Designation: A 380 – 06

Standard Practice for Cleaning, Descaling, and Passivation of Stainless Steel Parts, Equipment, and Systems[1]

This standard is issued under the fixed designation A 380; 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 Department of Defense.

1. Scope*

1.1 This practice covers recommendations and precautions for cleaning, descaling, and passivating of new stainless steel parts, assemblies, equipment, and installed systems. These recommendations are presented as procedures for guidance when it is recognized that for a particular service it is desired to remove surface contaminants that may impair the normal corrosion resistance, or result in the later contamination of the particular stainless steel grade, or cause product contamination. For certain exceptional applications, additional requirements which are not covered by this practice may be specified upon agreement between the manufacturer and the purchaser. Although they apply primarily to materials in the composition ranges of the austenitic, ferritic, and martensitic stainless steels, the practices described may also be useful for cleaning other metals if due consideration is given to corrosion and possible metallurgical effects.

1.1.1 The term passivation is commonly applied to several distinctly different operations or processes relating to stainless steels. In order to avoid ambiguity in the setting of requirements, it may be necessary for the purchaser to define precisely the intended meaning of passivation. Some of the various meanings associated with the term passivation that are in common usage include the following:

1.1.1.1 Passivation is the process by which a stainless steel will spontaneously form a chemically inactive surface when exposed to air or other oxygen-containing environments. It was at one time considered that an oxidizing treatment was necessary to establish this passive film, but it is now accepted that this film will form spontaneously in an oxygen-containing environment providing that the surface has been thoroughly cleaned or descaled.

1.1.1.2 Passivation is removal of exogenous iron or iron compounds from the surface of a stainless steel by means of a chemical dissolution, most typically by a treatment with an acid solution that will remove the surface contamination but will not significantly affect the stainlees steel itself. This process is described in a general way in 6.2.11 and defined precisely in 6.4 with further reference to the requirements of Annex A2 and Part II of the table on acid cleaning of steel. Unless otherwise specified, it is this definition of passivation that is taken as the meaning of a specified requirement for passivation.

1.1.1.3 Passivation is the chemical treatment of a stainless steel with a mild oxidant, such as a nitric acid solution, for the purpose of enhancing the spontaneous formation of the protective passive film. Such chemical treatment is generally not necessary for the formation of the passive film.

1.1.1.4 Passivation does not indicate the separate process of descaling as described in Section 5, although descaling may be necessary before passivation can be effective.

1.2 This practice does not cover decontamination or cleaning of equipment or systems that have been in service, nor does it cover descaling and cleaning of materials at the mill. On the other hand, some of the practices may be applicable for these purposes. While the practice provides recommendations and information concerning the use of acids and other cleaning and descaling agents, it cannot encompass detailed cleaning procedures for specific types of equipment or installations. It therefore in no way precludes the necessity for careful planning and judgment in the selection and implementation of such procedures.

1.3 These practices may be applied when free iron, oxide scale, rust, grease, oil, carbonaceous or other residual chemical films, soil, particles, metal chips, dirt, or other nonvolatile deposits might adversely affect the metallurgical or sanitary condition or stability of a surface, the mechanical operation of a part, component, or system, or contaminate a process fluid. The degree of cleanness required on a surface depends on the application. In some cases, no more than degreasing or removal

[1] This practice is under the jurisdiction of ASTM Committee A01 on Steel, Stainless Steel and Related Alloys and is the direct responsibility of Subcommittee A01.14 on Methods of Corrosion Testing.

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***A Summary of Changes section appears at the end of this standard.**

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of gross contamination is necessary. Others, such as food-handling, pharmaceutical, aerospace, and certain nuclear applications, may require extremely high levels of cleanness, including removal of all detectable residual chemical films and contaminants that are invisible to ordinary inspection methods.

NOTE 1—The term "iron," when hereinafter referred to as a surface contaminant, shall denote free iron.

1.4 Attainment of surfaces that are free of iron, metallic deposits, and other contamination depends on a combination of proper design, fabrication methods, cleaning and descaling, and protection to prevent recontamination of cleaned surfaces. Meaningful tests to establish the degree of cleanness of a surface are few, and those are often difficult to administer and to evaluate objectively. Visual inspection is suitable for the detection of gross contamination, scale, rust, and particulates, but may not reveal the presence of thin films of oil or residual chemical films. In addition, visual inspection of internal surfaces is often impossible because of the configuration of the item. Methods are described for the detection of free iron and transparent chemical and oily deposits.

1.5 This practice provides definitions and describes good pratices for cleaning, descaling, and passivation of stainless steel parts, but does not provide tests with acceptance criteria to demonstrate that the passivation procedures have been successful. For such tests, it is appropriate to specify one of the practices listed in Specification A 967.

1.6 *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.* (For more specific safety precautions see 7.2.5.3, 7.3.4, Section 8, A1.7, and A2.11.)

2. Referenced Documents

2.1 *ASTM Standards:* [2]

A 967 Specification for Chemical Passivation Treatments for Stainless Steel Parts

F 21 Test Method for Hydrophobic Surface Films by the Atomizer Test

F 22 Test Method for Hydrophobic Surface Films by the Water-Break Test

2.2 *Federal Standard:*[3]

Fed. Std. No. 209e for Clean Room and Work Station Requiring Controlled Environments

3. Design

3.1 Consideration should be given in the design of parts, equipment, and systems that will require cleaning to minimize the presence of crevices, pockets, blind holes, undrainable cavities, and other areas in which dirt, cleaning solutions, or sludge might lodge or become trapped, and to provide for effective circulation and removal of cleaning solutions. In equipment and systems that will be cleaned in place or that cannot be immersed in the cleaning solution, it is advisable to slope lines for drainage: to provide vents at high points and drains at low points of the item or system; to arrange for removal or isolation of parts that might be damaged by the cleaning solution or fumes from the cleaning solutions; to provide means for attaching temporary fill and circulation lines; and to provide for inspection of cleaned surfaces.

3.2 In a complex piping system it may be difficult to determine how effective a cleaning operation has been. One method of designing inspectability into the system is to provide a short flanged length of pipe (that is, a spool piece) at a location where the cleaning is likely to be least effective; the spool piece can then be removed for inspection upon completion of cleaning.

4. Precleaning

4.1 Precleaning is the removal of grease, oil, paint, soil, grit, and other gross contamination preparatory to a fabrication process or final cleaning. Precleaning is not as critical and is generally not as thorough as subsequent cleaning operations. Materials should be precleaned before hot-forming, annealing, or other high-temperature operation, before any descaling operation, and before any finish-cleaning operation where the parts will be immersed or where the cleaning solutions will be reused. Items that are subject to several redraws or a series of hot-forming operations, with intermediate anneals, must be cleaned after each forming operation, prior to annealing. Precleaning may be accomplished by vapor degreasing; immersion in, spraying, or swabbing with alkaline or emulsion cleaners, steam, or high-pressure water-jet (see 6.2).

5. Descaling

5.1 *General*—Descaling is the removal of heavy, tightly adherent oxide films resulting from hot-forming, heat-treatment, welding, and other high-temperature operations. Because mill products are usually supplied in the descaled condition, descaling (except removal of localized scale resulting from welding) is generally not necessary during fabrication of equipment or erection of systems (see 6.3). When necessary, scale may be removed by one of the chemical methods listed below, by mechanical methods (for example, abrasive blasting, sanding, grinding, power brushing), or by a combination of these.

5.2 *Chemical Descaling (Pickling)*—Chemical descaling agents include aqueous solutions of sulfuric, nitric, and hydrofluoric acid as described in Annex A1, Table A1.1, molten alkali or salt baths, and various proprietary formulations.

