Standard Specification for Chemical Passivation Treatments for Stainless Steel Parts

This standard is issued under the fixed designation A 967; 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 (e) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

Note—Section 6.1.1.1 was editorially corrected in July 2007.

1. Scope

1.1 This specification covers several different types of chemical passivation treatments for stainless steel parts. It includes recommendations and precautions for descaling, cleaning, and passivation of stainless steel parts. It includes several alternative tests, with acceptance criteria, for confirmation of effectiveness of such treatments for stainless steel parts.

1.2 Practices for the mechanical and chemical treatments of stainless steel surfaces are discussed more thoroughly in Practice A 380.

1.3 Several alternative chemical treatments are defined for passivation of stainless steel parts. Appendix X1 gives some nonmandatory information and provides some general guidelines regarding the selection of passivation treatment appropriate to particular grades of stainless steel. It makes no recommendations regarding the suitability of any grade, treatment, or acceptance criteria for any particular application or class of applications.

1.4 The tests in this specification are intended to confirm the effectiveness of passivation, particularly with regard to the removal of free iron and other exogenous matter. These tests include the following practices:

   1.4.1 Practice A—Water Immersion Test,
   1.4.2 Practice B—High Humidity Test,
   1.4.3 Practice C—Salt Spray Test,
   1.4.4 Practice D—Copper Sulfate Test,
   1.4.5 Practice E—Potassium Pericyanide–Nitric Acid Test, and
   1.4.6 Practice F—Free Iron Test.

1.5 The values stated in inch-pound units are to be regarded as the standard. The SI units given in parentheses are for information only.

1.6 The following precautionary caveats pertain only to the test method portions, Sections 14 through 18 of this specification: 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 determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

   2.1.1 A 380 Practice for Cleaning, Descaling, and Depassivation of Stainless Steel Parts, Equipment, and Systems
   2.1.2 B 117 Practice for Operating Salt Spray (Fog) Apparatus
   2.1.3 B 254 Practice for Preparation of and Electroplating on Stainless Steel
   2.1.4 QQ-P-35C Passivation Treatments for Corrosion-Resistant Steels

3. Terminology

3.1 Definition of Term Specific to This Standard—It is necessary to define which of the several commonly used definitions of the term passivation will be used in this specification. (See Discussion.)

3.1.1 Discussion—Stainless steels are autopassivating in the sense that the protective passive film is formed spontaneously on exposure to air or moisture. The presence of exogenous surface contamination, including dirt, grease, free iron from contact with steel topling, and so forth, may interfere with the formation of the passive film. The cleaning of these contaminants from the stainless steel surface will facilitate the spontaneous passivation by allowing the oxygen uniform access to the surface. The passive film may be augmented by chemical treatments that provide an oxidizing environment for the stainless steel surface.

3.1.1.1 In this specification, passivation, unless otherwise specified, is defined as the chemical treatment of a stainless steel with a mild oxidant, such as a nitric acid solution, for the...
purpose of the removal of free iron or other foreign matter, but
which is generally not effective in removal of heat tint or oxide
scale on stainless steel. In the case of stainless steels with
additions of sulfur for the purpose of improved machinability,
passivation may also include the removal of sulfides from the
surface of the metal for the purpose of maximizing corrosion
resistance.

3.1.1.2 The formation of the protective passive film on a
stainless steel, also called passivation in a more general
context, will occur spontaneously in air or other oxygen-
containing environment when the stainless steel surface is free
of oxide scale and exogenous matter.

3.1.1.3 Chemical treatments, such as sodium dichromate
solutions, may facilitate the more rapid formation of the
passive film on a stainless steel surface already free of scale or
foreign matter. Such treatments, also sometimes called passi-
vation in common usage, are designated as post-cleaning
in this specification in order to distinguish them from chemical treatments capable of removing free iron from
stainless steels.

