

Solvay Chemicals

technical

P U B L I C A T I O N



H₂O₂ Passivation Procedure

Introduction

Hydrogen peroxide is a strong chemical oxidant which decomposes into water and oxygen in the presence of a catalytic quantity of any transition metal (e.g., iron, copper, nickel, etc.). The primary concern with decomposition is the build-up of pressure which can lead to pressure bursts. To prevent this from occurring, any metal surface that comes in contact with hydrogen peroxide must be degreased, pickled and passivated, even if only used once. The degreasing and pickling steps chemically clean the metal surfaces. The passivating step oxidizes the metal surface. The thin oxide coating, which forms on the metal surface during passivation, renders the surface non-reactive to hydrogen peroxide and prevents the metal from decomposing the peroxide.

The passivation procedure consists of:

1. Grinding to remove weld spatter and smooth out scratches.
2. Degreasing to remove oil and grease films.
3. Pickling to chemically clean the surface.
4. Passivating with nitric acid to form an oxide film.
5. Testing with dilute hydrogen peroxide to ensure successful treatment.

Instructions for performing each step are given below. Passivating equipment for hydrogen peroxide service is a specialized operation, and should be performed by qualified personnel only.

For additional information, call Solvay Chemicals, Inc. at 1-800-SOLVAY-C.

Note: Before beginning any work, read and comprehend the entire procedure.

Safety and environmental

Read the Solvay Chemicals "Hydrogen Peroxide Safety and Handling" brochure and the Material Safety Data Sheets for each chemical that will be used in this operation, prior to beginning work. Ensure that all recommended safety precautions are observed. The Solvay Chemicals Safety and Handling brochure and Material Safety Data Sheet for hydrogen peroxide can be found at www.solvaychemicals.us.

Prepare for passivation by roping off the work area and posting warning signs. All open lights and tools which may spark must be removed from the passivation area. Smoking is prohibited within the passivation area. Prior to preparation of the chemical solutions, determine how to dispose of the spent chemicals. These chemicals must be disposed of in a safe and environmentally sound manner that is consistent with all applicable federal, state, and local regulations.

Application methods

The chemical solutions may be applied to the metal surfaces by the four different methods listed below.

- The metal surfaces may be sprayed with the solutions.
- The metal surfaces may be coated by circulating the solutions.
- The metal surfaces may be immersed into the solutions.
- The metal surfaces may be filled with the solutions. Any method may be used, provided the surface to be treated is exposed to the solutions for the time specified in the procedure. It is usually more economical to treat small parts by immersing these parts in, or filling these parts with, the solutions. Large vessels are usually more economically treated by spraying or circulating the solutions within the vessel.

Wear the proper safety equipment when handling hydrogen peroxide.

A hard hat with a brim

Face shields
(recommended under conditions such as an open pour)

Proper chemical-splash goggles (safety spectacles with side shields will provide adequate protection only when handling small quantities of dilute hydrogen peroxide normally encountered under laboratory conditions)

Chemical-resistant protective gloves made of PVC or rubber

Standard PVC or rubber acid suit

Chemical-resistant boots of rubber or PVC



Preparation

Chemicals required.

- Water for preparing solutions and rinsing must have a chloride content <20 mg/l, pH 7-8, and be free from iron-bearing suspended matter.
- Trisodium phosphate
- Sodium metasilicate
- Caustic soda
- Nitric acid
- Hydrogen peroxide

Passivation equipment.

 Recommended gasket materials:

- P.T.F.E. (polytetra fluoroethylene)
- Fluoroelastomer

For application by spraying, use a self-lubricating stainless steel/P.T.F.E. spinner nozzle capable of wetting the entire inside surface of the vessel.

- Tanks - stainless steel or high density polyethylene
- Hoses - Goodyear Fabchem® UHMW polyethylene
- Pumps - 316 stainless steel wetted parts
- Valves - 316 stainless steel with P.T.F.E. seals and seats

Hydrogen peroxide storage vessels and associated piping should be cleaned, treated and tested separately to avoid cross-contamination.

1. Remove all foreign material from inside piping and vessels.
2. Inspect:
 - a. Entire system for adequate venting.
 - b. Entire system for proper materials of construction (stainless steel or aluminum).
 - c. All welds. Repair defects and grind smooth. Grinding equipment should be dedicated exclusively to use on the material of construction. Use aluminum oxide grinding disks.
 - d. Vessel interior. Grind out pits and inclusions.
 - e. All equipment for grease or oil films.
 - f. Block valves (All ball valves and plug valves should be vented for hydrogen peroxide service.

Details for venting both types of valves are available upon request).

