

Standard Operating Procedure

Title: Cleaning, Derouging and Passivation of the Stainless Steel Parts of the Purified Water System

5.1 Passivation Treatment

Passivation is the process of forming a protective oxide film on stainless steel. The corrosion resistance of stainless steel is due to a thin chromium oxide layer on the surface of the metal. The chromium oxide layer renders the metal surface passive or resistant to corrosion. Failure to develop and maintain the chromium oxide layer results in an increase in the activity of the metal surface and can lead to corrosion in a corrosive environment. Passivation treatment is to be selected by Site Engineering Department. Nitric acid is more aggressive and is the engineering preference for passivating at ambient temperature. Phosphoric and Citric acids are preferable from a safety viewpoint.

When diluting the acid, **ALWAYS ADD ACID TO WATER. NEVER ADD WATER TO ACID.**

Treatment Type 1

Solution shall contain 25-45 (w/v) % Nitric Acid. Parts shall be exposed to this solution for 20 to 120 minutes at a temperature of 21°C to 32°C. The solution and residual effluent must be monitored closely. While very effective as a passivator, this solution may have environmental ramifications.

Treatment Type 2

The solution shall consist of 10 (w/v) % citric acid and 2 (w/v) % EDTA (ethylenediaminetetraacetic acid). Parts shall be exposed to this solution for 1 to 10 hours at a temperature of 65°C ± 5°C. EDTA is a chelating agent that keeps iron in solution over a wide pH range. Sodium Gluconate is an alternative chelating agent that could be used in place of EDTA. This solution has high reactivity with free iron, is less sensitive to exposure time, is far less corrosive to other materials, is less costly, and is considered environmentally friendly when used properly.

Treatment Type 3

Solution shall contain 20 (w/v) % Phosphoric acid. Parts shall be exposed to this solution for a minimum of 4 hours at a temperature of 65°C ± 5°C. Phosphoric acid is a weak oxidizing acid

Treatment Type 4

The solution shall consist of a commercially available pre-formulated passivating chemical such as CITRISURF 3050. The solution shall consist of 7-14 (w/v) % CITRISURF 3050. Parts shall be exposed to this solution for 1 to 4 hours at a temperature of 65°C ± 5°C.

Derouging Treatment

The phenomenon of rouging has been attributed to the destruction of the chromium oxide layer on the stainless steel surface and the formation of an iron oxide rich corrosion layer. Rouging is a known problem in pharmaceutical water systems that are generally constructed from austenitic CrNiMo steel grade AISI 316L. It is known that rouging is promoted by elevated temperatures above 60°C. Derouging can be performed using a number of different chemical formulations. Listed herein are the chemicals and derouging procedure for each formulation. Essentially

Standard Operating Procedure

Title: Cleaning, Derouging and Passivation of the Stainless Steel Parts of the Purified Water System

should be set to the maximum setting of 50Hz for all cleaning and passivation treatments. Ensure that for treatment types that require an operational temperature

above 60°C that the duration starts from the time when the solution reaches the operative temperature. The minimum high level in the tank should be 30 % (approx. 1500L) for any chemical treatment.

CAUTION: Water at or above 60°C can cause 3rd degree burns in only a few seconds. Avoid all skin contact.

5.3.14 After transferring the chemicals into the purified water storage tank, the pump and hose assembly shall be rinsed with RO water to remove any residual chemicals. This step has to be performed in between the transfer of different classes of chemicals. Example, if an alkaline was pumped first for cleaning and then an acid is required to be pump afterwards, then it is important that the pump and hose be rinsed by pumping RO water before the acid.

5.3.15 If passivation or derouging is to performed after cleaning with an alkaline treatment then the purified water system has to be rinsed after the alkaline treatment. Residual alkaline solution in the tank and pipework will reduce the working concentration of the derouging and passivation chemicals significantly due to neutralization reaction.

5.3.16 The hose should be dried with compressed air prior to storage, to prevent microbial / fungal growth.

5.4 Removal and Disposal of Cleaning Chemical

The alkaline or acid cleaning solution can be either collected in waste tote containers or neutralized and disposed into the trade waste stream. If nitric acid is used as the passivating agent then the acid must be collected in tote containers for disposal. Citric acid and phosphoric acid based chemicals can be neutralized to a pH of 7 to 10 and discharged to trade waste. Sodium Hydroxide solutions used for cleaning can be either diluted or neutralized to a pH of 7 to 10 and discharged to trade waste. Cleaning agents with high concentrations of surfactants require environmental impact assessment to determine the disposal method.

Neutralization reactions are exothermic and the exact amount of neutralizing agent can be determined through acid-base titration. If pre-formulated chemicals are used it is recommend that acid-base titration be used to determined the required amount of neutralizing solution. It is important to verify the concentration and volume of the pH Neutralizer required to neutralize the process solution, as to prevent overflow from the tank.

Option 1 Disposal of used chemical solution by an approved contractor.

The process solution can be pumped into waste totes for disposal by an approved contractor. The first rinse should also be collected in waste tote bins for disposal.

Option 2 Neutralize chemical solution in the storage tank and dump to trade waste.