CLEANING AND PASSIVATING OF STAINLESS STEEL EQUIPMENT

GENERAL CONSIDERATIONS

Stainless steel derives its corrosion resistance from a very thin layer of chromium oxide that forms at the metals surface when exposed to oxygen in the air. The chromium oxide film can form in air if the stainless steel is clean and dry. Testing indicates that maximum chromium oxide film thickness develops in three days. Further exposure to air does not yield additional corrosion protection.

Vessels delivered from equipment manufacturers generally have an oil covered inside surface. The surfaces may also have residual adhesive from the protective film that is often applied to stainless steel sheets by the manufacturer. The inside surface can also contain tramp iron that, if not removed, can lead to rusting and pitting corrosion. When polishing stainless steel to meet 3A dairy standards, some mills use an oil that contains an extreme pressure (EP) additive. The use of the EP additive yields a beautiful finish but is also very difficult to remove. Some stainless steel fabricators spray the inside of their vessels with mineral oil to prevent tramp iron from causing rust spots during transport and storage. Rust spots can lead to pitting corrosion. Mineral oil is not soluble in caustic or acidic cleaners. All manufacturing oils, EP additives and/or mineral oil, and any other soil, must be completely removed prior to passivating to prevent stains, streaks, future corrosion and most importantly to allow the passivation process to occur. For these reasons, new vessels must be thoroughly cleaned with products such as Accomplish that are specifically formulated for this task.

An oily surface cannot be passivated because oil blocks the acid and also oxygen from reaching the metal surface. The purpose of the initial caustic cleaning is to render the surface clean and oil free. A surface that “water beads” is said to be hydrophobic and therefore still contaminated with oil or other soils that must be removed. A surface that exhibits good “sheeting” is said to be hydrophilic and judged to be oil and soil free.

The use of an oxidizing acid, such as Passivation Acid, for passivation has two purposes: the acid dissolves any tramp iron and accelerates the formation of the passive chromium oxide film.

Studies have been performed which document the effectiveness of Passivation Acid on dissolving tramp metal, see results below. It is critical to the overall passivation of a stainless steel surface that all tramp metal be thoroughly removed. If it is not, it will act as an initiation site for corrosion.
Table: Effectiveness of Various Acids at Dissolving Mild Steel.

Note: 3 inch x 5 inch mild steel VW 35 D panels were immersed in solutions noted in table below for 1 hour at 140°F.

<table>
<thead>
<tr>
<th>Solution</th>
<th>% Weight Loss</th>
</tr>
</thead>
<tbody>
<tr>
<td>10% Passivation Acid</td>
<td>16.0</td>
</tr>
<tr>
<td>20% Passivation Acid</td>
<td>42.0</td>
</tr>
<tr>
<td>25% Passivation Acid</td>
<td>55.3</td>
</tr>
<tr>
<td>50% Passivation Acid (10% Nitric acid + 10% Phosphoric acid)</td>
<td>100.0</td>
</tr>
<tr>
<td>30% Ultrasil 76 (10% Nitric acid + 0.04% Phosphoric acid)</td>
<td>48.1</td>
</tr>
<tr>
<td>40% Ultrasil 76</td>
<td>71.6</td>
</tr>
<tr>
<td>50% Ultrasil 76</td>
<td>**</td>
</tr>
<tr>
<td>10% Phosphoric acid</td>
<td>3.5</td>
</tr>
<tr>
<td>20% Phosphoric acid</td>
<td>5.1</td>
</tr>
<tr>
<td>30% Phosphoric acid</td>
<td>5.4</td>
</tr>
<tr>
<td>10% Citric acid</td>
<td>0.9</td>
</tr>
<tr>
<td>20% Citric acid</td>
<td>1.0</td>
</tr>
<tr>
<td>30% Citric acid</td>
<td>1.2</td>
</tr>
</tbody>
</table>

** Excessive exothermic reaction, sample terminated.

Conclusion: Passivation Acid, a blend of nitric acid and phosphoric acid, at equivalent percent nitric acid concentrations was the most effective at dissolving mild steel. It is also less dangerous to use than higher nitric acid formulation.