3.1.1.4 The chemical treatments capable of removing heat
tint or oxide scale from stainless steel and capable of dissolving
the stainless steel itself, typically called pickling, are substan-
tially more aggressive than treatments used for passivation, as
defined in 3.1.1.1. The surface of stainless steel that has been
pickled is free of scale, free iron, and exogenous foreign
matter, and does not require a separate treatment for passi-
vation as defined in 3.1.1.1. The passivation process defined in
3.1.1.2 will occur without further chemical treatment but may
be augmented and improved by the post-cleaning treatments
defined in 3.1.1.3.

3.1.1.5 Electrochemical treatments, including electropick-
ling and electropolishing capable of removing heat tint or oxide
scale from stainless steel and capable of dissolving the stainless
steel itself, are substantially more aggressive than treatments
used for passivation, as defined in 3.1.1.1. The surface of
stainless steel resulting from these treatments is free of scale,
free iron, and exogenous foreign matter, and does not require a
separate treatment for passivation as defined in 3.1.1.1. The
passivation process defined in 3.1.1.2 will occur without
further chemical treatment, but may be augmented and im-
proved by the post-cleaning treatments defined in 3.1.1.3.

4. Ordering Information

4.1 It is the responsibility of the purchaser to specify a test
practice appropriate to any particular material and application.
This specification was written for the purpose of providing an
alternative to United States Federal Specification QQ-P-35C.
Determination of the suitability of this specification for that
purpose is the responsibility of the purchaser.

4.2 Unless specified by the purchaser, the chemical treat-
ment applied to the stainless steel parts shall be selected by the
seller from among the listed passivation treatments.

5. Materials and Preparation for Passivation Treatments

5.1 The passivation treatments shall be of one or more of the
following types. The effectiveness of a particular treatment for
a particular grade of stainless steel in a particular application is
demonstrated by meeting the specified testing requirements:

5.1.1 Treatments in nitric acid,
5.1.2 Treatments in citric acid,
5.1.3 Other chemical treatments, including electrochemical
treatments,
5.1.4 Neutralization, and
5.1.5 Post-cleaning treatments.

5.2 Materials:
5.2.1 The chemicals used for passivation treatments shall
produce passivated surfaces that meet the requirements of one
or more of the tests of this specification. Attention shall be
given to maintaining adequate volume, concentration, purity,
and temperature control appropriate to the size and amount of
stainless steel to be treated.

5.2.2 The processor shall maintain a record with regard to
concentration and temperature of the passivation solution
sufficient to demonstrate that the specified passivation condi-
tions were maintained for each lot of stainless steel parts
processed. Such records shall be available for inspection when
specified in the purchase order. The processor is not required to
reveal the precise composition of proprietary chemical mix-
tures but shall maintain a unique identification of the mixture
that will ensure its accurate representation for subsequent use.

5.2.3 The processor shall be responsible for the safe dis-
posal of all material generated by this process.

5.3 Preparation for Passivation Treatments:
5.3.1 The pretreatment methods and procedures used prior
to the passivation treatment, including mechanical and chemi-
cal methods, singly or in combination, for descaling and
pickling, shall be in accordance with Practice A 380. When
electrochemical cleaning is required, it shall be performed in
accordance with Practice B 254.

5.3.2 The resulting pretreated surface shall be substantially
free of oil, grease, rust, scale, and other foreign matter.

5.3.3 When the final pretreatment of a part includes pickling
of the entire surface of the part, no further passivation
treatment is required prior to testing of the surface unless
specified by the purchaser.

6. Treatments in Nitric Acid Solutions

6.1 Passivation Treatment:
6.1.1 Stainless steel parts shall be treated in one of the
following aqueous solutions and maintained within the speci-
fied temperature range for the specified time.

6.1.1.1 Nitric 1—The solution shall contain 20 to 25 volume
percent of nitric acid and 2.5 ± 0.5 weight percent of sodium
dichromate. The parts shall be immersed for a minimum of 20
min at a temperature in the range from 120 to 130°F (49 to
54°C).