5.2.1 *Acid Pickling*—Nitric-hydrofluoric acid solution is most widely used by fabricators of stainless steel equipment and removes both metallic contamination, and welding and heat-treating scales. Its use should be carefully controlled and is not recommended for descaling sensitized austenitic stainless steels or hardened martensitic stainless steels or where it can come into contact with carbon steel parts, assemblies, equipment, and systems. See also A1.3. Solutions of nitric acid alone are usually not effective for removing heavy oxide scale.

[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.

[3] Available from Standardization Documents Order Desk, Bldg 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

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5.2.2 Surfaces to be descaled are usually precleaned prior to chemical treatment. When size and shape of product permit, total immersion in the pickling solution is preferred. Where immersion is impractical, descaling may be accomplished by (*1*) wetting the surfaces by swabbing or spraying; or (*2*) by partially filling the item with pickling solution and rotating or rocking to slosh the solution so that all surfaces receive the required chemical treatment. The surface should be kept in contact with agitated solution for about 15 to 30 min or until inspection shows that complete scale removal has been accomplished. Without agitation, additional exposure time may be required. If rocking or rotation are impracticable, pickling solution may be circulated through the item or system until inspection shows that descaling has been accomplished.

5.2.3 Over-pickling must be avoided. Uniform removal of scale with acid pickling depends on the acid used, acid concentration, solution temperature, and contact time (see Annex A1). Continuous exposure to pickling solutions for more than 30 min is not recommended. The item should be drained and rinsed after 30 min and examined to check the effectiveness of the treatment. Additional treatment may be required. Most pickling solutions will loosen weld and heat-treating scale but may not remove them completely. Intermittent scrubbing with a stainless steel brush or fiber-bristle brush, in conjunction with pickling or the initial rinse, may facilitate the removal of scale particles and products of chemical reaction (that is, pickling *smut*).

5.2.4 After chemical descaling, surfaces must be thoroughly rinsed to remove residual chemicals; a neutralization step is sometimes necessary before final rinsing. To minimize staining, surfaces must not be permitted to dry between successive steps of the acid descaling and rinsing procedure, and thorough drying should follow the final water rinse. Chemical descaling methods, factors in their selection, and precautions in their use are described in the *Metals Handbook*.[4] When chemical descaling is necessary, it should be done while the part is in its simplest possible geometry, before subsequent fabrication or installation steps create internal crevices or undrainable spaces that may trap descaling agents, sludge, particles, or contaminated rinse water that might either result in eventual corrosion or adversely affect operation of the item after it is placed in service.

5.3 *Mechanical Descaling*—Mechanical descaling methods include abrasive blasting, power brushing, sanding, grinding, and chipping. Procedural requirements and precautions for some of these methods are given in the *Metals Handbook*.[4] Mechanical descaling methods have the advantage that they do not produce such physical or chemical conditions as intergranular attack, pitting, hydrogen embrittlement, cracks, or smut deposits. For some materials, in particular the austenitic stainless steels when in the sensitized condition and the martensitic stainless steels when in the hardened condition, mechanical descaling may be the only suitable method. Grinding is usually the most effective means of removing localized scale such as that which results from welding. Disadvantages of mechanical descaling are cost, as compared to chemical descaling, and the fact that surface defects (for example, laps, pits, slivers) may be obscured, making them difficult to detect.

5.3.1 Surfaces to be descaled may have to be precleaned. Particular care must be taken to avoid damage by mechanical methods when descaling thin sections, polished surfaces, and close-tolerance parts. After mechanical descaling, surfaces should be cleaned by scrubbing with hot water and fiber brushes, followed by rinsing with clean, hot water.

5.3.2 Grinding wheels and sanding materials should not contain iron, iron oxide, zinc, or other undersirable materials that may cause contamination of the metal surface. Grinding wheels, sanding materials, and wire brushes previously used on other metals should not be used on stainless steel. Wire brushes should be of a stainless steel which is equal in corrosion resistance to the material being worked on.

5.3.3 Clean, previously unused abrasives, such as glass beads or iron-free silica or alumina sand, are recommended for abrasive blasting. Steel shot or grit is generally not recommended because of the possibility of embedding iron particles. The use of stainless steel shot or grit reduces the danger of rusting and iron contamination, but cannot completely eliminate the possibility of embedding residues of iron-oxide scale.

5.3.4 If a totally iron and scale free surface is required, most abrasive blasting may be followed by a brief acid dip (see Annex A2).

6. Cleaning

6.1 *General*—Cleaning includes all operations necessary for the removal of surface contaminants from metals to ensure (*1*) maximum corrosion resistance of the metal; (*2*) prevention of product contamination; and (*3*) achievement of desired appearance. Cleanness is a perishable condition. Careful planning is necessary to achieve and maintain clean surfaces, especially where a high degree of cleanness is required. Selection of cleaning processes is influenced mainly by the type of contaminant to be removed, the required degree of cleanness, and cost. If careful control of fabrication processes, sequencing of cleaning and fabrication operations, and measures to prevent recontamination of cleaned surfaces are exercised, very little special cleaning of the finished item or system may be necessary to attain the desired level of cleanness. If there is a question concerning the effectiveness of cleaning agents or procedures, or the possible adverse effects of some cleaning agents or procedures on the materials to be cleaned, trial runs, using test specimens and sensitive inspection techniques may be desirable. Descriptions, processes, and precautions to be observed in cleaning are given in the *Metals Handbook*.[4] Proprietary cleaners may contain harmful ingredients, such as chlorides or sulfur compounds, which could adversely affect the performance of a part, equipment, or system under service conditions. It is recommended that the manufacturer of the cleaner be consulted if there is reason for concern.

NOTE 2—Instances are known where stainless steel vessels have stress cracked before start-up due to steaming out or boiling out with a chloride-containing detergent.

[4] "Surface Cleaning, Finishing, and Coating," *Metals Handbook,* Am. Soc. Metals, 9th ed., Vol 5, 1982.

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6.2 *Cleaning Methods*—Degreasing and general cleaning may be accomplished by immersion in, swabbing with, or spraying with alkaline, emulsion, solvent, or detergent cleaners or a combination of these; by vapor degreasing; by ultrasonics using various cleaners; by steam, with or without a cleaner; or by high-pressure water-jetting. The cleaning method available at any given time during the fabrication or installation of a component or system is a function of the geometric complexity of the item, the type of contamination present, the degree of cleanliness required, and cost. Methods commonly used for removing deposited contaminants (as opposed to scale) are described briefly below and in greater detail (including factors to be considered in their selection and use) in the *Metals Handbook*[4] and the *SSPC Steel Structures Painting Handbook.*[5] The safety precautions of 8.6 must be observed in the use of these methods. Particular care must be exercised when cleaning closed systems and items with crevices or internal voids to prevent retention of cleaning solutions and residues.

6.2.1 *Alkaline Cleaning* is used for the removal of oily, semisolid, and solid contaminants from metals. To a great extent the solutions used depend on their detergent qualities for cleaning action and effectiveness. Agitation and temperature of the solution are important.

6.2.2 *Emulsion Cleaning* is a process for removing oily deposits and other common contaminants from metals by the use of common organic solvents dispersed in an aqueous solution with the aid of a soap or other emulsifying agent (an emulsifying agent is one which increases the stability of a dispersion of one liquid in another). It is effective for removing a wide variety of contaminants including pigmented and unpigmented drawing compounds and lubricants, cutting fluids, and residues resulting from liquid penetrant inspection. Emulsion cleaning is used when rapid, superficial cleaning is required and when a light residual film of oil is not objectionable.

