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# Hypo Products

## Handbook

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## Sodium Hypophosphite

## Hypophosphorous Acid

## Manganese Hypophosphite

SPE-HYPO-HB 05/00



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## Foreword

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This handbook provides basic safety and handling information to those who buy, use, and handle the three Hypo Products marketed by Occidental Chemical Corporation: Sodium Hypophosphite, Hypophosphorous Acid (Hypo Acid) and Manganese Hypophosphite. OxyChem® emphasizes caution and proper care in handling these products and invites inquiries to its Technical Service group prior to the actual use of these chemicals. Anyone using these products should be thoroughly familiar with the proper handling and storage requirements. Those looking to install a new handling system may be assisted by the information in the following pages. Those who already handle these products can use the information presented here to properly maintain equipment and to train personnel who will be responsible for working with these products. This handbook intends to present the most widely accepted and safest storage and handling practices used by the chemical industry today. However, handling procedures and problems necessarily vary with the equipment, facilities and physical plant characteristics of each location. The handler and/or user is best qualified to determine what methods are appropriate to their operation to ensure safe handling and use practices.

OxyChem can provide additional assistance to those who may require more information than is contained in this book. OxyChem maintains a staff of trained engineers and technologists at its Technical Center in Niagara Falls, New York. To obtain additional literature or assistance, contact your nearest OxyChem Sales Office.

**For 24 hour Emergency Assistance call  
800-733-3665 [or] 972-404-3228**

**OxyChem Technical Service (8 AM - 5 PM EST)  
800-733-1165 [or] 716-278-7201  
E-Mail: [TechInfo@oxy.com](mailto:TechInfo@oxy.com)**

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## **Mission Statement OxyChem Manufacturing**

In support of OxyChem's mission, the manufacturing organization will provide quality products and services of ever increasing value at the lowest possible costs to satisfy the needs of our customers.

Our objective is to provide customers with a long-term supply of competitively priced products. Based upon our vertical product integration, focus on cost effective management and application of quality principles, OxyChem's goal is to be the preferred long-term supplier to our customers.

The protection of the environment and the health and safety of our employees, customers, and the communities in which we operate or transport products through, is our highest priority. We will build trust through open communication about our operations and our commitment to Responsible Care® which enhances our position as a preferred supplier.

## **Responsible Care® A Public Commitment**

As a member of the Chemical Manufacturers Association (CMA), Occidental Chemical Corporation is committed to support a continuing effort to improve the industry's responsible management of chemicals. We pledge to manage our business according to these principles:

- To recognize and respond to community concerns about chemicals and our operations.
- To develop and produce chemicals that can be manufactured, transported, used and disposed of safely.
- To make health, safety and environmental considerations a priority in our planning for all existing and new products and processes.
- To report promptly to officials, employees, customers and the public, information on chemical related health or environmental hazards and to recommend protective measures.
- To counsel customers on the safe use, transportation and disposal of chemical products.
- To operate our plants and facilities in a manner that protects the environment and the health and safety of our employees and the public.
- To extend knowledge by conducting or supporting research on the health, safety and environmental effects of our products, processes and waste materials.
- To work with others to resolve problems created by past handling and disposal of hazardous substances.
- To participate with government and others in creating responsible laws, regulations and standards to safeguard the community, workplace and environment.
- To promote the principles and practices of Responsible Care by sharing experiences and offering assistance to others who produce, handle, use, transport and dispose of chemicals.

# Introduction

Occidental Chemical Corporation manufactures three inorganic hypophosphite based products that will be referred to in this handbook as the “**Hypo Products.**” They are shown below along with their chemical formulations and alternative names by which they may be referred.