6.1.1.2 Nitric 2—The solution shall contain 20 to 45 volume
percent of nitric acid. The parts shall be immersed for a
minimum of 30 min at a temperature in the range from 70 to
90°F (21 to 32°C).

6.1.1.3 Nitric 3—The solution shall contain 20 to 25 volume
percent nitric acid. The parts shall be immersed for a minimum
of 20 min at a temperature in the range from 120 to 140°F (49
to 60°C).
6.1.1.4 Nitric 4—The solution shall contain 45 to 55 volume percent of nitric acid. The parts shall be immersed for a minimum of 30 min at a temperature in the range from 120 to 130°F (49 to 54°C).

6.1.1.5 Nitric 5—Other combinations of temperature, time, and concentration of nitric acid, with or without other chemicals, including accelerants, inhibitors, or proprietary solutions, capable of producing parts that pass the specified test requirements.

6.2 Water Rinse—Immediately after removal from the passivating solution the parts shall be thoroughly rinsed, using stagnant, countercurrent, or spray washes singly or in combination, with or without a separate chemical treatment for neutralization (see 9.1) of the passivation media, with a final rinse being carried out using water with a maximum total solids content of 200 ppm.

7. Treatments in Citric Acid

7.1 Passivation Treatment:

7.1.1 Stainless steel parts shall be treated in one of the following aqueous solutions and maintained within the specified temperature range for the specified time.

7.1.1.1 Citric 1—The solution shall contain 4 to 10 weight percent of citric acid. The parts shall be immersed for a minimum of 4 min at a temperature in the range from 140 to 160°F (60 to 71°C).

7.1.1.2 Citric 2—The solution shall contain 4 to 10 weight percent of citric acid. The parts shall be immersed for a minimum of 10 min at a temperature in the range from 120 to 140°F (49 to 60°C).

7.1.1.3 Citric 3—The solution shall contain 4 to 10 weight percent of citric acid. The parts shall be immersed for a minimum of 20 min at a temperature in the range from 70 to 120°F (21 to 49°C).

7.1.1.4 Citric 4—Other combinations of temperature, time, and concentration of citric acid, with or without other chemicals to enhance cleaning, including accelerants, inhibitors, or proprietary solutions capable of producing parts that pass the specified test requirements.

7.1.1.5 Citric 5—Other combinations of temperature, time, and concentrations of citric acid, with or without other chemicals to enhance cleaning, including accelerants, inhibitors, or proprietary solutions capable of producing parts that pass the specified test requirements.

7.2 Water Rinse—Immediately after removal from the passivating solution, the parts shall be thoroughly rinsed, using stagnant, countercurrent, or spray washes, singly or in combination, with or without a separate chemical treatment for neutralization of the passivation media (see 9.2), with a final rinse being carried out using water with a maximum total solids content of 200 ppm.

8. Treatments in Other Chemical Solutions, Including Electrochemical Treatments

8.1 It is recognized that the purpose of removal of all exogenous matter from a stainless steel surface, including the removal of free iron, can be accomplished by different media, with potential for benefits to be gained from use of proprietary skills and art, including proprietary passivation media. Such treatments may include externally applying an electrical potential on the stainless steel parts, as in the case of electropolishing. The suitability of such passivation treatments for use in meeting the requirements of this specification shall be determined by the capability of the processed parts meeting the specified test requirements.

8.2 Stainless steel parts shall be treated in a specified aqueous solution, with or without externally applied electrical potential, and maintained within a specified temperature range for a time sufficient for the processed parts to meet the specified test requirement.

8.3 Water Rinse—Immediately after removal from the passivating solution, the parts shall be thoroughly rinsed, using stagnant, countercurrent, or spray washes, singly or in combination, with or without a separate chemical treatment for neutralization of the passivation media (see 9.2), with a final rinse being carried out using water with a maximum total solids content of 200 ppm.

9. Neutralization

9.1 The chemical reactions of the passivating media on the surface of the stainless steel shall be stopped by rinsing of the stainless steel part, with or without a separate neutralization treatment.

