Government Publications:

Available from DODSSP, Subscription Services Desk, Building 4D, 700 Robbins Avenue, Philadelphia, PA 19111-5094.

MIL-S-5002	Surface Treatments and Inorganic Coatings for Metal Surfaces of Weapons Systems
MIL-C-13924	Coating, Oxide, Black, for Ferrous Metals
MIL-STD-105	Sampling Procedures and Tables for Inspection by Attributes
MIL-STD-753	Corrosion-Resistant Steel Parts: Sampling, Inspection and Testing for Surface Passivation
O-N-350	Nitric Acid, Technical
O-S-595	Sodium Dichromate, Dihydrate, Technical
FED-STD-313	Material Safety Data Sheets, Preparation and Submission of

2.2 ASTM Publications:

Available from ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959.

ASTM A380	Cleaning and Descaling Stainless Steel Parts, Equipment, and Systems
ASTM B117	Operating Salt Spray (Fog) Apparatus
ASTM B254	Preparation of and Electroplating on Stainless Steel

3. REQUIREMENTS:

3.1 Material:

The chemicals and reagents used for passivation of corrosion-resistant steel parts shall produce passivated surfaces which meet the requirements of this specification.

3.2 Pretreatment:

The pretreatment methods and procedures used prior to the passivation treatment shall be either in accordance with ASTM A380 or MIL-S-5002. The surface of the parts shall be free of oil, grease, rust, scale and other foreign matter and shall have no deleterious effect on material properties. Particular attention shall be given to the pretreatment cleaning of 400 series steels to be certain that all surface oxidation, including rust and heat treat scale, is completely removed. Unless otherwise specified, clean and descale in accordance with ASTM A380. When electrochemical cleaning is required, it shall be performed in accordance with ASTM B254.

3.3 Passivation treatment:

Corrosion-resistant steel parts shall be passivated by immersing them in one of the following aqueous solutions (see 6.2) and maintaining them within the temperature ranges and times described below and in Table I. The parts shall be completely immersed in the solution to avoid severe etching which would otherwise occur above the liquid line.

3.3.1 Type I: Type I withdrawn (see 6.9)

3.3.2 Type II: Type II solution shall contain 20-25 percent by volume of nitric acid in accordance with O-N-350 and 2.5 ± 0.5 percent by weight of sodium dichromate in accordance with O-S-595. Parts shall be processed for 20 minutes at a temperature range between 120-130°F (49°-54°C).

3.3.2.1 For parts made of high carbon/high chromium grades (440 series), straight chromium grades with 12-14% chromium (martensitic 400 series), or for corrosion-resistant steels containing relatively large amounts (0.15 percent) of sulfur or selenium (for example 303, 303Se, 347Se, 416, 416Se, 430F, 430FSe and precipitation hardenable steels), type II solution shall be used (see 6.2 and 6.3).

3.3.3 Type III: Type III withdrawn (see 6.9).

3.3.4 Type IV: Type IV withdrawn (see 6.9).

3.3.5 Type V: Type V withdrawn (see 6.9).

3.3.6 Type VI: Type VI solution shall contain 25-45 percent by volume of nitric acid ($HN0_3$) in accordance with O-N-350. Parts shall be processed for 30 minutes at a temperature range between 70-90°F (21-32°C).

3.3.6.1 Type VI solution can be used to process austenitic 200 and 300 series chromium nickel and chromium grades with 17% chromium or grater (with exception of the 440 series) corrosion-resistant steels.

3.3.7 Type VII: Type VII solution shall contain 20-25 percent by volume of nitric acid ($HN0_3$) in accordance with O-N-350. Parts shall be processed for 20 minutes at a temperature range between 120-140°F (49-60°C).

3.3.7.1 Type VII solution can be used to process parts made of the following corrosion-resistant steels: austenitic 200 and 300 series chromium-nickel, chromium grades with 17% chromium or greater (except 440 series) (see sections 6.2 and 6.3).

3.3.8 Type VIII: Type VIII solution shall contain 45-55 percent by volume of nitric acid in accordance with O-N-350. Parts shall be processed for 30 minutes at a temperature range between 120-130°F (49°-54°C).