<u>Product Name &amp; Formulation</u>	<u>Chemical &amp; Common Names</u>
<ul style="list-style-type: none"> <li>• Sodium Hypophosphite <math>\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}</math></li> </ul>	<ul style="list-style-type: none"> <li>• Phosphinic Acid, Sodium Salt, Monohydrate</li> <li>• Sodium Phosphinate Monohydrate</li> </ul>
<ul style="list-style-type: none"> <li>• Hypophosphorous Acid <math>\text{H}_3\text{PO}_2</math></li> </ul>	<ul style="list-style-type: none"> <li>• Phosphinic Acid</li> <li>• Hypophosphorous Acid</li> </ul>
<ul style="list-style-type: none"> <li>• Manganese Hypophosphite <math>\text{Mn}(\text{H}_2\text{PO}_2)_2 \cdot \text{H}_2\text{O}</math></li> </ul>	<ul style="list-style-type: none"> <li>• Phosphinic Acid, <math>\text{Mn}^{+2}</math> Salt (2:1) Monohydrate</li> <li>• Manganese Hypophosphite-1-Hydrate: metal salt</li> </ul>

In this handbook the properties, chemistry, handling and applications of these Hypo Products will be discussed. All three products are manufactured at our ISO 9002 certified facility in Niagara Falls, NY and have been commercially available for many years. Quality of product, safe on time delivery, and customer attention are the elements of product differentiation, which are emphasized by OxyChem. Good quality and high assay are important factors in reducing side reactions or undesired effects, while efficient deliveries are an essential part of a quality manufacturing operation. OxyChem technical people would be pleased to discuss the chemistry of these products and to offer suggestions concerning applications and safe handling.

# Properties and Specifications

Occidental Chemical's Sodium and Manganese Hypophosphite products are both in powder form and have little or no odor. Hypophosphorous Acid comes as a 50% solution. This solution a moderately strong monobasic acid with strong but slow reducing action and should be handled accordingly. Safety and handling instructions should be followed closely to protect people, equipment and the environment.

**Table 1**

## Summary of Hypo Product Specifications

	Sodium Hypophosphite	Hypophosphorous Acid 50%	Manganese Hypophosphite
Chemical Formula	$\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$	$\text{H}_3\text{PO}_2$	$\text{Mn}(\text{H}_2\text{PO}_2)_2 \cdot \text{H}_2\text{O}$
Appearance	-	Clear, water-white soln	Pink granular crystal or powder
Assay	98% min. <sup>†</sup> 102 - 105% <sup>††*</sup>	50.0 – 52.0% ( $\text{H}_3\text{PO}_2$ total acidity)	97% min.
Total Water	-	-	10.7% max.**
Odor	None <sup>†</sup>	-	-
Color, APHA	-	30 max.	-
Specific Gravity (25°C)	-	1.210 – 1.265	-
Arsenic (as $\text{As}_2\text{O}_3$ ), ppm	1 max. (as As)	-	10 max.
Iron (Fe), ppm	2 max. <sup>††</sup>	3 max.	35 max.
Lead (Pb), ppm	1 max. <sup>††</sup>	-	20 max.
Heavy Metals (as Pb)	2 ppm max.	-	-
Phosphate ( $\text{PO}_4$ )	Passes NFX Test <sup>†</sup>	-	0.10% max.
Phosphite (as $\text{Na}_2\text{HPO}_3$ )	0.5% max. <sup>††</sup>	-	-
Calcium (Ca)	Passes NFX Test <sup>†</sup> 50 ppm <sup>††</sup>	-	0.05% max.
Carbonate (as $\text{CO}_3$ )	-	-	Nil - NFX Test

<sup>†</sup> NFX Grade  
<sup>††</sup> Electroless Nickel Grade  
\* The product is over-dried to prevent caking. The assay calculation is based on monohydrate.  
\*\* Includes approximately 9% water of hydration.

Appropriate test procedures and Certificates of Analyses (COA) for the Hypo Products are available upon request from OxyChem's Technical Service Department.