9.2 The suitability of a neutralization procedure is determined by the capability of the processed parts meeting the specified test requirements (see Note 1).

Note 1—The selection of medium and procedures for a neutralization depends on the chemistry of the passivation and on economic considerations. An example of a neutralizing treatment would be immersion of the part for a minimum of 30 min in a solution of 5% NaOH at 160 to 160°F (71 to 82°C), followed by a water rinse.

10. Post-Cleaning Treatments

10.1 Although the passive film characteristic of stainless steel will form spontaneously in an air or any other oxygen-containing environment, the processor shall, when specified, apply a chemical treatment that will accelerate the formation of the passive film on a chemically clean stainless steel surface. An example of a medium that serves to accelerate the formation of the passive film but does not contribute to the removal of free iron from the stainless steel surface would be an aqueous solution of sodium dichromate.

10.2 When specified, within one hour after the final water rinse as required in 6.2, 7.2, or 8.3, all ferritic and martensitic steel parts shall be immersed in an aqueous solution containing 4 to 6 weight percent of sodium dichromate at a temperature in the range from 140 to 160°F (60 to 71°C) for a minimum of 30 min, followed by a rinse in accordance with 6.2, 7.2, or 8.3. The parts shall then be thoroughly dried.

10.3 The purchaser may specify other post-cleaning treatments.

11. Finish

11.1 The passivated parts shall exhibit a chemically clean surface and shall, on visual inspection, show no etching, pitting, or frosting resulting from the passivation procedures.
12. Testing Agency

12.1 When required, the purchaser shall be permitted to perform such inspections as necessary to determine that the testing agency is capable of performing the specified test.

13. Lot, Frequency of Testing, and Selection of Test

13.1 Definition of Lot—A lot shall consist of one of the following, at the option of the processor:

13.1.1 The passivated parts of similar alloy and manufacturing methods that are pretreated and passivated in a single day or within a time frame that will ensure consistent passivation results.

13.1.2 The passivated parts of the same product of one size from one heat in one shipment; or

13.1.3 When few parts are involved, the passivated parts from an entire production run.

13.2 Unless a greater frequency of testing is specified on the purchase order, one test per lot shall be sufficient.

13.3 One or more of the following tests, when specified on the purchase order, shall be performed on each lot of stainless steel parts. Not all of the following tests are suitable for all grades of stainless steel. (See Note 2.)

13.3.1 Practice A—Water Immersion Test,
13.3.2 Practice B—High Humidity Test,
13.3.3 Practice C—Salt Spray Test,
13.3.4 Practice D—Copper Sulfate Test, and
13.3.5 Practice E—Potassium Ferricyanide–Nitric Acid Test.

Note 2—Some of the tests may produce positive indications not associated with the presence of free iron on the stainless steel surface. An example would be application of Practice C on some lesser-alloyed martensitic or ferritic stainless steels.

14. Practice A—Water Immersion Test

14.1 This test is used for the detection of free iron or any other anodic surface contaminants on stainless steel.

14.2 The sample representing the lot of passivated parts shall be alternately immersed in a non-rusting tank of distilled water for 1 h and allowed to dry in air for 1 h. This cycle shall be repeated a minimum of twelve times.

14.3 The tested sample shall not exhibit rust or staining attributable to the presence of free iron particles embedded in the surface.

15. Practice B—High Humidity Test

15.1 This test is used for the detection of free iron or any other anodic surface contaminants on stainless steel.

15.2 The test shall be performed using a humidity cabinet capable of maintaining the specified test conditions.

15.3 The sample representing the lot of passivated parts shall be cleaned by immersion in acetone or methyl alcohol or by swabbing with a clean gauze saturated with acetone or methyl alcohol, and dried in an inert atmosphere or desiccated container. The cleaned and dried part shall be subjected to 97 ± 3 % humidity at 100 ± 5°F (38 ± 3°C) for a minimum of 24 h.