John Tverberg, P.E, FASME, Metals and Materials Consulting Engineers, Dallastown, PA, gave a presentation at the 2005 IAFP annual meeting in which he discussed the use of citric acid in the passivation of stainless steel. In his presentation he stated that you could use either of the following acid steps in the pre-cleaning of stainless steel in a stainless steel passivation program:

1. 10% citric acid + 5% EDTA at 170°F for 4-6 hours, preferably >16 hours.
2. 20% nitric at 120°F for 30-60 min.

He stated that using citric acid procedure #1 would not necessarily dissolve tramp metals, more specifically large metal contaminants such as nuts, bolts and wrenches that may have been left in industrial equipment during the manufacturing whereas nitric acid can dissolve these. It is critical in the passivation process to effectively and thoroughly remove all tramp metals and other contaminants.

Citric acid and EDTA primarily chelate the metals and may help keep small metal particles suspended in solution rather than actually dissolving the metals as nitric acid does. Using citric acid for cleaning industrial equipment as part of the passivation program requires the addition of EDTA as well as high temperature and as long a contact time as possible. These additional conditions are critical to making citric acid work for pre-cleaning stainless steel as part of the total passivation program.

This is not an endorsement for the use of citric acid for passivating stainless steel as we have no documentation as to whether or not it works in our customers’ industrial equipment with the type of metal contaminants that we can encounter.
PASSIVATION PROCEDURE

I. Pre-Passivation Requirements

A. Before you do anything, and this should be standard practice before bringing any chemical into any plant, obtain water samples from all water sources used in the plant for C&S and submit them to the R&D Technical Services Laboratory for standard water analysis. Submit these water samples following the standard TSR protocol using a proper TSR form.

If the water contains >50-75 ppm chloride, be aware of the fact that there could be increased corrosion potential. If you have any questions please contact Technical Service before proceeding.

Chlorides are part of the corrosion equation. The higher the chloride concentrations present in the water the greater the potential for corrosion of equipment when in an acidic environment. If the water used to prepare an acid solution contains high levels of chlorides and the CIP is not functioning properly when applying the acid solution, such as the equipment does not drain and/or rinse thoroughly, this will result in increased contact time and thus, increased corrosion potential. Corrosion potential is a function of pH (acidic conditions), chloride concentration, contact time, temperature, equipment surface composition and condition, and the presence or absence of a passive layer. Thus, it is important to know the water chemistry and to ensure that the CIP program is operating properly before proceeding with the passivation procedure. This will help to prevent any damage to the equipment surface that could occur during the acid cleaning portion of the passivation procedure.

B. Using a bright light thoroughly inspect and examine all equipment that will be passivated. Document materials of construction, condition and visual appearance such as scratches, pitting, rust, filming, and any type of damage. Document the presence of any soil, including type and amount such as oil or adhesive residue, polishing dust, foot prints, any foreign materials, etc. Document in writing anything and everything that you observe before you begin. Perform this step with a responsible plant representative. Prepare a written report and have it signed and dated by all inspecting parties. Be specific about observations and the location of equipment where damage or soil was observed.

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C. The inside surface of a new stainless steel tank might look clean and shiny when shining a bright light into the tank, however, the surfaces could have electrostatically charged polishing dust adhering to them that you cannot see by shining a light on the surface.

To determine if there is polishing dust present you will need to wipe the surface of the equipment with a white cloth or white paper towel and look at the towel under normal room or outside light. The towel will have a black appearance under normal room or outside light if polishing dust is present. Do not look at the towel while inside of a tank or vessel and try to make a decision about whether or not polishing dust is present because you will not see it under the tank lighting.

If polishing dust is present you must remove it before proceeding to the passivation procedure. To learn more about the significance of polishing dust and how to remove it please refer to ADVISOR Bulletin under Applications/Cleaning Procedures, Black Polishing Dust Removal, C-1.50.

D. Review with the customer the instruction manual furnished by the equipment manufacturer. Take note of specific instructions for initial cleaning and/or passivating of equipment. If discrepancies exist discuss and arrive at a consensus before starting the cleaning and passivating process. Put this in writing and have the document signed and dated by a responsible plant representative. Do Not invalidate the equipment manufacturer’s warranty.
II. Preparation Procedure Instructions

A. Review the instruction manual from the equipment manufacturer. Observe specific instructions relating to initial cleaning and passivating of the equipment as for example temperature limitations imposed by choice of insulating material.