**Table 2**  
**Typical Properties of the Hypo Products**

	Sodium Hypophosphite	Hypophosphorous Acid 50%	Manganese Hypophosphite
Chemical Formula	$\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$	$\text{H}_3\text{PO}_2$	$\text{Mn}(\text{H}_2\text{PO}_2)_2 \cdot \text{H}_2\text{O}$
Molecular Weight	106	66.00	202.93
CAS Number	10039-56-2 (monohydrate) 7681-53-0 (anhydrous salt)	6303-21-5	10043-84-2
Primary Application	Electroless Nickel Plating	Color Stabilizer or Polymerization Catalyst	Nylon fiber production
Appearance	White Crystal	Clear Liquid	Pink Crystal
Odor	Odorless	Odorless/faint acidic	Odorless
Specific Gravity @ 25°C	0.88 packed 0.78 loose	1.250	1.2 packed
Bulk Density @ 25°C, (lb/ft <sup>3</sup> ) (lb/gal)	49 - 55 N/A	76.5 10.2	75 packed N/A
Vapor Density	N/A	N/A	N/A
Melting Point (°C/°F)	N/A	-25/-13	N/A
Boiling Point (°C/°F)	N/A	108/226	N/A
Decomposition Temp (°C/°F)	>285/545	<108/226	>350/662
Dehydrates at (°C/°F)	100/212	100/212	150/302
Flash Point (COC)	None	None	None
Autoignition Temperature	None	None	None
Solubility in Water @ 25°C	100%	Infinite	12.5%
Enthalpy of Solution	~0.86 KCal/lb (endothermic)	-	-
pH	6-8 (50% soln)	0.78 (50% soln)	4.4 (1% soln)
Ionization Constant	-	$8.0 \times 10^{-2}$ (pK = 1.1)	-
Vapor Pressure, mm Hg @ 25°C @ 50°C @ 80°C	N/A	18.7 72.7 279.1	N/A
Viscosity, centipoise @ 20°C centipoise @ 60°C	N/A	8.0 5.7	N/A

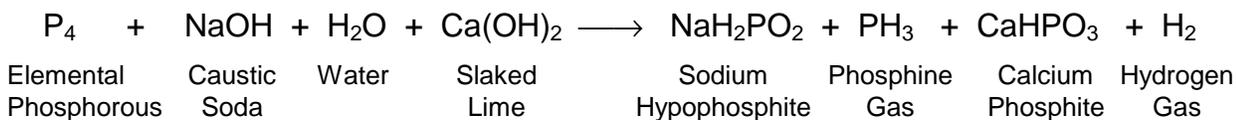
N/A = Not Applicable

# Manufacturing

The three Hypo Products are a classical case of vertical integration. Manganese Hypophosphite is produced from a reaction involving Hypophosphorous Acid, which is made from OxyChem's Sodium Hypophosphite, and this requires one of our basic chemicals, caustic soda, as a raw material.

## Sodium Hypophosphite

The production of Sodium Hypophosphite does not have a clear-cut stoichiometric balance, but involves the following basic chemistry:

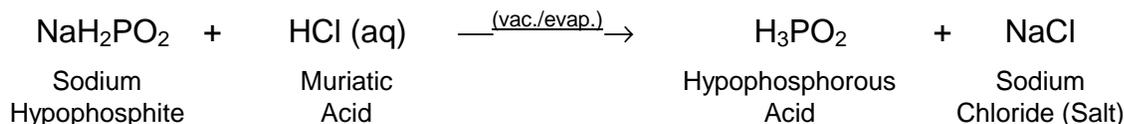


The Sodium Hypophosphite produced by this reaction is then dried and crystallized, while the phosphine gas is burned to generate Phosphoric Acid ( $\text{H}_3\text{PO}_4$ ).

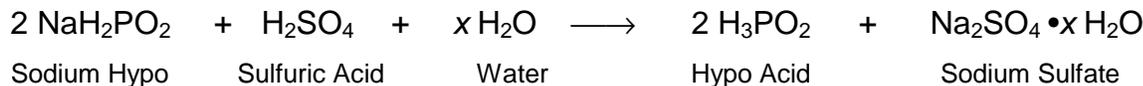
## Hypophosphorous Acid

There are three traditional technologies used for commercially produced Hypophosphorous Acid. Although there are other potential processes that involve phosphine or calcium hypophosphite and oxalic acid, they are of lesser importance due to their high cost or poor product quality. The chemistry of the three traditional routes is depicted as follows:

### 1) Hydrochloric Acid Route



### 2) Sulfuric Acid Route





# Applications

## Sodium Hypophosphite

- Electroless Nickel - A critical ingredient used in Electroless Nickel (EN) Plating solutions. In the same manner electric current supplies electrons in the electrolytic process, Sodium Hypophosphite acts as a reducing agent to supply the electrons necessary to the EN process. Phosphorous content of the EN alloy deposit is also supplied by this hypophosphite component. The EN process allows for uniform plating thickness on not only metal objects, but also plastics and ceramics.
- Chemical Intermediate - Used as a raw material in the production of other products, including Hypophosphorous Acid. It may be used in synthetic organic chemistry, especially in deamination via reduction of diazo derivatives.
- Reducing Agent - May be used as a reducing agent or antioxidant in chemical processing.
- Analytical Reagent - The NFX Grade has use as an analytical reagent.
- Polymerization Catalyst - May be used as a catalyst in some polymerization reactions.
- Polymer Stabilizer - May be used as a stabilizer to prevent degradation of polymers during extrusion or in other heated processing.
- Fire Retardant - May be used as a partial fire retardant.
- Ion Exchange Resin - May be used as a source of electrons in resin regeneration.