3.3.8.1 Type VIII solution can be used for parts made from high carbon and high chromium grades (400 series) and precipitation-hardening stainless steels.

TABLE I. Passivation treatments

Type	Temperature	Time, minimum (minutes) 1/	% (by wt.) Sodium Dichromate	% (by vol.) Nitric Acid
I				Withdrawn
II	120-130°F (49-54°C)	20	2-2.5	20-25
III				Withdrawn
IV				Withdrawn
V				Withdrawn
VI	70-90°F (21-32°C)	30	none	25-45
VII	120-150°F (49-60°C)	20	none	20-25
VIII	120-130°F (49-54°C)	30	none	45-55

1/ Caution: Care must be exercised as excessive time may damage the part.

3.4 Water rinse:

Immediately after removal from the passivating solution the parts shall be thoroughly rinsed; final rinse is carried out in clean water (see 6.4).

3.5 Chromate treatment:

When specified and within one hour after the final water rinse (3.4), all ferritic and martensitic steel parts shall be immersed in an aqueous solution containing 4 to 6 percent by weight of sodium dichromate in accordance with O-S-595, at a temperature of 140° to 160°F (60 - 71°C) for a period of 30 minutes (see 6.1.1). This immersion shall be followed by a rinse in clean water (see 6.4). The parts shall then be thoroughly dried.

3.6 Finish:

The passivated parts shall exhibit a chemically clean surface and shall show no etching, pitting or frosting when examined. A slight discoloration will be allowed (see 4.3 and 6.5).

3.7 Staining:

3.7.1 Water immersion or high humidity tests: The passivated parts shall not exhibit staining attributable to the presence of free iron particles imbedded in the surface when subjected to the water immersion test or the high humidity test (see 4.4.1).

3.7.2 Salt spray or copper sulfate tests: The salt spray test or the copper sulfate test may be specified in addition to or in lieu of either the water immersion test or the high humidity test. When the salt spray test is specified, the passivated surface shall be capable of withstanding salt spray exposure without evidence of rust or staining (see 4.4.2, 6.11).

3.7.2.1 Copper sulfate test: When testing austenitic 300 series chromium nickel steels, it is permissible to use the copper sulfate test as a substitute for the salt spray test. The surface of the resultant passivated parts shall not exhibit copper deposits (see 4.4.1, 4.4.2, 4.4.2.2).

3.8 Workmanship:

The passivated parts shall be free of iron contamination and other foreign materials which could adversely affect the suitability, use or life of the passivated part (see 4.3, 6.13).

3.9 Solution analysis:

The processor shall maintain a record of the control procedures used on each of the processing solutions used in these passivation treatments. Solutions shall be analyzed to verify that the concentrations of nitric acid and sodium dichromate meet the specified requirements. Analyses shall be scheduled at intervals which will demonstrate optimum passivation performance. Frequency of analyses will be determined by the contractor's processing equipment (tank volume), the workload and the time differential between treatments. A chemical record shall be maintained on all control analyses performed, additions made or treatments administered to the processing solution. Upon request of the acquisition activity, such records, including reports of test results, shall be made available (see 4.5 and 6.2).

3.9.1 The contractor shall be responsible for the safe reutilization and disposal of all material generated by this process (see 4.7, 6.12 and 6.13).

4. QUALITY ASSURANCE PROVISIONS:

4.1 Responsibility for inspection:

Unless otherwise specified in the contract or purchase order, the contractor is responsible for the performance of all inspection requirements (examinations and tests) as specified herein. Except as otherwise specified in the contract or purchase order, the contractor may use his own or any other facilities suitable for the performance of the inspection requirements specified herein, unless disapproved by the Government. The Government reserves the right to perform any of the inspections set forth in this specification where such inspections are deemed necessary to ensure supplies and services conform to prescribed requirements.

4.2 Inspection provisions:

4.2.1 Lot: A lot shall consist of passivated parts of similar alloy and manufacturing methods that are pretreated and passivated in one day's production or within a timeframe which will ensure consistent passivation results. A lot shall consist of the same product of one size from one heat in one shipment. When the quantity of passivated parts in one day's production does not warrant daily testing, the lot size shall be as agreed upon by the procuring activity and the contractor (see 6.2).