15.4 The tested sample shall not exhibit rust or staining attributable to the presence of free iron particles imbedded in the surface.

16. Practice C—Salt Spray Test

16.1 This test is used for the detection of free iron or any other anodic surface contaminants on stainless steel.

16.2 The sample representing the lot of passivated parts shall be tested by the salt spray test conducted in accordance with Practice B 117 for a minimum of 2 h using a 5 % salt solution.

16.3 The tested sample shall not exhibit rust or staining attributable to the presence of free iron particles imbedded in the surface.

17. Practice D—Copper Sulfate Test

17.1 This test is recommended for the detection of free iron on the surface of austenitic stainless steels in the 200 and 300 series, precipitation hardened stainless steels, and ferritic 400 series stainless steels having a minimum of 16 % chromium. This test is not recommended for martensitic 400 series stainless steels or for ferritic 400 series stainless steels with less than 16 % chromium because these steels will give a positive indication irrespective of the presence or absence of anodic surface contaminants. This test shall not be applied to parts to be used in food processing.

17.2 The test solution is prepared by dissolving 4 g of copper sulfate pentahydrate (CuSO₄.5H₂O) in 250 mL of distilled water to which 1 mL of sulfuric acid (H₂SO₄, sp gr 1.84) has been added.

17.3 The test solution is swabbed on the surface of the sample representing the lot of passivated parts, applying additional solution as needed to keep the surface wet for a period of at least 6 min. At the end of this period, the surface shall be carefully rinsed and dried with care taken not to disturb copper deposits if present.

17.4 The tested sample shall not exhibit copper deposits.

18. Practice E—Potassium Ferricyanide–Nitric Acid Test

18.1 This test is recommended when detection of very small amounts of free iron is required. It is recommended for detection of free iron on austenitic 200 and 300 series stainless steels. This test is not recommended for detection of free iron on ferritic or martensitic 400 series stainless steels, because these steels will give a positive indication irrespective of the presence or absence of anodic surface contaminants. This test shall not be applied to parts to be used in food processing.

18.2 The test solution is prepared by adding 10 g of chemically pure potassium ferricyanide to 500 mL of distilled water, adding 30 mL of 70 % nitric acid, agitating until all of the ferricyanide is dissolved, and diluting to 1000 mL with distilled water. The test solution shall be mixed fresh on the day of the test.

18.3 The test solution is swabbed on the surface of the sample representing the lot of passivated parts. The formation of a dark blue color within 30 s denotes the presence of metallic iron.

18.4 The tested sample shall not exhibit the dark blue color indicative of free iron on the surface.

18.5 When the test is negative, the surface shall be thoroughly washed with warm water to removal all traces of the test solution. When the test is positive, the dark blue stain shall
be removed with a solution of 10% acetic acid and 8% oxalic acid, followed by a thorough hot water rinse.

19. Practice F—Free Iron Test

19.1 This test is used for the detection of free iron on the surface of stainless steel. It is especially useful for large parts that have been uniformly cleaned but that are inconvenient for reasons of size of equipment or ease of handling of the part to place in the environments defined in Practice A (Section 14) or Practice B (Section 15). Unless otherwise specified by the purchaser, the number of tests and the locations of the tests shall be at the option of the processor to assure a representative testing of the part.

19.2 The test is performed by placing a clean cloth pad that has been thoroughly soaked with distilled or demineralized water on the surface of the part at a part temperature of 50°F (10°C) or greater for a period of not less than 60 minutes. The cloth shall be in contact with the steel for an area of at least 20 square inches (130 cm²). The pad shall be maintained wet through the test period, either by a method of retarding external evaporation, by the further addition of potable water, or by backing the pad with a sponge or similar water source. The cloth pad used shall be used for only one such test, being changed for each test as to avoid risk of contamination. After removal of the cloth pad, the surface of the part shall be allowed to dry in air before inspection.

19.3 The tested part shall not exhibit rust or staining attributable to the presence of free iron particles embedded in the surface.