6.2.3 *Solvent Cleaning* is a process for removing contaminants from metal surfaces by immersion or by spraying or swabbing with common organic solvents such as the aliphatic petroleums, chlorinated hydrocarbons, or blends of these two classes of solvents. Cleaning is usually performed at or slightly above room temperature. Except for parts with extremely heavy contamination or with hard-to-reach areas, or both, good agitation will usually eliminate the need for prolonged soaking. Virtually all metal can be cleaned with the commonly used solvents unless the solvent has become contaminated with acid, alkali, oil, or other foreign material. Chlorinated solvents are not recommended for degreasing of closed systems or items with crevices or internal voids.

6.2.4 *Vapor Degreasing* is a generic term applied to a cleaning process that employs hot vapors of a volatile chlorinated solvent to remove contaminants, and is particularly effective against oils, waxes, and greases. The cleanness and chemical stability of the degreasing solvent are critical factors in the efficiency of the vapor and possible chemical attack of the metal. Water in the degreasing tank or on the item being cleaned may react with the solvent to form hydrochloric acid, which may be harmful to the metal. No water should be present in the degreasing tank or on the item being cleaned. Acids, oxidizing agents, and cyanides must be prevented from contaminating the solvent. Materials such as silicones cause foaming at the liquid-vapor interface and may result in recontamination of the workpiece as it is removed from the degreaser. Vapor degreasing with chlorinated solvents is not recommended for closed systems or items with internal voids or crevices.

6.2.5 *Ultrasonic Cleaning* is often used in conjunction with certain solvent and detergent cleaners to loosen and remove contaminants from deep recesses and other difficult to reach areas, particularly in small work-pieces. Cavitation in the liquid produced by the high frequency sound causes micro agitation of the solvent in even tiny recesses of the workpiece, making the method especially desirable for cleaning parts or assemblies having an intricate configuration. For extremely high levels of surface cleanness, high-purity solvents (1 ppm total nonvolatile residue) are required.

6.2.6 *Synthetic Detergents* are extensively used as surface-active agents because they are freer rinsing than soaps, aid in soils dispersion, and prevent recontamination. They are effective for softening hard water and in lowering the surface and interfacial tensions of the solutions. Synthetic detergents, in particular, should be checked for the presence of harmful ingredients as noted in 6.1.

6.2.7 *Chelate Cleaning*—Chelates are chemicals that form soluble, complex molecules with certain metal ions, inactivating the ions in solution so they cannot normally react with another element or ions to produce precipitates or scale. They enhance the solubility of scales and certain other contaminants, do not precipitate different scales when the cleaning solution becomes spent, and can be used on some scales and contaminants that even mineral acids will not attack. When properly used (chelating agents must be continuously circulated and must be maintained within carefully controlled temperature limits), intergranular attack, pitting, and other harmful effects are minimal. Chelating agents are particularly useful for cleaning installed equipment and systems.

6.2.8 *Mechanical Cleaning (also see 5.3)*—Abrasive blasting, vapor blasting using a fine abrasive suspended in water, grinding, or wire brushing are often desirable for removing surface contaminants and rust. Cleanliness of abrasives and cleaning equipment is extremely important to prevent recontamination of the surfaces being cleaned. Although surfaces may appear visually clean following such procedures, residual films which could prevent the formation of an optimum passive condition may still be present. Subsequent treatment such as additional iron-free abrasive cleaning methods, acid cleaning, passivation, or combinations of these is, therefore, required for stainless steel parts, equipment, and systems to be used where corrosion resistance is a prime factor to satisfy performance and service requirements, or where product contamination must be avoided.

6.2.9 *Steam Cleaning* is used mostly for cleaning bulky objects that are too large for soak tanks or spray-washing equipment. It may be used with cleaning agents such as

[5] *Good Painting Practices,* Steel Structures Painting Council, Vol 1, 1982, Chapters 2.0–2.9, 3.1–3.2.

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emulsions, solvents, alkalis, and detergents. Steam lances are frequently used for cleaning piping assemblies. Steam pressures from 50 to 75 psi (345 to 515 kPa) are usually adequate (see 6.1).

6.2.10 *Water-Jetting* at water pressures of up to 10 000 psi (70 mPa) is effective for removing grease, oils, chemical deposits (except adsorbed chemicals), dirt, loose and moderately adherent scale, and other contaminants that are not actually bonded to the metal. The method is particularly applicable for cleaning piping assemblies which can withstand the high pressures involved; self-propelled nozzles or "moles" are generally used for this purpose.

6.2.11 *Acid Cleaning* is a process in which a solution of a mineral or organic acid in water, sometimes in combination with a wetting agent or detergent or both, is employed to remove iron and other metallic contamination, light oxide films, shop soil, and similar contaminants. Suggested solutions, contact times, and solution temperatures for various alloys are given in Annex A2. Acid cleaning is not generally effective for removal of oils, greases, and waxes. Surfaces should be precleaned to remove oils and greases before acid cleaning. Common techniques for acid cleaning are immersion, swabbing, and spraying. Maximum surface quality is best achieved by using a minimum cleaning time at a given acid concentration and temperature. After acid cleaning the surfaces must be thoroughly rinsed with clean water to remove all traces of the acid and thoroughly dried after the final water rinse. To minimize staining, surfaces must not be permitted to dry between successive steps of the acid cleaning and rinsing procedure. A neutralizing treatment may be required under some conditions; if used, neutralization must be followed by repeated water rinsing to remove all trace of the neutralizing agent followed by thorough drying after the final water rinse. Acid cleaning is not recommended where mechanical cleaning or other chemical methods will suffice on the basis of intended use and, as may be necessary, on inspection tests (see 7.2 and 7.3). Requirements for superfluous cleaning and inspection testing can result in excessive costs. Acid cleaning, if not carefully controlled, may damage the surface and may result in further contamination of the surface.

6.3 *Cleaning of Welds and Weld-Joint Areas*—The joint area and surrounding metal for several inches back from the joint preparation, on both faces of the weld, should be cleaned immediately before starting to weld. Cleaning may be accomplished by brushing with a clean stainless steel brush or scrubbing with a clean, lint-free cloth moistened with solvent, or both. When the joint has cooled after welding, remove all accessible weld spatter, welding flux, scale, arc strikes, etc., by grinding. According to the application, some scale or heat temper may be permissible on the nonprocess side of a weld, but should be removed from the process side if possible. If chemical cleaning of the process side of the weld is deemed necessary, the precautions of this standard must be observed. Austenitic stainless steels in the sensitized condition should generally not be descaled with nitric-hydrofluoric acid solutions. Welds may also be cleaned as described in Table A2.1, Part III, Treatment *P* and *Q* (also see 5.2.3 and 5.2.4).

6.4 *Final Cleaning or Passivation, or Both*—If proper care has been taken in earlier fabrication and cleaning, final cleaning may consist of little more than scrubbing with hot water or hot water and detergent (such as trisodium phosphate, TSP), using fiber brushes. Detergent washing must be followed by a hot-water rinse to remove residual chemicals. Spot cleaning to remove localized contamination may be accomplished by wiping with a clean, solvent-moistened cloth. If the purchaser specifies passivation, the final cleaning shall be in accordance with the requirements of Table A2.1, Part II. When the stainless steel parts are to be used for applications where corrosion resistance is a prime factor to achieve satisfactory performance and service requirements, or where product contamination must be avoided, passivation followed by thorough rinsing several times with hot water and drying thoroughly after the final water rinse is recommended, whenever practical.