3. Remove, dismantle and hand treat:
 - a. Instruments
 - b. Pressure and flow controllers
 - c. Check valves (inside parts only)
 - d. Sight or gauge glasses
 - e. Pumps (if possible, dismantle and treat)
4. Install necessary plugs, spool pieces and flanges to enable circulation of treatment chemicals.
5. Flush system with water until discharge is clean.
6. Leak check the system.
7. Repair leaks and drain system.

Degreasing

Degreasing should be performed on all new systems but may be omitted for systems which are known to be free of grease and oil.

1. See "Materials" section for preparation of degreasing solution,
2. Heat solution to 120-125 °F Maintain this temperature during treatment.
3. Apply degreasing solution for two hours.
4. Rinse with water until pH is neutral.
5. Inspect carefully. If any grease or oil film remains, repeat treatment.
6. If system was disassembled, reassemble and leak test with water.

Pickling

1. Identify the material of construction that will be treated (aluminum or stainless steel).
2. See "Materials" section for preparation of pickling solution.
3. Apply pickling solution at ambient temperature for one hour.
Note: Caustic soda is highly reactive with aluminum. Severe metal damage can occur if the concentration is too high or the contact time too long. Therefore, be certain that contact with aluminum does not exceed one hour.
4. Reclaim and store spent chemicals in labeled compatible containers.
5. Rinse with water until the pH is neutral or equal to the pH of rinse water being used.
6. For vessels only: Inspect visually to ensure that no sediment or other foreign material remains in the vessel. The vessel should be completely clean before proceeding.

Passivation

1. See "Materials" section for preparation of passivating solution.
2. Apply passivation solution at ambient temperature for two hours.
3. Reclaim chemicals in labeled compatible containers.
4. Rinse with water until pH is neutral or equal to the pH of the rinse water being used.

Note: Make sure that all equipment is completely rinsed with water to neutral pH before addition of hydrogen peroxide.

Inspection

Good lighting is necessary for the following observations.

A passivated surface shows a matte silvery finish; unpassivated surfaces are a lustrous gray.

After passivation, iron will show as a brown stain, copper as a green stain and magnesium as a black stain or spot, and tungsten inclusions in welds will sparkle in good light. All inclusions, brown stains, and green stains must be removed by grinding and the area repickled and re-passivated.

1. Visually inspect the entire vessel interior from the manway. If the surface has a uniform silver gray matte finish, go to step 4. If the surface has black stains or does not appear to be properly passivated, go to step 2.
2. Enter the vessel, making sure to follow all safety procedures and requirements. Extreme cleanliness is essential to prevent contamination. Avoid scratching the interior by wearing rubber boots wrapped with clean plastic.
3. Test questionable areas with 3-5% hydrogen peroxide. If bubbles appear, remove the cause of the bubbles and repeat the pickling and passivation steps.
4. Replace all equipment that had been removed and leak test with water.