20. Rejection and Retest

20.1 Any lot failing to meet the specified test requirements of the purchase order shall be rejected. A rejected lot may, at the option of the processor, be re-passivated, with or without re-pretreatment, and then be retested. The number of samples tested from a lot subject to retest shall be twice the original specified test frequency, to the limit of the number of pieces in the lot. All samples must pass the specified acceptance criterion for the specified test for the retested lot to be accepted.

21. Precision and Bias

21.1 No statement is made concerning either the precision or bias of Practices A, B, C, D, and E because the results state merely whether there is conformance to the criteria for success specified in the procedure.

22. Certification

22.1 When specified in the purchase order, a report of the practice and tests used, including the record of process conditions when specified in accordance with 3.1.1.2, shall be supplied to the purchaser.

23. Keywords

23.1 cleaning of stainless steel; descaling; passivation; tests for cleanliness of stainless steels;

APPENDIX

(Nonmandatory Information)

X1. INFORMATION REGARDING PASSIVATION TREATMENTS

Note X1.1—The following information is based on a section of Federal Specification QQ-P-35C (Oct. 28, 1988) identified as information of a general or explanatory nature that may be helpful, but is not mandatory. Minor changes have been made in the text to facilitate references to the main document and to correct technical inaccuracies.

X1.1 Intended Use—The passivation treatments provided by this specification are intended to improve the corrosion resistance of parts made from stainless steel of all types.

X1.1.1 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 stainless steel parts. These particles must be removed or they will appear as rust or stain spots. This condition may be prevented by chemically treating the parts to remove the iron particles or other foreign particles, and then allowing the passive film to form on the cleaned surface, with or without chemical enhancement of the formation of this oxide film.

X1.1.2 This specification is not intended for the black oxide coating of parts typically used for photographic or optical instruments.

X1.2 Ordering Data—Purchasers should select the preferred options permitted by this specification and include the following information in the purchase order:

X1.2.1 Title, number, and date of this specification;

X1.2.2 Identification of material by type and applicable product specification;

X1.2.3 Test practices to be imposed (see 1.4);

X1.2.4 Definition of lot size, if other than described in this specification; and

X1.2.5 Required documentation, if other than the minimum required by this specification.

X1.3 Grades of Stainless Steel—Different types of stainless steel are selected on the basis of properties required, for example, corrosion resistance and design criteria, and fabrication requirements. Table X1.1 is a compilation that serves as a guide for the selection of passivation treatment for different grades, but is far from complete either in grades or in passivation treatments.

X1.4 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, or spray rinses, or both, prior to final rinse.
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**FIG. X1.1 Recommended Nitric Acid Passivation Treatments for Different Grades of Stainless Steel**

X1.5 *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.

X1.6 *Test Specimens*—When using test specimens instead of parts, the specimens can effectively represent the parts only if they have been exposed to the same processing steps, such as machining, grinding, heat treating, welding, and so forth, as the parts they are to represent.

X1.7 *Carburized Surfaces*—Stainless steel parts with carburized surfaces cannot be passivated because the carbon combines with the chromium forming chromium carbides on the surface.

X1.8 *Nitrided Surfaces*—Stainless steel parts with nitrided surfaces should not be passivated because the treatment will severely corrode the nitrided case.

X1.9 This specification provides for the same passivation treatments as Fed. Spec. QQ-P-35C, but also includes a number of alternative passivation treatments. The effectiveness of any passivation treatment is demonstrated by the parts meeting the specified testing requirements after treatment.

X1.10 *Martensitic Grade 440C*—High-strength grades 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.

X1.11 The salt spray test is typically used to evaluate austenitic stainless steels and may not be applicable to all martensitic or ferritic stainless steels.
SUMMARY OF CHANGES

Committee A01 has identified the location of selected changes to this standard since the last issue, A 967 – 01, that may impact the use of this standard. (Approved Sept. 1, 2005.)

(1) Revised Section 1.3.
(2) Deleted requirement on age of copper sulfate solution in Section 17.2.

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