1 Scope

1.1 This specification defines minimum requirements for cleaning, pickling, and passivating of austenitic and austenitic-ferritic stainless steel parts, assemblies, and equipment.

2 Reference Documents

2.1 The specified otherwise. In case of any conflict between the current specification and the referred documents, following documents are referenced in this specification. All documents shall be current issue unless the current document shall take precedence.

2.2 Sulzer procedures referred to in this specification can be downloaded from www.sulzer.com/q-documents

ASTM A380	Standard Practice for Cleaning, Descaling, and Passivation of Stainless Steel Parts, Equipment, and Systems.
ASTM A967	Standard Specification for Chemical Passivation Treatments for Stainless Steel Parts.
Sulzer	
TP-0012	NDT – Visual Examination of Castings.
TP-0014	NDT – VT of welded pipework.
TP-0015	NDT – Visual examination of machined surfaces.
SQS-0006	Handling of Stainless Steel Materials, Sulzer Quality Specification.

3 Definitions

3.1 **Acid Pickling:** The chemical treatment of metallic materials with a solution containing nitric and hydrofluoric acid that results in the removal of surface oxides or scale formed during welding or heat treatment. This method also removes other foreign metals and other substances.

3.2 **Passivation:** The chemical treatment of a stainless steel alloy or iron based superalloy with a mild oxidizer, nitric acid or citric acid, to remove free iron or other contaminants embedded in the surface, for example tool steel from machining. Passivation treatment also produces an oxide film with superior corrosion resistance to the oxide film that occurs naturally. Passivation is not effective in removing oxide scale or heat tint.

3.3 **Cleaning:** 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.

4 Process

4.1 This section discusses about series of steps necessary to achieve desired pickled and passivated surface. These steps written in chronological order shall be followed by the vendor. The vendor is expected to review the below steps and align his internal process documents with this document. In case of conflict, Sulzer must be contacted with the request for clarification.

4.2 **Cleaning or Degreasing:** Components shall be free of paint, oil, grease, welding flux, slag, heat-treating and hot-forming scale, dirt, trash, metal and abrasive particles and chips, and other gross contamination. Tightly adherent scale must be removed by suitable abrasive processes.

4.2.1 **Mechanical Cleaning:** Mechanical cleaning methods include abrasive blasting, power brushing, sanding, grinding, and chipping. Mechanical methods have the advantage that they do not produce such physical or chemical conditions as intergranular attack, pitting, hydrogen embrittlement, cracks, or smut deposits.

Grinding wheels, sanding materials, stainless shot and wire brushes that are suitable for stainless steel components shall be used for mechanical cleaning. Grinding is usually the most effective means of removing localized scale resulting from welding. During grinding the generation of heat in the base material shall be kept to a minimum level in order to avoid the development of

detrimental phases. See also SQS-0006. Any abrasives used for cleaning components shall be free of iron, iron oxide, zinc, or other un-desirable materials that may cause contamination of the metal surface. Clean, previously unused abrasives, such as glass beads or iron-free silica or alumina sand or stainless steel shot shall be used for abrasive blasting. Wire brushes shall be stainless steel.

4.2.2 **Cleaning of Welds and Weld-Joint Areas:** When the joint has cooled after welding, remove all accessible weld spatter, welding flux, scale, arc strikes, etc., by grinding. Refer to SQS-0006 for detailed information. No heat tint or scale (including mild yellow marks) is allowed after grinding.

4.2.3 **Degreasing and general cleaning** may be accomplished by immersion in, swabbing with, or spraying with alkaline, emulsion, solvent, detergent, chelate, or acid cleaners or a combination of these; by vapor degreasing; by ultrasonics using various cleaners; by various mechanical methods; by steam, with or without a cleaner; or by high-pressure water-jetting.
Alkaline cleaning is the preferred method for the removal of oily and greasy contaminates. These contaminates, if present, may result in incomplete pickling of the surface. Note: Acid pickling solution is not effective in removing oils, greases and waxes. Also, the material shall be free from operator finger prints. Refer to SQS-0006 for further details. Alkaline cleaning solutions shall be prepared per the manufacturer's instructions. Proprietary cleaners may contain harmful ingredients, such as chlorides or sulphur 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.
Replace chemical cleaners, particularly vapor-degreasing solvents on a regular basis. 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 is necessary.