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## Manganese Hypophosphite

- Nylon Fibers - Critical role in the manufacture of nylon carpet fibers. Improves fiber's UV stability and dye color fastness.
  - Pharmaceuticals - Primary chemical intermediate for the production of various products.
  - Chemical Intermediate - Used in the preparation of certain linear condensation polymers.
- 
-

## Hypophosphorous Acid

- |                       |   |
|-----------------------|---|
| Bleaching Agent       | - Used as a bleaching or decolorizing agent for plastics, synthetic fibers (primarily polyester) and chemicals.   |
| Color Stabilizer      | - Used as a decolorizing agent and for color stabilization during the manufacture of chemicals and several plastics, including: nylon fibers, polyamides, polyester fiber, polyacrylonitrile, alkyd resins, epoxies, fatty acid esters, and glycerols.<br>- Also used to prevent color in copper descaling. |
| Hypophosphite Salts   | - Used in the production of Hypophosphite Salts, (i.e., Calcium, Magnesium, Manganese, Potassium, Iron and Ammonium) which are in turn used in synthetic fibers as: wetting, dispersing, emulsifying and anti-static agents.  |
| Chemical Intermediate | - Used in organic synthesis and organo phosphinic acid production.  |
| Neutralizing Agent    | - Used as a moderately strong monobasic acid.   |
| Catalyst              | - Used as a polymerization and polycondensation catalyst.   |
| Wetting Agent         | - Used as a wetting, dispersing, or emulsifying agent in electroplating.  |
| Reducing Agent        | - May be used for its strong but slow reducing action.  |
| Antioxidant           | - May be used as an antioxidant.  |
| Pharmaceutical        | - May be used as a stimulant in pharmaceuticals.  |

# Receiving Shipments

All Department of Transportation (DOT), Environmental Protection Agency (EPA) and Occupational Safety and Health Administration (OSHA) rules and regulations should be reviewed before handling the Hypo Products. All regulations must be followed and should be incorporated into your written unloading and handling procedures. State and local authorities should be contacted to ensure that a facility meets all regional requirements. All employees handling these products should receive proper training, be familiar with the appropriate Material Safety Data Sheets (MSDS) and applicable regulations.

Storage facilities must be designed to minimize human and environmental exposure to industrial chemicals. When appropriate, personal protective equipment must be used by properly trained employees. A safety shower and eye wash station should be readily available. (Also reference the "Safety Precautions" section of this manual.)

## Labeling Information

Information required on the package labels may be found in the last section of OxyChem's corresponding Material Safety Data Sheet.

## Methods of Shipment

The Hypo Products are available from OxyChem in the following package sizes:

### Sodium Hypophosphite

Fiber Drums: (non-returnable, poly-lined)

Net Wt.	Gross Wt.
* 50 lb. (23 Kg)	56 lb. (25 Kg)
† 55 lb. (25 Kg)	61 lb. (28 Kg)
110 lb. (50 Kg)	117 lb. (53 Kg)
300 lb. (136 Kg)	313 lb. (142 Kg)
400 lb. (181 Kg)	416 lb. (189 Kg)

\* NFX Grade (All others are for Electroless Nickel Grade)

† Also available in 55 lb. paper bags.

Bulk Bags: (Woven polypropylene, PE-lined)

Net Wt.	Capacity
1,100 lb. (500 Kg)	25 cu. ft.
2,000 lb. (907 Kg)	47 cu. ft.