4.2.2 Sampling:

4.2.2.1 Sampling for visual examination: Samples shall be taken at random from each lot in accordance with MIL-STD-105, Inspection Level S-4. The Acceptable Quality Level (AQL) shall be 1.0 percent defective.

4.2.2.2 Sampling for other tests: Samples shall be taken at random from each lot in accordance with MIL-STD-105, Inspection Level S-3. The AQL shall be 1.0 percent defective. Identically processed specimens, made from the same material used to fabricate the parts, may be used for test purposes in lieu of large parts (see 6.6.). When authorized for use, the test specimens shall be randomly distributed throughout the lot during processing. When multiple tests are to be performed, separate samples are required for each test.

4.3 Visual examination:

Samples selected in accordance with 4.2.2.1 shall be visually examined for etching, pitting or frosting (3.6), and workmanship (3.8).

4.4 Test methods:

The following tests are utilized to evaluate the effectiveness of the passivation treatment. Unless otherwise specified, martensitic grade 440C shall be exempt from these tests (see 6.10).

4.4.1 Water immersion and high humidity tests: Samples selected in accordance with 4.2.2.2 shall be subjected to either a water immersion test or a high humidity test to determine conformance with 3.7.1.

4.4.1.1 Water immersion test: The water immersion test shall be conducted in accordance with Method 100 of MIL-STD-753.

4.4.1.2 High humidity test: The high humidity test shall be conducted as follows:

a. Parts shall be cleaned by immersing them in acetone or methyl alcohol, then swabbing them with clean gauze saturated with acetone or methyl alcohol and drying in an inert atmosphere or desiccated container.

b. The cleaned and dried parts shall be subjected to 95 - 100 percent humidity at 100° - 115°F (38 - 46°C) in a suitable humidity cabinet for 24 - 26 hours.

4.4.2 Salt spray and copper sulfate tests: The salt spray test may be specified in addition to or in lieu of either the water immersion test or the high humidity test. When such a test is specified, samples shall be selected in accordance with 4.2.2.2 and shall conform to 3.7.2 and 3.7.2.1 (see 6.11).

4.4.2.1 Salt spray test: The salt spray test shall be conducted in accordance with ASTM B117 for a minimum of 2 hours using a 5 percent salt solution.

4.4.2.2 Copper sulfate test: The copper sulfate test shall be conducted in accordance with method 102 of MIL-STD-753.

4.5 Certification of analysis:

When specified (see 6.2), a certificate of the quantitative analysis (3.9) of the passivating solution shall be furnished the contracting officer.

4.6 Rejection and retest:

Any lot failing to meet the requirements specified herein shall be rejected. A rejected lot may be resubmitted for inspection provided that the defective parts have been re-pretreated per 3.2, as necessary, re-passivated and re-tested. Re-passivated lots shall be subjected to tightened inspection in accordance with MIL-STD-105.

4.7 Reuse and disposal of treatment solutions:

The reutilization and disposal of all materials generated by this process shall be in accordance with ASTM A380, sections 8.2 and 8.7. Also see 6.12, 6.13 for additional information.

5. PREPARATION FOR DELIVERY:

(This section is not applicable to this specification.)

6. NOTES:

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

6.1 Intended use:

The passivation treatments provided by this specification are intended to improve the corrosion resistance of parts made from austenitic, ferritic and martensitic corrosion-resistant steels of the 200, 300 and 400 series and precipitation hardened corrosion-resistant steels. High carbon, high chromium martensitic 440C grades which are selected for high hardness and resistance may be exempt from passivation treatments at the discretion of the procuring activity.

6.1.1 During processing operations such as forming, machining, tumbling and lapping, iron particles or other foreign particles may become smeared over or imbedded into the surface of corrosion-resistant steel parts. These particles must be removed or they will appear as rust or stain spots. This condition may be prevented by immersing the parts in a solution of nitric acid and sodium dichromate, which will dissolve the particles and allow a thin, transparent passive film to form over the restored surface. Parts with newly formed passive films, should be handled as little as possible in the 24 hours period following this treatment.

6.1.2 This specification is not intended for black oxide coating of parts to be used for photographic and optical instruments; for such applications refer to MIL-C-13924.