4.2.4 **Final Cleaning before Pickling and Passivation:** After **mechanical cleaning**, thorough rinsing with pressurized water is mandatory before the next process step. This ensures that dust and any loosely adherent scale aren't transferred to the acid bath.
If **aqueous chemical cleaning** (like alkaline cleaning) solutions are used, then the parts shall be thoroughly rinsed with clean water to remove all traces of residual chemicals.
Thorough drying after the final water rinse is required if the material components are not immediately taken to next process step (acid pickling).

4.3 **Acid Pickling**: Acid pickling shall be performed as per process parameters of Table 1 (Acid Pickling). These conditions may be altered based on material, process conditions and experience of the manufacturer.

4.3.1 Acid Pickling shall be performed only after proper heat treatment or post weld heat treatment (PWHT). Nitric-hydrofluoric acid solution removes both metallic contamination, and welding and heat-treating scales. Solutions of nitric acid alone are usually not effective for removing heavy oxide scale.

4.3.2 Uncontrolled pickling must be avoided as this may reduce the corrosion resistance of stainless steel. To prevent uncontrolled pickling, acid concentration, temperature, Fe content and component immersion time in the acid bath shall be controlled carefully.
Uncontrolled pickling may be either over pickling or under pickling. **Over pickling** occurs when acid concentrations, temperature and process time are higher than required as per Table 1. This can also be caused by repeated pickling processes. A dull surface finish rather than metallic lustre (Figure 1) is an indication of over pickling. Over-pickled components cannot be repaired and shall be scrapped. **Under pickling** is a case where oxide scales and Fe contamination are remaining after pickling and passivation. Anything which is not meeting requirements as per

Figure 1, shall be considered as under-pickled and shall be rejected. Under-pickling may be corrected by carefully going through another pickling cycle.

4.3.3 Care shall be exercised when pickling closed systems and items with crevices or internal cavities like pump casings and impellers to prevent retention of pickling solutions. To avoid this, extended rinsing with pressurized water is required.

Table 1: Acid Pickling and Passivation of Stainless Steels According to ASTM A380[3,4,5,6,7].

Treatment	Solution, Volume, % (Volume corresponds to 100% concentrated acid)	Temperature °C [°F]	Time, Minutes
Acid Pickling	HNO_3, 15–25 % plus HF, 1–8 %	20–60 [70–140]	5–30 [1]
Passivation	HNO_3, 20–45 %	50–70 [120–160]	10 [2]
		20–40 [70–100]	30 [2]

Notes:

1. Minimum contact times necessary to obtain the desired surface shall be used to prevent over-pickling. Tests shall be made to establish correct procedures for specific applications.
2. Minimum immersion time required for the material to remain in passivation bath.
3. Pickling and passivation acid solutions are weakened and contaminated by materials and soil being removed from surfaces as they are immersed in acids. It is necessary to check concentrations by appropriate titration methods. External labs may be consulted for the service.
4. **Cleaning of Pickling and Passivation Tanks:** 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 shall not exceed 5 weight % or 50 g/l (in pickling solution) and shall not exceed 2 weight % or 20 g/l (in passivation solution). If the Fe concentration exceeds the required, then pickling and passivation acid solutions shall be replaced after careful disposing (see section 6) followed by tank cleaning.
5. The solution iron concentration may be measured by test kits available to measure the Fe content with a “color reaction” (1,10-Phenanthroline). External laboratory measurements can also help in identifying the Fe content in pickling and passivation solutions.
6. Care shall be taken to prevent the accumulation of sludge in the bottom of tanks; the formation of oil, scums, and undissolved matter on the bath surface; and high concentrations of emulsified oil, dissolved material, and suspended solids in the liquids.
7. Pickling and passivating solutions containing nitric acid will severely attack carbon steel items including the carbon steel in stainless steel-clad assemblies. Thus, contact shall be avoided.