### Hypophosphorous Acid

HDPE Plastic Drums: (non-returnable)

Net Wt.	Gross Wt.	Gallons
150 lb. (68 Kg)	158 lb. (71.7 Kg)	14.7
484 lb. (220 Kg)	507 lb. (230 Kg)	47.6

### Manganese Hypophosphite

Plastic Pails or Poly-lined Fiber Drums:

Net Wt.	Gross Wt.
50 lb. (22.7 Kg)	53.4 lb. (24.2 Kg)
100 lb. (45.4 Kg)	106.4 lb. (48.3 Kg)

All these packages are considered as one-way and non-returnable. Once empty, they should be disposed of properly and should not be re-used.

Based on the demand for these products, they are not currently available in bulk quantities.

# Hypo Products Storage & Handling

- 1) Only responsible, well-trained and well-supervised employees should be entrusted with the unloading and handling of chemicals. A worker should be present while product is being transferred and operations should proceed only during daytime or when adequate lighting is available. A written copy of handling procedures should be readily available.
- 2) The employees responsible for chemical handling should be knowledgeable of the products they handle, their potential hazards and what precautions are to be taken. They must adhere to appropriate safety practices.
- 3) Operations involving any industrial chemicals should be designed to minimize worker exposure.
- 4) All operations should be conducted with no emission of fumes, liquid, or dust. However, because of possible accidental emissions, workers responsible for product handling should exercise extreme care and wear proper safety equipment. For liquid products this should include, but is not limited to: chemical splash goggles, hard hat, face shield, NIOSH/MSHA approved respirator, acid type rainsuit, rubber gloves and rubber boots. For products in a powder form, this should include: safety glasses, hard hat, NIOSH/MSHA approved dust respirator, rubber gloves and safety shoes. Keep other personnel away from the area unless they are wearing safety equipment.
- 5) Avoid chemical contact with the body. If contacted, wash thoroughly with large quantities of cool water and consult a physician. For corrosive materials, such as Hypophosphorous Acid, a safety shower and eyewash must be readily accessible and should be tested regularly. Those located outdoors should be designed for all year service. Thermostatically controlled electric heat tracing is preferred for safety showers. Steam tracing should be avoided due to the potential danger of overheating the shower water. Visualize an escape route for yourself in the event of an incident and locate any remote shut-off valves or switches.
- 6) Spill control equipment must be available and personnel should be trained to handle both large and small spills. If a spill occurs:
  - Protect yourself from injury
  - Try to stop or minimize the spill
  - Warn others of the danger area
  - Notify emergency response personnel for proper cleanup
  - Notify appropriate authorities
- 7) Keep heat, sparks, open flames, pilot lights, and lighted cigarettes away from areas where the Hypo Products are used since toxic, corrosive and explosive gases may be formed. No smoking or flames should be permitted in any chemical work areas.
- 8) Avoid spills or splashing. Open containers carefully to avoid spurting. Clean up spills immediately and dispose of in accordance with all federal, state and local regulations.
- 9) Do not add water directly to Hypophosphorous Acid. If dilution is desired, slowly add acid to water. Avoid inadvertent mixing with alkalis such as caustic soda, or contact with metals that may cause a possible violent reaction.
- 10) Before any repairs on acid handling equipment are attempted, tanks, pipes, valves, etc., should be drained and purged with water. The hazard of a hydrogen explosion is present when cutting into empty acid lines or empty tanks. Forcing a stream of N<sub>2</sub>, CO<sub>2</sub> or other inert gas into the line may minimize this hazard.
- 11) Once the Hypo Product containers are empty, they should be cleaned out thoroughly and rendered useless. Do not reuse containers for other purpose as product residue may still remain.

## Product Storage

Store the Hypo Products in a cool, dry, fire resistant area separate from oxidizing agents and away from direct heat and sunlight. Containers should be kept tightly closed when not in use.

Appropriate material identification and warning labels should be clearly visible on storage tanks, piping, valves and containers used in connection with these materials. Storage facilities must be designed to minimize human and environmental exposure to the Hypo Products and their fumes or dust. When appropriate, personal protective equipment, safety shower and eye wash station must be readily available and used by properly trained employees.

### Sodium & Manganese Hypophosphite

Sodium and Manganese Hypophosphite are both hygroscopic and must be stored away from sources of moisture. Absorption of moisture will cause these products to clump, thus causing handling problems. In the case of Sodium Hypophosphite, OxyChem deliberately drives off some of the water of hydration from the monohydrate crystal ( $\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$ ) in order to help combat this potential problem.