6.2 Ordering data:

Purchasers should select the preferred options permitted herein and include the following information in procurement documents:

a) Title, number and date of this specification.
b) Nomenclature and grade of corrosion - resistant material of parts.
c) Type of passivation treatment (1.2.1, 3.3).
d) Lot size, if other than one day's production (4.2.1).
e) When quantitative analysis certificate is required (3.9, 4.5).

6.3 Grades of steel:

Austenitic, ferritic or martensitic types of steels are selected on a basis of properties required (corrosion resistance and design criteria) and fabrication requirements. Table II is a compilation which serves as a guide for some steel grades and their recommended passivation treatment.

TABLE II. Recommended passivation requirements for different steel alloys.

Steel Grades are indicated by Unified Numbering System Numbers
(Numbers in parentheses are AISI, AMS, or ASTM).

Type of Alloy	Solution Types II	VI	VII	VIII
Precipn Hdng	K66286 (A286)			K66286 (A286)
Precipn Hdng	S13800 (13-8Mo)			S13800 (13-8Mo)
Precipn Hdng	S15500 (15-5)			S15500 (15-5)
Precipn Hdng	S15700 (15-7Mo)			S15700 (15-7Mo)
Precipn Hdng	S17400 (17-4)			S17400 (17-4)
Precipn Hdng	S17700 (17-7)			S17700 (17-7)
Austenitic		S20100 (201)	S20100 (201)	
Austenitic		S20200 (202)	S20200 (202)	
Austenitic		S30100 (301)	S30100 (301)	
Austenitic		S30200 (302)	S30200 (302)	
Free Machine	S30300 (303)			
Free Machine	S30323 (303Se)			
Free Machine	S30310 (303X)			
Free Machine	S30323 (303Se)			
Free Machine	S30330 (303Cu)			
Free Machine	S30345 (303Ma)			
Free Machine	S30360 (303Pb)			
Austenitic		S30400 (304)	G30400 (304)	
Austenitic		S30403 (304L)	S30403 (304L)	
Austenitic		S30409 (304H)		S30409 (304H)
Austenitic		S30430 (XM-7)	S30430 (XM-7)	
Austenitic		S30451 (304N)	S30451 (304N)	
Austenitic		S30500 (305)	S30409 (305)	
Austenitic		S30800 (308)	S30800 (308)	
Austenitic		S30900 (309)	S30900 (309)	
Austenitic		S30908 (309S)	S30908 (309S)	
Austenitic		S30940 (309Cb)	S30940 (309Cb)	
Austenitic		S31000 (310)	S31000 (310)	
Austenitic		S31008 (310S)	S31008 (310S)	
Austenitic		S31100 (311)	S31100 (311)	
Austenitic		S31400 (314)	S31400 (314)	

TABLE II. Recommended passivation requirements for different steel alloys. (Continued)

Steel Grades are indicated by Unified Numbering System Numbers
(Numbers in parentheses are AISI, AMS, or ASTM).

Type of Alloy	Solution Types II	VI	VII	VIII
Austenitic		S31500 (315)	S31500 (315)	
Austenitic		S31600 (316)	S31600 (316)	
Austenitic		S31603 (316L)	S31603 (316L)	
Austenitic		S31609 (316H)		S31609 (316H)
Austenitic		S32100 (321)	S32100 (321)	
Austenitic		S32109 (321H)		S31621 (321H)
Austenitic		S32900 (329)	S32900 (329)	
Austenitic		S34700 (347)	S34700 (347)	
Austenitic		S34709 (347H)		S34709 (347H)
Free Machine	S34720 (347S)			
Free Machine	S34723 (347Se)			
Precipn Hdng	S35000 (AM350)			S35000 (AM350)
Precipn Hdng	S35500 (AM355)			S35500 (AM355)
Precipn Hdng	S36200 (Almar 362)			S36200 (Almar362)
Martensitic	S40300 (403)			S40300 (403)
Ferritic	S40500 (405)			S40500 (405)
Ferritic	S40900 (409)			S40900 (409)
Martensitic	S41000 (410)			S41000 (410)
Martensitic	S41400 (414)			S41400 (414)
Martensitic	S41600 (416)			
Martensitic	S41623 (416Se)			
Martensitic	S42000 (420)			
Ferritic	S42900 (429)		S42900 (429)	S42900 (429)
Ferritic			S43000 (430)	
Free Machine	S43020 (430F)			
Free Machine	S43023 (430FSe)			
Martensitic	S43100 (431)			S43100 (431)
Ferritic	S43400 (434)		S43400 (434)	
Ferritic	S43600 (436)		S43600 (436	
Martensitic	S44002 (440A)			S44002 (440A)
Martensitic	S44003 (440B)			S44003 (440B)
Martensitic	S44004 (440C)			S44004 (440C)