6.5 *Precision Cleaning*—Certain nuclear, space, and other especially critical applications may require that only very high-purity alcohols, acetone, ketones, or other *precision cleaning agents* be used for final cleaning or recleaning of critical surfaces after fabrication advances to the point that internal crevices, undrainable spaces, blind holes, or surfaces that are not accessible for thorough scrubbing, rinsing, and inspection are formed. Such items are often assembled under clean-room conditions (see 8.5.5) and require approval, by the purchaser, of carefully prepared cleaning procedures before the start of fabrication.

6.6 *Cleaning of Installed Systems*—There are two approaches to cleaning installed systems. In the first, which is probably adequate for most applications, cleaning solutions are circulated through the completed system after erection, taking care to remove or protect items that could be damaged during the cleaning operation. In the second approach, which may be required for gaseous or liquid oxygen, liquid metal, or other reactive-process solutions, piping and components are installed in a manner to avoid or minimize contamination of process-solution surfaces during erection so that little additional cleaning is necessary after erection; post-erection flushing, if necessary, is done with the process fluid. If process surfaces are coated with an appreciable amount of iron oxide, a chelating treatment or high-pressure water-jetting treatment should be considered in place of acid treatment (see 6.2.7 and 6.2.10).

6.6.1 *Post-Erection Cleaning*—Circulate hot water to which a detergent has been added, for at least 4 to 8 h. A water temperature of at least 140 to 160°F (60 to 71°C) is recommended (see 6.1). Rinse by circulating clean hot water until the effluent is clear. If excessive particulate matter is present, the cleaning cycle may be preceded with a high-pressure steam blow, repeating as necessary until a polished-aluminum target on the outlet of the system is no longer dulled and scratched by particulates loosened by the high-velocity steam. Valves and similar items must be protected from damage during a steam blow.

6.6.2 If metallic iron is indicated by one of the methods suggested in Section 7, it can be removed by circulating one of the acid cleaning solutions suggested in Annex A2 at room temperature until laboratory determination for iron, made on samples of the solution taken hourly, indicates no further

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increase in iron content, after which circulation may be stopped and the system drained. After this treatment, circulate clean hot water (that is, without detergent) through the system for 4 h to remove all traces of acid and corrosion product resulting from the acid treatment, or until the pH of the rinse water returns to neutral.

6.6.3 In critical systems where post-erection cleaning is not desirable (for example, liquid oxygen or nuclear reactor primary coolant systems), on-site erection may be conducted under clean-room conditions. Erection instructions may require that wrapping and seals of incoming materials and equipment be kept intact until the item is inside the clean area, and that careful surveillance be exercised to prevent foreign materials (for example, cleaning swabs or tools) from being dropped or left in the system. Where contamination does occur, the cleaning procedure usually is developed through consultation between the erector and the purchaser (or his site representative). Frequently, post-erection flushing is accomplished by circulating the process fluid through the system until contamination is reduced to tolerable levels.

6.6.4 When cleaning critical installed systems, do not permit the process surfaces to dry between successive cleaning and rinsing steps, or between the final rinse and filling with the layup solution.

7. Inspection After Cleaning

7.1 *General*—Inspection techniques should represent careful, considered review of end use requirements of parts, equipment, and systems. There is no substitute for good, uniform, cleaning practices which yield a metallurgically sound and smooth surface, followed by adequate protection to preserve that condition. Establishment of the most reliable tests and test standards for cleanness are helpful in attaining the desired performance of parts, equipment, and systems. Testing should be sufficiently extensive to ensure the cleanness of all surfaces exposed to process fluids when in service. The following represent some tests that have been successfully applied to stainless steels. The purchaser shall have the option of specifying in his purchase documents that any of these quality assurance tests be used as the basis for acceptability of the cleanness or state of passivity of the stainless steel item.

7.2 *Gross Inspection*:

7.2.1 *Visual*—Items cleaned in accordance with this practice should be free of paint, oil, grease, welding flux, slag, heat-treating and hot-forming scale (tightly adherent scale resulting from welding may be permissible on some surfaces), dirt, trash, metal and abrasive particles and chips, and other gross contamination. Some deposited atmospheric dust will normally be present on exterior surfaces but should not be present on interior surfaces. Visual inspection should be carried out under a lighting level, including both general and supplementary lighting, of at least 100 footcandles (1076 lx), and preferably 250 footcandles (2690 lx) on the surfaces being inspected. Visual inspection should be supplemented with borescopes, mirrors, and other aids, as necessary, to properly examine inaccessible or difficult-to-see surfaces. Lights should be positioned to prevent glare on the surfaces being inspected.

7.2.2 *Wipe Tests*—Rubbing of a surface with a clean, lint-free, white cotton cloth, commercial paper product, or filter paper moistened (but not saturated) with high-purity solvent (see 6.5), may be used for evaluating the cleanness of surfaces not accessible for direct visual inspection. Wipe tests of small diameter tubing are made by blowing a clean white felt plug, slightly larger in diameter than the inside diameter of the tube, through the tube with clean, dry, filtered compressed air. Cleanness in wipe tests is evaluated by the type of contamination rubbed off on the swab or plug. The presence of a smudge on the cloth is evidence of contamination. In cases of dispute concerning the harmful nature of the contamination, a sample of the smudge may be transferred to a clean quartz microscope slide for infrared analysis. The wipe test is sometimes supplemented by repeating the test with a black cloth to disclose contaminants that would be invisible on a white cloth.

7.2.3 *Residual Pattern*—Dry the cleaned surface after finish-cleaning at 120°F (49°C) for 20 min. The presence of stains or water spots on the dried surfaces indicates the presence of residual soil and incomplete cleaning. The test is rapid but not very sensitive.

7.2.4 *Water-Break Test*—This is a test for the presence of hydrophobic contaminants on a cleaned surface. It is applicable only for items that can be dipped in water and should be made with high-purity water. The test procedure and interpretation of results are described in Test Method F 22. The test is moderately sensitive.

7.2.5 *Tests for Free Iron: Gross Indications*—When iron contamination is clearly visible, items should be cleaned in accordance with this practice.

7.2.5.1 *Water-Wetting and Drying*—Formation of rust stains may be accelerated by periodically wetting the surface with preferably distilled or deionized water or clean, fresh, potable tap water. The wet-dry cycles should be such that the sample remains dry for a total of 8 h in a 24-h test period. After completion of this test, the surface should show no evidence of rust stains or other corrosion products.

7.2.5.2 *High-Humidity Test*—Subject the surface to a 95 to 100 % humidity at 100 to 115°F (38 to 46°C) in a suitable humidity cabinet for 24 to 26 h. After completion of this test, the surface should show no evidence of rust stains or other corrosion products.

7.2.5.3 *Copper Sulfate Test*—This method is recommended for the detection of metallic iron or iron oxide on the surface of austenitic 200 and 300 Series, the precipitation hardening alloys, and the ferritic 400 Series stainless steels containing 16% chromium or more. It is not recommended for the martensitic and lower chromium ferritic stainless steels of the 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—**This test must not be applied to surfaces of items to be used in food processing.) The test solution is prepared by first adding sulfuric acid to distilled water and then dissolving copper sulfate in the following proportions (**Warning—** Always add acid to cold water.):

	250-cm^3 Batch
Distilled water	
Sulfuric acid (H_2SO_4, sp gr 1.84)	1 cm^3
Copper sulfate ($CuSO_4 \cdot 5H_2O$)	4 g

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Swab the surface to be inspected with test solution, applying additional solution if needed to keep the surface wet for a period of 6 min. The specimen shall be rinsed and dried in a manner not to remove any deposited copper. Copper deposit will indicate the presence of free iron.