In addition, these two products should not be stored near heat sources that could raise the product temperature near  $100^\circ\text{C}$  ( $212^\circ\text{F}$ ). These temperatures will cause the products to dehydrate, generating steam pressure.

Most customers use Sodium and Manganese Hypophosphite directly from the drum or bulk bag. However, if the customer wants to store these products in a bin or silo, we recommend that a dry nitrogen pad be used. It is also recommended that a vibrator(s) be installed on the discharge cone of the bin to assist in product flow.

### Hypophosphorous Acid

Handling and storage equipment for Hypophosphorous Acid service should be designed for acid resistance.

Primary or secondary containment is desirable around the storage areas to hold any spilled product. It should be designed to prevent product from entering surface or ground water in the event of a container failure. A preferred diking arrangement incorporates a leak-proof concrete dike equipped with an acid resistant liner. This arrangement will not only prevent contamination of the environment, but also allow for possible recovery of the spilled product rather than going for disposal. All dikes around liquid products should be dead-ended and isolated from sewers.

## Shelf Life

As with any product, the shelf life of the Hypo Products is partially dependent on how they are handled and stored. However, if sealed in their original containers and stored indoors in a cool, dry area, with minimal temperature fluctuations, separate from oxidizing agents (which are incompatible) and away from direct heat and sun light, these products should be expected to have a shelf life of at least a year from the ship date.

Although the Hypo Products should be chemically stable for an indefinite time, it is generally recommended that all chemical storage adhere to a FIFO (First-In, First-Out) inventory practice to avoid long term storage. If moisture gets into the Sodium and Manganese Hypophosphite containers, these products tend to clump and may eventually create handling difficulties.

Note: Do not subject containers of Hypophosphorous Acid to heat conditions which might result in concentrations greater than 50%  $\text{H}_3\text{PO}_2$  (evaporation of water). Concentrations greater than 50% can yield thermally unstable solutions.

## Hypo Acid Drum Transfers

Drum bungs should be cracked open and then unscrewed carefully to avoid damage to closures and to prevent the possible eruption of product in the rare case where a drum is under pressure. Pressure should never be used to discharge the contents of a drum. Durable fittings should be specified in designing drum emptying systems. Hard piping is recommended when feasible.

Three methods are commonly used to empty drums: gravity flow, pumping, and pulling the product into a reduced pressure tank. If the feed tank or reactor can be placed under a slight vacuum, transfer by this last method would be preferred. Here the drum contents can easily be removed by inserting a plastic dip tube into the drum and allowing the product to be sucked into the receiver. If gravity flow is to be used, lifting and tilting devices will be needed to facilitate the operation. Pumping, on the other hand, requires the investment in a reliable pump and then thorough cleaning after transfers are complete. The pump's wetted surfaces should be acid resistant.

## Materials of Construction

The information provided below has been assembled from a variety of sources. Although very limited test conditions existed in some instances, this information is intended to provide direction when determining the appropriate materials of construction (MOC) for handling the Hypo Products. However, the materials suggested below should not be considered as all inclusive and the following comments are offered only as a guideline. When designing a system to handle the Hypo Products, additional testing with specific parameters is recommended.

### Sodium & Manganese Hypophosphite

Sodium and Manganese Hypophosphite in their dry crystalline form are relatively non-corrosive. Stainless steel, glass, Teflon<sup>®</sup> and several other plastics may be used.

For solutions of these two products, low carbon 316L stainless steel or polypropylene are generally sufficient. Fiberglass reinforced plastic (FRP) - Hetron 197 has been used successfully at ambient temperatures.

Carbon steel is NOT recommended.

### Hypophosphorous Acid

Hypophosphorous Acid is a clear corrosive liquid, with a 50% solution having a pH of <1. At ambient temperatures, 316L stainless steel or glass lined carbon steel have provided good service life.

Polypropylene (PP), polyethylene (PE) and chlorinated polyvinyl chloride (CPVC) are generally the preferred plastics for use in Hypo Acid service. Polyvinyl chloride (PVC) is not recommended, favoring the above mentioned materials and due to insufficient data to accurately predict its service life.

Note: Acid concentrations and temperature, as well as the quality of construction and design, all have an effect on a product's service life.