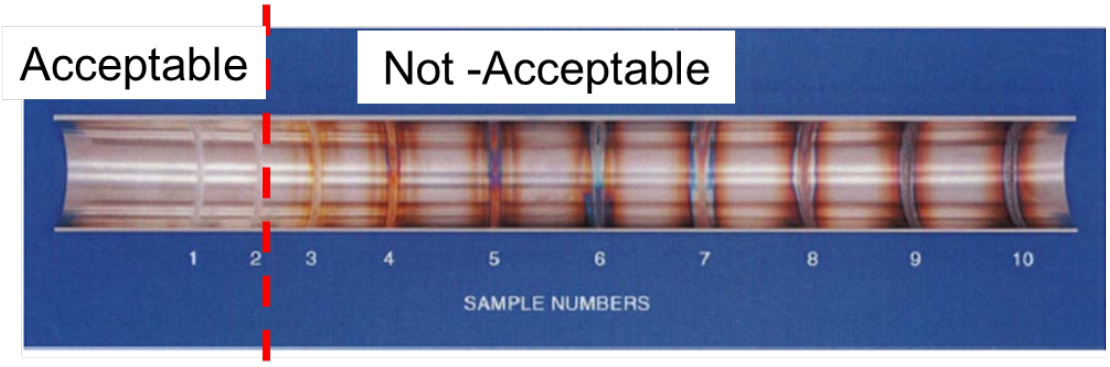


Figure 1: Heat Tint/oxide scale for stainless steel shall be from 1 to 2 in the figure. This is the minimum metallic lustre required which is acceptable.

4.3.4 After acid pickling, the components shall be rinsed using fresh water with a pH between 6.5 to 7.5 and an appropriate low conductivity equal to tap water. To minimize staining, surfaces shall not be permitted to dry between the acid pickling and the rinsing procedure. Thorough drying after the final water rinse is required if the material components are not immediately taken to the next process step.

Neutralisation (Optional): After acid pickling and water rinsing, an aqueous caustic permanganate solution containing NaOH, 10 weight % and $KMnO_4$, 4 weight %, at 70 to 80°C [160 to 180°F] for 5 to 60 min, may be used as a final dip for removal of smut, followed by thorough water rinsing. Thorough drying after the final water rinse is required if the material components are not immediately taken to next process step.

4.4 **Passivation:**

4.4.1 The material to be passivated shall be free of oil, grease, rust, scale, and other foreign matter. Passivation shall be performed preferentially as per process parameters of ASTM A380 Table 1 (Passivation). In case a passivation with nitric acid isn't possible a process according to ASTM A967 (see Appendix A) may be applied.

4.4.2 Immediately after removal from the passivating solution, the parts shall be thoroughly rinsed, using stagnant, counter current, or spray washes. The final rinse shall be carried out using fresh water with a pH between 6.5 to 7.5 and an appropriate low conductivity equal to tap water. It is recommended that final rinsing shall be carried out using de-mineralized water. To minimize staining, surfaces shall not be permitted to dry between successive steps of the passivation and rinsing procedure. Thorough drying shall follow the final water rinse except if the material components are not immediately taken for next process step (Neutralization, if applicable).

4.4.3 Neutralisation (Optional): The selection of medium and procedures for a neutralization depends of 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 70 to 80°C [160 to 180°F], followed by a complete water rinse. Thorough drying shall follow the final water rinse.

4.4.4 Finish: 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.

4.5 **Inspection and testing:**

4.5.1 Inspection and testing of the components shall be executed as stated in the respective Purchase Order (PO), Purchasing Specification (PS) or Inspection and Test Plan (ITP).

4.5.2 **Visual inspection test (VT)** is required after each of the above process steps (from section 4.2 to section 4.4) and shall be supplemented with borescopes, mirrors, and other aids, as necessary, to properly examine inaccessible or difficult-to-see surfaces. Parts shall be free of oil, slag, grease, paint and rust. In addition any heat tint indicated as colored area is not acceptable after pickling and passivation. Acceptance criteria may be explained in vendor's procedure with pictures. Please refer also to the Sulzer procedures TP-0012, 0014 and 0015 for testing requirements and acceptance limits.