NOTE 3—The copper sulfate test as set forth above is not applicable to surgical and dental instruments made of hardened martensitic stainless steels. Instead, a specialized copper sulfate test is extensively used for the purpose of detecting free iron and determining overall good manufacturing practice. Copper deposits at the surface of such instruments are wiped with moderate vigor to determine if the copper is adherent or nonadherent. Instruments with nonadherent copper are considered acceptable. The specialized test solution is prepared by first adding 5.4 cm^3 of sulfuric acid (H_2SO_4, sp gr 1.84) to 90 cm^3 of distilled water and then dissolving 4 g of copper sulfate ($CuSO_4 \cdot 5H_2O$).

7.3 *Precision Inspection*:

7.3.1 *Solvent-Ring Test* is a test to reveal the presence of tightly adherent transparent films that may not be revealed by visual inspection or wipe tests. A comparison standard is prepared by placing on a clean quartz microscope slide a single drop of high-purity solvent and allowing it to evaporate. Next place another drop on the surface to be evaluated, stir briefly, and transfer, using a clean capillary or glass rod, to a clean quartz microscope slide and allow the drop to evaporate. Make as many test slides as necessary to give a reasonable sample of the surface being examined. If foreign material has been dissolved by the solvent, a distinct ring will be formed on the outer edge of the drop as it evaporates. The nature of the contaminant can be determined by infrared analysis, comparing the infrared analysis with that of the standard.

7.3.2 *Black Light Inspection* is a test suitable for the detection of certain oil films and other transparent films that are not detectable under white light. In an area that is blacked out to white light, inspect all visible accessible surfaces with the aid of a new, flood-type, ultraviolet lamp. For inaccessible areas, use a wipe test as described in 7.2.2 and subject the used cloth or plug to ultraviolet lamp inspection in a blacked-out area. Fluorescence of the surface, cloth, or plug indicates the presence of contaminants. The nature of the contamination can be determined by subjecting a sample of the contaminant, that has been transferred to a clean quartz microscope slide, to infrared analysis. The test will not detect straight-chain hydrocarbons such as mineral oils.

7.3.3 *Atomizer Test* is a test for the presence of hydrophobic films. It is applicable to both small and large surfaces that are accessible for direct visual examination, and is about 100× more sensitive than the water-break test. The test procedure and interpretation of results are described in Test Method F 21. High-purity water should be used for the test.

7.3.4 *Ferroxyl Test for Free Iron* is a highly sensitive test and should be used only when even traces of free iron or iron oxide might be objectionable. It should be made only by personnel familiar with its limitations. The test can be used on stainless steel to detect iron contamination, including iron-tool marks, residual-iron salts from pickling solutions, iron dust, iron deposits in welds, embedded iron or iron oxide, etc. The test solution is prepared by first adding nitric acid to distilled water and then adding potassium ferricyanide, in the following proportions:

Distilled water	94 weight %	1000 cm^3	1 gal
Nitric acid (60–67 %)	3 weight %	20 cm^3	⅕ pt
Potassium ferricyanide	3 weight %	30 g	4 oz

Apply solution with an aluminum, plastic, glass, or rubber atomizer having no iron or steel parts, or a swab (atomizer spray is preferred).

7.3.4.1 The appearance of a blue stain (within 15 s of application) is evidence of surface iron contamination (several minutes may be required for detection of oxide scale). The solution should be removed from the surface as quickly as possible after testing using water or, if necessary, white vinegar or a solution of 5 to 20 weight % acetic acid and scrubbing with a fiber brush. Flush the surface with water several times after use of vinegar or acetic acid.[6]

NOTE 4—Potassium ferricyanide is not a dangerous poison as are the simple cyanides. However, when heated to decomposition or in contact with concentrated acid, it emits highly toxic cyanide fumes.

NOTE 5—Rubber gloves, clothing, and face shields should be worn when applying the test solution, and inhalation of the atomized spray should be avoided.

NOTE 6—The test is not recommended for process-surfaces of equipment that will be used for processing food, beverages, pharmaceuticals, or other products for human consumption unless all traces of the test solution can be thoroughly removed.

NOTE 7—The test solution will change color on standing and must be mixed fresh prior to each use.

8. Precautions

8.1 *Minimizing Iron Contamination*—Iron contamination on stainless steel parts, components, and systems is almost always confined to the surface. If reasonable care is taken in fabrication, simple inexpensive cleaning procedures may suffice for its removal, and very little special cleaning should be required. Fabrication should be confined to an area where only the one grade of material is being worked. Powder cutting should be minimized or prohibited. Handling equipment such as slings, hooks, and lift-truck forks should be protected with clean wood, cloth, or plastic buffers to reduce contact with the iron surfaces. Walking on corrosion-resistant alloy surfaces should be avoided; where unavoidable, personnel should wear clean shoe covers each time they enter. Kraft paper, blotting paper, paperboard, flannel, vinyl-backed adhesive tape or paper, or other protective material should be laid over areas where personnel are required to walk. Shearing tables, press breaks, layout stands, and other carbon-steel work surfaces should be covered with clean kraft paper, cardboard, or blotting paper to reduce the amount of contact with the carbon steel. Hand tools, brushes, molding tools, and other tools and supplies required for fabrication should be segregated from similar items used in the fabrication of carbon steel equipment, and should be restricted to use on the one material; tools and supplies used with other materials should not be brought into the fabrication area. Tools and fixtures should be made of hardened tool steel or chrome-plated steel. Wire brushes should be stainless steel, or of an alloy composition similar to the steel being cleaned, and should not have been previously used on

[6] For further information see *Journal of Materials,* ASTM, Vol 3, No. 4, December 1968, pp. 983-995.

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other materials. Only new, washed sand, free of iron particles, and stainless steel chills and chaplets should be used for casting.

8.2 *Reuse of Cleaning and Pickling Solutions*—Cleaning and pickling agents are weakened and contaminated by materials and soil being removed from surfaces as they are cleaned. Solutions may become spent or depleted in concentration after extended use, and it is necessary to check concentrations and to replace or replenish solutions when cleaning or pickling action slows. It may be impractical or uneconomical to discard solutions after a single use, even in precision cleaning operations (that is, finish-cleaning using very high-purity solvents and carried out under clean-room and rigidly controlled environmental conditions). When solutions are re-used, care must be taken to prevent the accumulation of sludge in the bottom of cleaning tanks; the formation of oil, scums, and undissolved matter on liquid surfaces; and high concentrations of emulsified oil, metal or chemical ions, and suspended solids in the liquids. Periodic cleaning of vats and degreasing tanks, decanting, periodic bottom-drain, agitation of solutions, and similar provisions are essential to maintain the effectiveness of solutions. Care must be taken to prevent water contamination of trichloroethylene and other halogenated solvents, both while in storage and in use. Redistillation and filtering of solvents and vapor-degreasing agents are necessary before reuse. Makeup is often required to maintain concentrations and pH of cleaning solutions at effective levels. Do not overuse chemical cleaners, particularly acids and vapor-degreasing solvents; if light films or oily residues remain on the metal surfaces after use of such agents, additional scrubbing with hot water and detergent, followed by repeated rinsing with large quantities of hot water, may be necessary.