Note: The following information came from an internal study and is intended to show the relative chemical resistance for several plastics and metals. More extensive dynamic testing is required in order to accurately determine their actual expected service life. It is also important to note that acid concentration and temperature will greatly effect the corrosion rate and durability for any material of construction.

### **Hypo Acid with Plastics**

For 15% acid at 45°C, polypropylene (PP), polyethylene (PE) and chlorinated polyvinyl chloride (CPVC) are superior to polyvinyl chloride (PVC), and are listed in order of preference.

Acrylonitrile butadiene styrene (ABS) exhibited excessive weight gain, while vinyl ester fiber reinforced plastic (VE-FRP) contaminated the product. Both of these materials should be excluded from consideration.

At a 30% acid concentration and 50°C, PP and PE offer the best chemical resistance, while CPVC appears to be suitable. PVC absorbs too much acid, which would allow permeation when under pressure and cause it to lose strength. More rapid absorption and permeation will occur at lower (vs. higher) acid concentrations. ABS also gave poor performance.

For 50% acid under boiling conditions (108°C), all the aforementioned plastics showed some distortion (and also discoloration for a few) except for VE-FRP, which appeared unaffected.

### **Hypo Acid with Metals**

At a 50% acid concentration under boiling conditions (108°C), an austenitic stainless steel (UNS #NO8367) showed no sign of attack during a 22 day study. The corrosion rate was determined to be 2.6 and 4.0 mils/year for the vapor and liquid phases, respectively.

Some degree of corrosion was observed with Nickel (N200), Monel (M400) and Stainless Steel (both 310 and 316), and they would not generally be recommended at this elevated temperature.

## **Equipment Construction**

### **Storage Tanks (Hypo Acid only)**

Passivated 316L (low carbon) stainless steel has been found to be adequate for the storage of Hypophosphorous Acid at ambient temperatures. However, the material can become active and the corrosion rate would increase at elevated temperatures or if the surface is mechanically disturbed to the extent that the bare metal surface is exposed.

High-density (high molecular weight) cross-linked polyethylene (HDPE) can also be used for vertical tank construction. Although these tanks cannot take pressure and are generally small, they are corrosion resistant. Polyethylene tanks should be inspected on a yearly basis. Note: OxyChem's Hypo Acid product drums are constructed of blow-molded HDPE.

### **Rigid Piping**

Low carbon 316L stainless steel piping is generally used for the Hypo Products at ambient temperatures (includes solutions of Sodium and Manganese Hypophosphite). Polypropylene and CPVC piping have also been used in 50% hypo acid service. Transfer lines should have as short a run as possible and be made of 2 inch flanged piping. They should be well anchored/braced and sloped at least 6 inches per 100 feet for complete drainage. Flanged pipe lends itself to easy maintenance in the event of a leak from a line or in case an in-line valve has to be replaced. Any auxiliary rigid piping under 2 inch diameter which carries liquid should be threaded and the threads taped with Teflon tape. In the event the transfer lines have to be long and/or run into a maze of other piping, machinery or buildings, they should be tagged and/or color-coded and show flow direction.

A manufacturer of fiberglass reinforced plastic (FRP) piping reports that their material is compatible with Hypo Acid, but the actual service conditions are unknown. (Reference: Fibercast Company, P.O. Box 968, Sand Springs, OK 94063, Phone: 800/331-4406 or 918/245-6651).

All piping should be visually inspected for leaks on a daily basis. All leaks should be repaired as

soon as possible. Rigid piping should have a thorough annual inspection.

### **Flexible Piping (Hoses)**

When transferring solutions of the Hypo Products, it is occasionally convenient to use some form of flexible hose. Those made of 316 stainless steel or Teflon (TFE) type lining are generally recommended.

Flexible piping should be inspected for signs of deterioration every time it is used. Pressure test each hose section at least once a year. Do not allow hoses to be stored or used in a position where the recommended bend radius is exceeded.

### **Pumps**

Small drum pumps and diaphragm pumps made of 316 stainless steel have been used for the transfer of Hypophosphorous Acid out of drums. Pumping in the process is often serviced by magnetic driven sealless or self-priming centrifugal pumps with 316 stainless steel or Teflon lined wetted parts for acid resistant. Sealed pumps may use either Teflon impregnated asbestos packing material or a mechanical seal. Crane Type 9 mechanical seals made with a 316 stainless steel cage, ceramic seat and graphite seal have been used in the past.