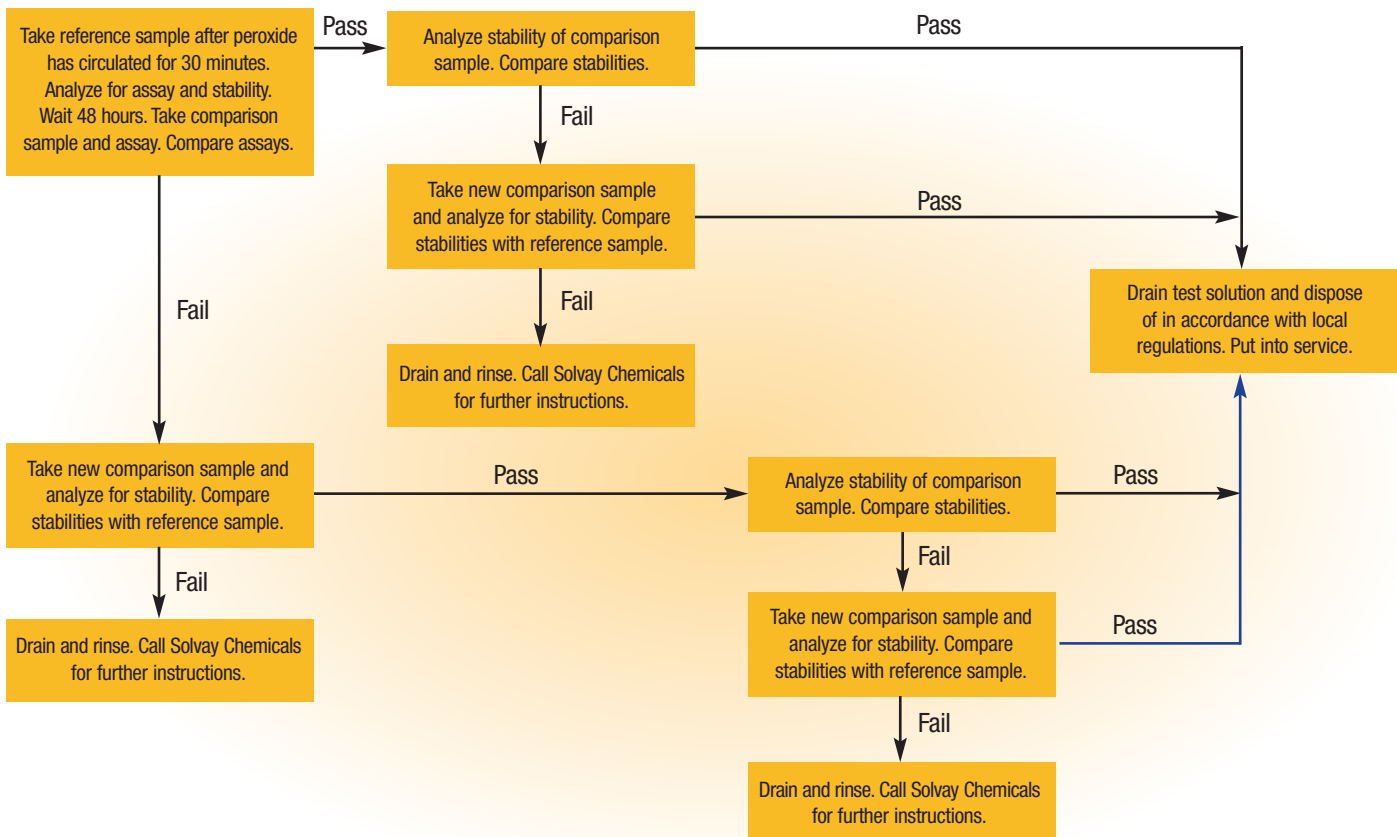
Testing

See "Materials" section for preparation of hydrogen peroxide test solution.

Piping

1. Blind off one end of spool or pipe and fill with hydrogen peroxide (H_2O_2) test solution. Position pipe so that no air is trapped, as trapped air will give a false indication of decomposition.
2. Observe for 30 minutes. Contamination will cause hydrogen peroxide to decompose and emit streams of bubbles.
3. If no bubbles are seen, drain the test solution and rinse with water. Seal all openings to avoid contamination.
4. If bubbles are seen, remove the source and repeat pickling and passivation procedures.

Figure 2: Passivation Testing Procedure



Vessels

1. Circulate and spray hydrogen peroxide test solution for 30 minutes, wetting the entire surface. Leave the test solution in the vessel until the passivation testing procedure is complete.
2. Follow the passivation testing procedure in Figure 2.

All samples should contain 500 mL of solution and should be taken using new, clean polyethylene bottles.

Definitions. Compare Assays: Analyze both samples according to Solvay Chemicals procedure “Determination of Hydrogen Peroxide Concentration (0.1%-5.0%); (XX-122).” The comparison fails if the difference between the Reference Sample and the Comparison Sample is more than 0.3%. This procedure can be found in the “Resource Library” section on our website.

Compare Stability: Analyze both samples according to Solvay Chemicals procedure “Determination of Gasometric Stability of Hydrogen Peroxide at 100°C (XX-116).” The comparison fails if the difference between the Reference Sample and the Comparison Sample exceeds 0.2 mL oxygen/minute/25 mL hydrogen peroxide. This procedure can be found in the “Resource Library” section on our website.

Materials

Volume required. Prepare enough of each solution to treat small parts by immersing or filling, plus enough to establish and maintain circulation for tanks and vessels. A volume of solution equal to about 5% of the tank’s volume is usually adequate.

Hydrogen peroxide solution should be at least 5% of the tank volume.

Degreasing solution. Prepare a degreasing solution in water using:

0.20 lbs/gal-trisodium phosphate

0.20 lbs/gal-sodium metasilicate

Pickling solution.

- a. Aluminum material - Prepare a 1% wt/wt solution of caustic soda diluted with water.
- b. Stainless steel material only - Prepare a 5% wt/wt solution of caustic soda diluted with water.
- c. Combination of aluminum and stainless steel - Prepare a 1% wt/wt solution of caustic soda diluted with water.