8.3 *Rinse Water*—Ordinary industrial or potable waters are usually suitable for most metal-cleaning applications. Biologically tested potable water should be used for final rinsing of food-handling, pharmaceutical, dairy, potable-water, and other sanitary equipment and systems. Rinsing and flushing of critical components and systems after finish-cleaning often requires high-purity deionized water, having strict controls on halide content, pH, resistivity, turbidity, and nonvolatile residues. Analytical methods that may be used for establishing the purity of rinse water should be demonstrated to have the sensitivity necessary to detect specified impurity levels; the analytical methods given in the *Annual Book of ASTM Standards,* Vol 03.05, are recommended for referee purposes in case of dispute. To minimize the use of costly high-purity water, preliminary rinses can often be made with somewhat lesser quality water, followed by final rinsing with the high-purity water. It is also possible in many cases to use effluent or overflow from the final rinse operation for preliminary rinsing of other items.

8.4 *Circulation of Cleaning Solutions and Rinse Water*—For restricted internal surfaces (for example, small diameter piping systems or the shell or tube side of a heat exchanger), high-velocity, turbulent flow of cleaning solutions and rinse water may be necessary to provide the scrubbing action needed for effective cleaning and rinsing. The velocity required is a function of the degree of cleanness required and the size of particles that are permissible in the system after the start of operation. For example, if particles between 500 and 1000 µm are acceptable to remain, a mean flushing velocity of 1 to 2 ft/s (0.3 to 0.6 m/s) may be sufficient for pipe diameters of 2 in. and smaller; to remove 100 to 200-µm particles, a mean flushing velocity of 3 to 4 ft/s (0.9 to 1.2 m/s) may be required.

8.5 *Protection of Cleaned Surfaces*—Measures to protect cleaned surfaces should be taken as soon as final cleaning is completed, and should be maintained during all subsequent fabrication, shipping, inspection, storage, and installation.

8.5.1 Do not remove wrappings and seals from incoming materials and components until they are at the use site, ready to be used or installed. If wrappings and seals must be disturbed for receiving inspection, do not damage them, remove no more than necessary to carry out the inspection, and rewrap and reseal as soon as the inspection is complete. For critical items that were cleaned by the supplier, and that will not be given further cleaning at the use site or after installation, the condition of seals and wrappings should be inspected regularly and at fairly short intervals while the item is in storage.

8.5.2 Finish-cleaned materials and components should not be stored directly on the ground or floor, and should not be permitted, insofar as practicable, to come in contact with asphalt, galvanized or carbon steel, mercury, zinc, lead, brass, low-melting point metals, or alloys or compounds of such materials. Acid cleaning of surfaces that have been in contact with such materials may be necessary to prevent failure of the item when subsequently heated. The use of carbon or galvanized steel wire for bundling and galvanized steel identification tags should be avoided.

8.5.3 Store materials and equipment, when in process, on wood skids or pallets or on metal surfaces that have been protected to prevent direct contact with stainless steel surfaces. Keep openings of hollow items (pipe, tubing, valves, tanks, pumps, pressure vessels, and so forth) capped or sealed at all times except when they must be open to do work on the item, using polyethylene, nylon, TFE-fluorocarbon plastic, or stainless steel caps, plugs, or seals. Where cleanness of exterior surfaces is important, keep the item wrapped with clear polyethylene or TFE-fluorocarbon plastic sheet at all times except when it is actually being worked on. Avoid asphalt-containing materials. Canvas, adhesive paper or plastics such as poly(vinyl chloride) may decompose in time to form corrosive substances, for example, when exposed to sunlight or ultraviolet light. The reuse of caps, plugs, or packaging materials should be avoided unless they have been cleaned prior to reuse.

8.5.4 Clean stainless steel wire brushes and hand tools before reuse on corrosion-resistant materials; if they have not been cleaned and if they could have been used on electrolytically different materials, the surfaces contacted by the tools should be acid-cleaned. The use of soft-face hammers or terne (lead)-coated, galvanized, or unprotected carbon steel tables, jigs, racks, slings, or fixtures should be avoided (see 8.5.2).

8.5.5 If close control of particulate contamination is required, particularly of internal surfaces, the latter stages of assembly and fabrication may have to be carried out in a clean room. For most large items an air cleanliness class (see Fed.

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Std. 209e) at the work surface of Class 50 000 to 100 000 (that is, a maximum of from 50 000 to 100 000 particles 0.5 µm or larger suspended in the air) is probably sufficient.

NOTE 8—Clean room is a specially constructed enclosure in which intake air is filtered so that the air at a work station contains no more than a specified number of particles of a specified size; special personnel and housekeeping procedures are required to maintain cleanness levels in a clean room (see Fed. Std. 209e).

8.5.6 Workmen handling finished cleaned surfaces of critical items should wear clean lint-free cotton, nylon or dacron cloth or polyethylene film gloves. Rubber or plastic gloves are suitable during precleaning operations or cleaning of noncritical surfaces.

8.5.7 Installed piping systems are often *laid up wet;* that is, they are filled with water (or process fluid) after in-place cleaning until ready to be placed in service. Storage water should be of the same quality as the makeup water for the system, and should be introduced in a manner that it directly replaces the final flush water without permitting the internal surfaces of the system to dry.

8.5.8 Equipment and assemblies for critical applications may be stored and shipped with pressurized, dry, filtered, oil-free nitrogen to prevent corrosion until they are ready to be installed. Means must be provided for maintaining and monitoring the gas pressure during shipping and storage. If the item is to be shipped to or through mountains or other areas where the altitude varies greatly from that where it was pressurized, consideration must be given to the effect of that change in altitude on the pressure inside the item, and possible rupture or loss of seals.

8.5.9 Pressure-sensitive tape is often used for sealing or protective covers, seals, caps, plugs, and wrappings. If possible, the gummed surface of the tape should not come in contact with stainless steel surfaces. If tape has come in contact with the metal, clean it with solvent or hot water, and vigorous scrubbing.

8.5.10 Protective adhesive papers or plastics are often used to protect the finish of sheet stock and parts. These materials may harden or deteriorate when subjected to pressure or sunlight, and damage the surface. These materials may also decompose in time to form substances as described in 8.5.3. Protective material should be removed when its function is complete or its condition monitored for decomposition or deterioration until it is removed.

8.6 *Safety*—Cleaning operations often present numerous hazards to both personnel and facilities. Manufactures' Safety Data Sheets (MSDS) should be consulted to determine the hazards of handling specific chemicals.

8.6.1 Precautions must be taken to protect personnel, equipment, and facilities. This includes provisions for venting of explosive or toxic reaction-product gases, safe disposal of used solutions, provision of barriers and warning signs, provisions for safe transfer of dangerous chemicals, and maintenance of constant vigilance for hazards and leaks during the cleaning operation.

8.6.2 The physical capability of the item or system to be cleaned, together with its foundations, to withstand the loads produced by the additional weight of fluids used in the cleaning operation, must be established before the start of cleaning operations.

8.6.3 Insofar as possible, chemicals having explosive, toxic, or obnoxious fumes should be handled out of doors.

8.6.4 The area in which the cleaning operation is being conducted should be kept clean and free of debris at all times, and should be cleaned upon completion of the operation.

8.7 *Disposal of Used Solutions and Water*—Federal, state, and local safety and water pollution control regulations should be consulted, particularly when large volumes of chemical solutions must be disposed of. Controlled release of large volumes of rinse water may be necessary to avoid damaging sewers or stream beds.

9. Keywords

9.1 austenitic stainless steels; cleaning; corrosion; corrosive service applications; descaling; ferritic stainless steels; martensitic stainless steels; pickling; stainless steels

ANNEXES

(Mandatory Information)

A1. RECOMMENDATIONS AND PRECAUTIONS FOR ACID DESCALING (PICKLING) OF STAINLESS STEEL (See Table A1.1.)