TABLE II. Recommended passivation requirements for different steel alloys. (Continued)

Steel Grades are indicated by Unified Numbering System Numbers
(Numbers in parentheses are AISI, AMS, or ASTM).

	Solution Types			
Type of Alloy	II	VI	VII	VIII
Free Machine	S44020 (440F)			
Free Machine	S44023 (440FSe)			
Ferritic	S44200 (442)		S44200 (442)	
Ferritic			S44600 (446)	
Ferritic	S44625 (XM-27)		S44625 (XM-27)	

6.4 Clean water:

Clean water is defined as water containing a maximum total solid content of 200 ppm. Rinsing can be accomplished by a combination of stagnant, countercurrent and/or spray rinses prior to final rinse.

6.5 Chemically clean surface:

A chemically clean surface is defined as a surface upon which water, when applied momentarily to the surface, will remain on that surface in an even, continuous film, and in addition is free of any foreign material or residual film deposit which would be detrimental to the quality of the part.

6.6 Test specimens:

When using test specimens in lieu of parts, they can only effectively represent the parts if they have been exposed to the same processing steps, such as machining, grinding, heat treating, welding, etc, as the parts they are to represent.

6.7 Carburized surfaces:

Stainless steel parts with carburized surfaces cannot be passivated because the carbon combines with the chromium forming chromium carbides on the surface.

6.8 Nitrided stainless steel:

Nitrided stainless steel should not be passivated because the treatment will severely corrode the nitrided case. Note: If passivation is required it must be performed prior to this special treatment.

6.9 Cross reference of classification types:

Revision B to QQ-P-35 references six types of treatment solutions for passivation of corrosion resistant steels. New treatment types will be numbered differently from the original in an effort to avoid confusion or compromise interchangeability. Accordingly two new treatment types have been added and four types have been withdrawn as indicated below:

Type I - Withdrawn (see Table II, for guidance)
Type II - Medium Temperature Nitric Acid Solution With Sodium Dichromate Additive.
Type III - Withdrawn (see Table II, for guidance)
Type IV - Withdrawn (see Table II, for guidance)
Type V - Withdrawn (see Table II, for guidance)
Type VI - Low Temperature Nitric Acid Solution.
Type VII - Medium Temperature Nitric Acid Solution.
Type VIII - Medium Temperature High Concentration Nitric Acid Solution

6.10 Martensitic grade 440C:

High strength alloys such as 440C are subject to hydrogen embrittlement or intergranular attack when exposed to acids. Cleaning by mechanical methods or other chemical methods is recommended.

6.11 The salt spray test has been used to evaluate some martensitic grades of steels, but is usually specified for the austentic 300 grades.

6.12 Material safety:

Hazardous items include substances mixtures, and materials which may cause personal injury, property damage or environmental deterioration through transportation, use or disposal (see 6.2).

6.13 Materials safety data sheets:

Contracting officers will identify those activites requiring copies of completed material safety data sheets prepared in accordance with FED-STD-313. The pertinent government mailing addresses for submission of data are listed in appendix B of FED-STD-313.

6.14 Free iron measurement:

The free iron test is useful for measuring the effectiveness of passivation. Test meters are available for measuring the surface potential of a material. The meters are used to measure the presence of free iron. The subsequent corrosion voltage measurements can be related to the passivation state of the metal.

6.15 Subject term (keyword) listing:

austenitic
copper sulfate test
descaling
ferritic
free iron measurement
high humidity test
lot
martensitic
nitric acid
precipitation hardening
salt spray test
sampling
sodium dichromate
solution analysis
stainless steel
visual examination
water immersion

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