Table One: Specific gravity of solutions

Concentration (%)	Caustic Soda NaOH	Hydrogen Peroxide H ₂ O ₂
1	1.008	1.004
5	1.051	1.014
10	1.107	1.032
15	1.161	1.051
20	1.217	1.069
25	1.272	1.088
30	1.325	1.108
35	1.377	1.128
40	1.428	1.149
45	1.476	1.170
50	1.523	1.192
70	—	1.284

Dilution formula:

V_c = volume of concentrated solution

V_d = volume of dilute solution

V_w = volume of water to be added to concentrated solution to make dilute solution

C_c = concentration of concentrated solution

C_d = concentration of dilute solution

SG_c = specific gravity of concentrated solution (from table)

SG_d = specific gravity of dilute solution (from table)

$V_c = (V_d)(SG_d)(C_d)/(SG_c)(C_c)$

$V_w = V_d - V_c$

Examples:

Prepare 100 gallons of 1% caustic pickling solution by diluting 28% caustic soda.

V_d = 100

SG_d = 1.008 (from table)

SG_c = 1.325 (from table for 30%)

C_d = .01

C_c = .28

$V_c = (100)(1.008)(.01)/[(1.325)(.28)] = 2.72$ gallons of 28% caustic soda

$V_w = 100 - 2.7 = 97.3$ gallons of water

Prepare 200 gallons of 5% hydrogen peroxide testing solution by diluting 50% hydrogen peroxide.

$V_d = 200$

$SG_d = 1.014$

$SG_c = 1.192$

$C_d = 0.5$

$C_c = .50$

$V_c = (200) (1.014) (.05) / [(1.192)(.50)] = 17.0$ gallons of 50% hydrogen peroxide

$V_w = 200 - 17.0 = 183.0$

Passivating solution. Prepare a 30-35% wt/wt solution of nitric acid by diluting 68% nitric acid with an equal amount of water.

Testing solution. Prepare a 3-5% wt/wt hydrogen peroxide solution. Enough test solution must be prepared to completely cover the bottom welds and nozzles equal to about 5% of the vessel volume.

Note: Ensure that the chemical solutions have been properly diluted. Mixtures of strong hydrogen peroxide, nitric acid, and caustic soda may undergo violent decomposition resulting in ejection of hot solutions or pressure bursts.

Solvay Chemicals is dedicated to customer satisfaction

We strive to make your experience with Solvay Chemicals peroxides safe, efficient, and hassle-free. Most of the important product and contact information is readily available at www.solvayChemicals.us. You may also contact us by calling 1-800-SOLVAY-C (1-800-765-8292), or writing to Solvay Chemicals, Inc. at 3333 Richmond Avenue, Houston, Texas 77098.

Solvay Chemicals Quality Policy

Solvay Chemicals strives to bring you the best in products, service and technology. Exceeding, not just meeting, your expectations is the basis for our pursuit of continual improvement. To demonstrate our commitment, Solvay Chemicals' Quality Management System is registered to the ISO 9001:2000 International Quality Management System Standard.

Our registration encompasses the production and distribution of hydrogen peroxide at both of our manufacturing facilities in Deer Park, Texas and Longview, Washington, as well as administrative activities at our Houston headquarters.

Safety

Like all other powerful chemicals, hydrogen peroxide must be treated with respect and handled appropriately. For a full discussion of safe handling of this product, please see our publication "Hydrogen Peroxide Safety and Handling," available upon request, or as a download from our website at www.solvayChemicals.us.



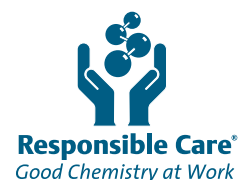
Delivery

In North America, Solvay Chemicals ships product from two plant sites and a number of strategically located distribution terminals. We operate a fleet of high-purity aluminum and stainless steel tank trucks and railcars dedicated to hydrogen peroxide service.

We also can provide stainless steel IM101 ISO containers to deliver, store, and dose liquid hydrogen peroxide. These isotainers are ideally suited to seasonal or short-term needs. In emergency situations, our Quick Response program will get isotainers of hydrogen peroxide to your site right away. For information on our excellent delivery capability, call 1-800-SOLVAY-C, or see our brochure "Isotainers for Quick Response", which is available on our website at www.solvayChemicals.us.

Responsible Care®

Recognizing the importance of preserving the environment of the planet we share, and the health and safety of the employees who produce our products, Solvay Chemicals actively supports the Responsible Care® program of the American Chemistry Council.



Solvay Chemicals, Inc.

Administration/Sales/Marketing

3333 Richmond Avenue

Houston, Texas 77098

Telephone: 713/525-6500

Fax: 713/525-7806

1-800-SOLVAY-C (800-765-8292)

Mailing Address:

P.O. Box 27328

Houston, Texas 77227

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