A1.1 Where size and shape permit, immersion in the acid solution is preferred; when immersion is not practicable, one of the following room-temperature methods may be used:

A1.1.1 For interior surfaces, partially fill item with solution and rock, rotate, or circulate so that all inside surfaces are thoroughly wetted. Keep surfaces in contact with acid solution until inspection shows that scale is completely removed. Additional exposure without agitation may be needed. Treat exterior surfaces in accordance with A1.1.2.

A1.1.2 Surfaces that cannot be pickled by filling the item may be descaled by swabbing or spraying with acid solution for about 30 min, or until inspection shows that scale is completely removed.

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TABLE A1.1 Acid Descaling (Pickling) of Stainless Steel

Alloy[A]	Condition[B]	Treatment			
		Code	Solution, Volume, %[C]	Temperature °F (°C)	Time, Minutes
200, 300, and 400 Series, precipitation hardening, and maraging alloys (except free-machining alloys)	fully annealed only	A	H_2SO_4, 8–11 %[D] Follow by treatment D or F, Annex A2, as appropriate	150–180 (66–82)	5–45 max[E]
200 and 300 Series; 400 Series containing Cr 16 % or more; precipitation-hardening alloys (except free-machining alloys)	fully annealed only	B	HNO_3, 15–25 % plus HF, 1–8 %[F,G]	70–140 max (21–60)	5–30[E]
All free-machining alloys and 400 Series containing less than Cr 16 %	fully annealed only	C	HNO_3, 10–15 % plus HF, ½–1½ %[F,G]	70 (up to 140 with caution)	5–30[E]

[A] This table is also applicable to the cast grades equivalent to the families of wrought materials listed.
[B] Other heat treatments may be acceptable if proven by experience: see 5.2.1, A2.4, and A2.5 for further information.
[C] Solution prepared from reagents of following weight %: H_2SO_4, 98; HNO_3, 67; HF, 70.
[D] Tight scale may be removed by a dip in this solution for a few minutes followed by water rinse and nitric-hydrofluoric acid treatment as noted.
[E] Minimum contact times necessary to obtain the desired surface should be used in order to prevent over-pickling. Tests should be made to establish correct procedures for specific applications.
[F] For reasons of convenience and handling safety, commercial formulations containing fluoride salts may be found useful in place of HF for preparing nitric-hydrofluoric acid solutions.
[G] After pickling and water rinsing, an aqueous caustic permanganate solution containing NaOH, 10 weight % and $KMnO_4$, 4 weight %, 160 to 180°F (71 to 82°C), 5 to 60 min, may be used as a final dip for removal of smut, followed by thorough water rinsing and drying.

A1.2 Severe pitting may result from prolonged exposure to certain acid solutions if the solution becomes depleted or if the concentration of metallic salts becomes too high as a result of prolonged use of the solution; the concentration of iron should not exceed 5 weight %; take care to prevent over-pickling.

A1.3 Nitric-hydrofluoric acid solutions may intergranularly corrode certain alloys if they have been sensitized by improper heat treatment or by welding. Crevices resulting from intergranular attack can collect and concentrate halogens under service conditions or during cleaning or processing with certain chemicals; these halogens can cause stress-corrosion cracking. These alloys should generally not be acid-pickled while in the sensitized condition. Consideration should be given to stabilized or low-carbon grades if acid pickling after welding is unavoidable.

A1.4 Some latitude is permissible in adjusting acid concentrations, temperatures, and contact times. In general, lower values in this table apply to lower alloys, and higher values to higher alloys. Close control over these variables is necessary once proper values are established in order to preserve desired finishes or close dimensional tolerances, or both.

A1.5 Materials must be degreased before acid pickling and must be vigorously brushed with hot water and a bristle brush or with high-pressure water jet on completion of pickling; pH of final rinse water should be between 6 and 8 for most applications, or 6.5 to 7.5 for critical applications. To minimize staining, surfaces must not be permitted to dry between successive steps of the acid descaling and rinsing procedure. Thorough drying should follow the final water rinse.

A1.6 Hardenable 400 Series alloys, maraging alloys, and precipitation-hardening alloys in the hardened condition are subject to hydrogen embrittlement or intergranular attack by acids. Descaling by mechanical methods is recommended where possible. If acid pickling is unavoidable, parts should be heated at 250 to 300°F (121 to 149°C) for 24 h immediately following acid treatment to drive off the hydrogen and reduce the susceptibility to embrittlement.

A1.7 Proper personnel protection, including face shields, rubber gloves, and rubber protective clothing, must be provided when handling acids and other corrosive chemicals. Adequate ventilation and strict personnel-access controls must be maintained in areas where such chemicals are being used.

A2. RECOMMENDATIONS AND PRECAUTIONS FOR ACID CLEANING OF STAINLESS STEEL (See Table A2.1.)

A2.1 Treatments shown are generally adequate for removal of contamination without seriously changing surface appearance of parts. Passivated parts should exhibit a clean surface and should show no etching, pitting, or frosting. The purchaser shall specify whether a slight discoloration is acceptable. Passivated parts should not exhibit staining attributable to the presence of free iron particles imbedded in the surface when subjected to the test described in 7.2.5.1. For specific requirements for items to be used in corrosive service or where surface appearance is critical, trials should be conducted to establish satisfactory procedures.

A2.2 The high-carbon and free-machining alloys may be subject to etching or discoloration in nitric acid. This tendency can be minimized by the use of high acid concentrations with inhibitors such as $Na_2Cr_2O_7 \cdot 2H_2O$ and $CuSO_4 \cdot 5H_2O$. Oxidizing action increases with increasing concentration of nitric acid; additional oxidizing action is provided by $Na_2Cr_2O_7 \cdot 2H_2O$. Avoid acid cleaning when possible; use mechanical cleaning followed by scrubbing with hot water and

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detergent, final thorough water rinsing and drying.

A2.3 Inhibitors may not always be required to maintain bright finishes on 200 and 300 Series, maraging, and precipitation-hardening alloys.

A2.4 Hardenable 400 Series, maraging, and precipitation-hardening alloys in the hardened condition are subject to hydrogen embrittlement or intergranular attack when exposed to acids that can cause the generation of hydrogen on the item being cleaned. Cleaning by mechanical methods or other chemical methods is recommended. If acid treatment is unavoidable, parts should be heated at 250 and 300°F (121 to 149°C) for 24 h immediately following acid cleaning to drive off hydrogen and reduce susceptibility to embrittlement. The cleaning methods described in Parts II and III of Table A2.1 will not lead to the generation of hydrogen on hardenable 400 Series, maraging, and precipitation-hardening alloys in the hardened condition. Therefore, the post-cleaning thermal treatment is not required when these solutions are used for cleaning.

A2.5 Nitric-hydrofluoric acid solutions may intergranularly corrode certain alloys if they have been sensitized by improper heat treatment or by welding. Crevices resulting from intergranular attack can collect and concentrate halogens under service conditions or during cleaning or subsequent processing; these halogens can cause stress-corrosion cracking. Such alloys should not be cleaned with nitric-hydrofluoric acid solutions while in the sensitized condition. Consideration should be given to use of stabilized or low-carbon alloys if this kind of cleaning after welding is unavoidable.

A2.6 Severe pitting may result from prolonged exposure to certain acids if the solution becomes depleted or if the concentration of metallic salts becomes too high as a result of prolonged use of the solution; the concentration of iron should not exceed 2 weight %; take care to avoid overexposure.

A2.7 Nitric acid solutions are effective for removing free iron and other metallic contamination, but are not effective against scale, heavy deposits of corrosion products, temper films, or greasy or oily contaminants. Refer to Annex A1 for recommended practices where scale, heavy deposits of corrosion products, or heat-temper discoloration must be removed. Use conventional degreasing methods for removal of greasy or oil contaminants before any acid treatment.