4.5.3 **Ferroxyl Test for Free Iron:** As per ASTM A380 7.3.4 or ASTM A967 Practice E (14.5) to ensure that the treated surfaces are free from iron contamination. The use of commercially available Ferroxyl test kits is acceptable. In this case, the instructions as per test kit shall be followed. This test shall not be used for process-surfaces of equipment that will be used for processing food, beverages, pharmaceuticals, or other products for human consumption.

4.5.4 **Open circuit Potential Testers:** Compact and portable testing devices like Passi-Tester shall be used which can qualify the stability and thickness of the passive chromium oxide layer of stainless steel.

4.5.5 **Gross Indications of Iron:** When iron contamination is clearly visible, items shall be cleaned and passivated again in accordance with this practice and be retested by Ferroxyl test.

5 Handling

5.1 The vendor shall follow the recommendations of SQS-0006 for handling requirements in general. In addition to SQS-0006, further precautions concerning to acid pickling and passivation are discussed in this chapter.

5.2 Pickled and Passivated components shall not be stored directly on the ground or floor, and shall 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. The use of carbon or galvanized steel wire for bundling and galvanized steel identification tags shall be avoided. Pickled and Passivated components shall be lifted using contamination free stainless-steel chains and non-metallic ropes or equipment.

5.3 The vendor shall 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 pickled and passivated 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, PTFE-fluorocarbon plastic, or stainless-steel caps, plugs, or seals.

5.4 **Good Practice:** All the process control parameters and precautions, chemical solutions used, their maintained concentrations and frequencies of replacement with new chemicals, frequency of tank cleaning and measured Fe contents in acid bath, safety datasheets shall be documented and maintained by the vendor constantly. When requested, these documents shall be submitted to Sulzer Quality for review.

6 Environment Health and Safety

6.1 Safety equipment must be used by all personnel performing pickling and passivation operations along with external quality inspectors. Personal protection equipment (PPE) shall contain, and not be limited to, rubber hand gloves, PVC suit, rubber shoes, nose mask, helmet, eye goggles, along with eye fountain and body showers.

6.2 Material/ Safety Data Sheets (MSDS/SDS) shall be consulted and available at site to determine the hazards of handling specific chemicals especially while handling Hydrofluoric Acid. Disposing of used pickling and passivation chemicals shall be done after dilution, and care must be taken to ensure that their pH is controlled between 6.5 to 7.5. For handling and disposing of acids local safety, pollution and environmental regulations shall be followed.

6.3 The area in which the pickling and passivation operation is being conducted shall be kept clean and free of debris always and shall be cleaned upon completion of the operation. A Pickling and Passivation booth shall be provided with sufficient venting for escape of acid fumes and gases.

6.4 Only trained people shall be allowed to operate and handle pickling and passivation operations at site. Proper PPE and training shall be provided to all staff involved entering this area.

7 Certification

7.1 When specified in the purchase order, a report of the practice and tests used shall be supplied to the purchaser.

7.2 When specified in the purchase order, a record of process conditions used shall be supplied to the purchaser.

Appendix A: Passivation Treatments in Citric Acid Solution (as per ASTM A967)

A.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 60 to 70°C [140 to 160°F].

A.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 50 to 60°C [120 to 140°F].

A.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 20 to 50°C [70 to 120°F].

A.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

A.5 **Citric 5:** 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. Immersion bath to be controlled at a pH of 1.8 to 2.2.

A.6 **Water Rinse:** Immediately after removal from the passivating solution, the parts shall be thoroughly rinsed, using stagnant, counter current, or spray washes, singly or in combination, with or without a separate chemical treatment for neutralization of the passivation media (see Section 4.4.3 in this document), with a final rinse being carried out using demineralized water with a maximum total solids content of 200 ppm.

A.7 **Inspection and Testing:** As per section 4.5, inspection and testing shall be carried out.