B. Review the first aid procedure and MSDS.

C. Review precautions that must be observed when handling the cleaning and passivating chemicals.

D. Examine entire system, both CIP and vessel, to make sure all components are stainless steel. Examples are: agitator bearing, fittings of CIP control instrumentation, etc. Look at everything carefully; do not assume. Remove and/or replace non-stainless parts with stainless steel ones as needed.

E. Determine CIP solution volume.

F. Before starting the cleaning and passivating operation, have a water hose running in the immediate area for emergency first aid use.

G. Circulate water and check system for leaks. Fix leaks prior to charging system with chemicals. Ensure CIP is operating properly before proceeding.
CLEANING PROCEDURE INSTRUCTIONS

SAFETY NOTE: Highly caustic product, follow proper safety procedures! Wear proper PPE, such as goggles, face shield, rubber gloves and protective clothing!

1. Charge system with 10% by volume of Accomplish, additional caustic can be added if deemed necessary.

   The product that Ecolab Food & Beverage Division recommends for the initial cleaning of new SS equipment prior to passivation is called Accomplish™. This is a unique blended caustic product with wetting agents and chelators. It is recommended to be used at 10%. If you experience problems with adhesive residue or the various oils often used on new stainless steel equipment it might be necessary to add additional caustic to the detergent solution and/or wash the equipment multiple times. The key to this step in the passivation process of new equipment is to get all of the soils completely removed from the surface and get it down to bare metal. Also, if you find polishing dust, which is statically charged and difficult to remove, you will need to do a precleaning step, as stated above, using a quaternary ammonium chloride product, QAC. The QAC, such as our product called Ster-Bac, is a cationic surfactant that helps break the static charge of the polishing dust. It is typically used at a 10% concentration and preferably manually brushed onto the surface in order to break the static charge and help lift the polishing dust from the surface. After any polishing dust is removed the Accomplish cleaning step(s) is performed.

2. Circulate for a minimum of one hour at 150°F, if within equipment manufacturer's temperature limitations.

3. Flush Accomplish solution to drain.

4. a) Execute 10 burst rinses.
   b) Test last burst rinse for absence of foaming and neutrality by determining that pH or titration matches the tap water.
   c) Execute additional burst rinses as needed to match pH of tap water and/or elimination of foam in the rinse water.

5. Allow 10 minutes drainage time and inspect for good water sheeting action. If water beading is observed, repeat cleaning procedure.

6. Do not passivate tank unless all oil and soil is removed as judged by obtaining good water sheeting action and absence of water beading. Repeat cleaning until good sheeting of the final rinse water is observed.

7. Systems that are equipped with spray balls or roto jets may require the addition of Foam Nox to control foam of Accomplish wash solution. Add 1 oz. Foam Nox per 20 gallons of Accomplish wash solution or an amount as required to control foam.
PASSIVATION INSTRUCTIONS

SAFETY NOTE: Highly acid product, follow proper safety procedures! Wear proper PPE, such as goggles, face shield, rubber gloves and protective clothing!

1. Charge system with 50% by volume with Passivation Acid (Use Passivation Acid only and never USE "E" SERIES ACIDS or AC-55-5 Red).

2. Circulate for one hour at 140°F (or maximum temperature certified by equipment manufacturer.)

3. Flush acid solution to drain.

   Caution: Before flushing the spent acid solution to the drain, inquire about the drain system materials of construction in order to make sure that the drain system is compatible with high nitric acid concentrations. If it is not compatible, adequately and safely neutralize the spent acid solution prior to flushing to the drain. If you have any questions please contact Technical Support for assistance.

4. If acid solution is dumped onto a tile floor, immediately rinse floor with water to avoid damage to grout.

5. a) Execute 10 burst rinses.
   b) Test last burst rinse for neutrality by determining that pH or titration matches the tap water.
   c) Execute additional burst rinses as needed to match pH of tap water.

6. **Allow tank to air dry. For best results allow 3 days.** This is the critical step in the production of the passive protective layer. The passive layer forms when oxygen reacts with the chromium in the stainless steel forming the more resistant chromium oxide layer.

   If compressed air is used to dry equipment it must be dry and completely free of oil that could recontaminate the surface and prevent the passive layer from forming.

7. Prior to putting tank into service, run the regular cleaning and sanitizing program.