A2.8 The citric acid-sodium nitrate treatment is the least hazardous for removal of free iron and other metallic contamination and light surface contamination. Spraying of the solution, as compared to immersion, tends to reduce cleaning time.

A2.9 Some latitude is permissible in adjusting acid concentrations, temperatures, and contact times; close control over these variables is essential once proper values have been established. Care must be taken to prevent acid depletion and buildup of metallic salt concentrations with prolonged use of solutions. In general, increasing the treatment temperature may accelerate or improve the overall cleaning action but it may also increase the risk of surface staining or damage.

A2.10 Materials must be degreased before acid treatment, and must be vigorously scrubbed with hot water and bristle brushes or with high-pressure water-jet immediately after completion of acid treatment; pH of final rinse water should be between 6 and 8 for most applications, or 6.5 to 7.5 for critical applications. To minimize staining, surfaces must not be permitted to dry between successive steps of the acid cleaning or passivation and rinsing procedure. Thorough drying should follow the final water rinse.

A2.11 Proper personnel protection, including face shields, rubber gloves, and rubber protective clothing, must be provided when handling acids and other corrosive chemicals. Adequate ventilation and strict personnel access controls must be maintained where such chemicals are being used.

A2.12 Pickling and cleaning or passivating solutions containing nitric acid will severely attack carbon steel items including the carbon steel in stainless steel-clad assemblies.

TABLE A2.1 Acid Cleaning of Stainless Steel

Alloy	Condition	Treatment			
		Code	Solution, Volume, %[A]	Temperature, °F (°C)	Time, Minutes
PART I—Cleaning with Nitric-Hydrofluoric Acid					
Purpose—For use after descaling by mechanical or other chemical methods as a further treatment to remove residual particles of scale or products of chemical action (that is, smut), and to produce a uniform "white pickled" finish.					
200 and 300 Series, 400 Series containing Cr 16 % or more, and precipitation-hardening alloys (except free-machining alloys).	fully annealed only	D	HNO_3, 6–25 % plus HF, 1/2 to 8 %[B,C]	70–140 (21–60)	as necessary
Free-machining alloys, maraging alloys, and 400 Series containing less than Cr 16 %.	fully annealed only	E	HNO_3, 10 % plus HF, 1/2 to 1 1/2 %[B,C]	70 (up to 140 with caution) (21–60)	1–2
PART II—Cleaning-Passivation with Nitric Acid Solution (see Specification A 967 for passivation specifications)					
Purpose—For removal of soluble salts, corrosion products, and free iron and other metallic contamination resulting from handling, fabrication, or exposure to contaminated atmospheres (see 6.2.11)					
200 and 300 Series, 400 Series, precipitation hardening and maraging alloys containing Cr 16 % or more (except free-machining alloys).[D]	annealed, cold-rolled, thermally hardened, or work-hardened, with dull or nonreflective surfaces	F	HNO_3 20–50 %	120–160 (49–71) 70–100 (21–38)	10–30 30–60[C]

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TABLE A2.1 *Continued*

Alloy	Condition	Treatment			
		Code	Solution, Volume, %[A]	Temperature, °F (°C)	Time, Minutes
Same[D]	annealed, cold-rolled, thermally hardened, or work-hardened with bright-machined or polished surfaces	G	HNO_3 20–40 % plus $Na_2Cr_2O_7 \cdot 2H_2O$, 2–6 weight %	120–155 (49–69) 70–100† (21–38)	10–30 30–60[C]
400 Series, maraging and precipitation-hardening alloys containing less than Cr 16 % high-carbon-straight Cr alloys (except free-machining alloys).[D]	annealed or hardened with dull or non-reflective surfaces	H	HNO_3, 20–50 %	110–130 (43–54) 70–100 (21–38)	20–30 60
Same[D]	annealed or hardened with bright machined or polished surfaces	I[E]	HNO_3 20–25 % plus $Na_2Cr_2O_7 \cdot 2H_2O$, 2–6 weight %	120–130 (49–54) 70–100 (21–38)	15–30 30–60
200, 300, and 400 Series free-machining alloys.[D]	annealed or hardened, with bright-machined or polished surfaces	J[E]	HNO_3, 20–50 % plus $Na_2Cr_2O_7 \cdot 2H_2O$, 2–6 weight %[F]	70–120 (21–49)	25–40
Same[D]	same	K[G]	HNO_3, 1–2 % plus $Na_2Cr_2O_7 \cdot 2H_2O$, 1–5, weight %	120–140 (49–60)	10
Same[D]	same	L[E]	HNO_3, 12 % plus $CuSO_4 \cdot 5H_2O$, 4 weight %	120–140 (49–60)	10
Special free-machining 400 Series alloys with more than Mn 1.25 % or more than S 0.40 %[D]	annealed or hardened with bright-machined or polished surfaces	M[E]	HNO_3, 40–60 % plus $Na_2Cr_2O_7 \cdot 2H_2O$, 2–6 weight %	120–160 (49–71)	20–30
PART III—Cleaning with Other Chemical Solutions					
	Purpose—General cleaning.				
200, 300, and 400 Series (except free-machining alloys), precipitation hardening and maraging alloys	fully annealed only	N	citric acid, 1 weight % plus, $NaNO_3$, 1 weight %	70 (21)	60
Same	same	O	ammonium citrate, 5–10 weight %	120–160 (49–71)	10–60
Assemblies of stainless and carbon steel (for example, heat exchanger with stainless steel tubes and carbon steel shell)	sensitized	P	inhibited solution of hydroxyacetic acid, 2 weight % and formic acid, 1 weight %	200 (93)	6 h
Same	same	Q	inhibited ammonia-neutralized solution of EDTA (ethylene-diamene-tetraacetic acid) followed by hot-water rinse and dip in solution of 10 ppm ammonium hydroxide plus 100 ppm hydrazine	up to 250 (121)	6 h

[A] Solution prepared from reagents of following weight %: HNO_3, 67; HF, 70.

[B] For reasons of convenience and handling safety, commercial formulations containing fluoride salts may be found useful in place of HF for preparing nitric-hydrofluoric acid solutions.

[C] After acid cleaning and water rising, a caustic permanganate solution containing NaOH, 10 weight %, and $KMnO_4$, 4 weight %, 160 to 180°F (71 to 82°C), 5 to 60 min, may be used as a final dip for removal of smut, followed by thorough water rinsing and drying.

[D] The purchaser shall have the option of specifying in his purchase documents that all 400 Series ferritic or martensitic parts receive additional treatment as follows: Within 1 h after the water rinse following the specified passivation treatment, all parts shall be immersed in an aqueous solution containing 4 to 6 weight % $Na_2Cr_2O_7 \cdot 2H_2O$, at 140 to 160°F (60 to 71°C), 30 min. This immersion shall be followed by thorough rinsing with clean water. The parts then shall be thoroughly dried.

[E] See A2.2.

[F] If flash attack (clouding of stainless steel surface) occurs, a fresh (clean) passivating solution or a higher HNO_3 concentration will usually eliminate it.

[G] Shorter times may be acceptable where established by test and agreed upon by the purchaser.

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SUMMARY OF CHANGES

Committee A01 has identified the location of selected changes to this standard since the last issue, A 380 – 99 (2005), that may impact the use of this standard. (Approved May 1, 2006.)

(*1*) Added new Section 1.5.
(*2*) Added Specification A 967 to Section 2.
(*3*) Revised Table A2.1.
(*4*) Section A2.4 was revised.

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