Probably the most serious problem with chemical pumps, particularly those in service with strong corrosive liquids like Hypo Acid, is the environmental and safety issues created by leakage from the stuffing box or mechanical shaft seal. As a result, magnetic driven sealless pumps with their dependable leak-free service have gained tremendous popularity for in-process transfers. Your specific operating conditions should be determined before consulting a pump manufacturer.

Note: Pumps should never be throttled on the suction side. Escaping fumes from pump seals will cause corrosion and become a maintenance nuisance. Therefore, all pumps should be visually examined for leaks on a daily basis and inspected more thoroughly on a yearly basis.

### **Valves**

Durco or Xomox type plugcock valves with a 316 stainless steel body and Teflon seat are generally recommended for Hypo Acid. Various other types, such as ball valves, can also be used, but less elaborate fittings are the most satisfactory.

All valves should be visually inspected for leaks on a daily basis and a detailed inspection performed at least yearly.

### **Gaskets**

Envelope type gaskets made of Teflon (TFE) are recommended for vessel closures. Flanges should use a Teflon type gasket.

### **Meters & Gauges**

Most standard meters and gauges having acid resistant construction are adequate for use with the Hypo Products. Brooks type flow meters with 316 stainless steel bodies and glass tubes have been used successfully.

### **Fume Scrubbers & Dust Collectors**

Fume scrubbers are not generally required with the Hypo Products; however, one operated on either water or a weak alkaline solution should be used as necessary.

A dust collector may be required when handling Sodium and Manganese Hypophosphite. A scrubber operating with water will also work to remove these dusts, although collecting it on a dry basis may be advantageous for disposal. Local applicable air pollution control regulations may prescribe the type and design of control systems and/or operating requirements.

## **Recommended Inspection**

Equipment should be thoroughly inspected on an annual basis or as necessary.

Whenever chemical handling equipment is being used, i.e., pumps, flanges, valves etc., it is good practice to visually inspect them for leaks. Parts of the process that are most susceptible to loss of integrity should be inspected on a prescheduled basis. Bolted connections that are subject to vibration, relaxation, or deterioration should be checked for tightness every six months or less.

## Cleaning of Empty Drums

The first step in any drum cleaning procedure is to remove as much product as possible from the drum. This is generally simple with drums or pails containing powdered crystalline products such as Sodium or Manganese Hypophosphite. In the case of liquids, however, this means the leg of the drum pump or the wand used to draw material from the drum must extend down to the bottom rim of the tilted drum. When an upended drum is emptied by gravity, 100 to 200 ml of liquid will typically remain.

Because a small amount of product may remain in "empty" drums, each container or inner liner should be rinsed until the rinse solution has minimal residue. Water is generally used for this purpose, although in the case of Hypophosphorous Acid, a sodium bicarbonate solution may be preferred.

Prior to drum cleaning, be certain that the operator wears appropriate protective clothing and a face shield. In addition, the area should be provided with good ventilation or the procedure done outdoors.

During the cleaning process, any material that cannot be used or chemically reprocessed should be disposed of in a manner meeting all government regulations. Disposal should be in a licensed landfill or treatment facility. In some cases, the waste may be neutralized on site and deemed as non-hazardous. Further assistance may be obtained by contacting an approved chemical disposal service.

Drums previously containing a corrosive liquid may be placed upside-down in an enclosure over a nozzle that protrudes into the 2 inch bung opening. A drain carries the rinse water to a storage tank for reuse or to a treatment system. Fumes collected in the enclosure are vented to a scrubber. A variation of this method is to maintain the drum in an upright position. The rinse water or sodium bicarbonate solution is then introduced into the empty drum through the ¾ inch opening via a wand equipped with a spray-nozzle. The 2 inch bung opening should be vented to a scrubber. Once filled, storing the drums for a few hours will help insure that all

product in drum crevices will be removed. If desired, additional drums could then be cleaned by pumping the rinse solution from one drum into another. In the case of Hypo Acid, this may continue until the pH of the sodium bicarbonate solution approaches neutral. After the initial alkaline rinse, each drum should be rinsed at least twice with fresh water to remove any trace material.

Collect and dispose of all rinse water in accordance with all Federal, State and Local regulations.

## Disposal of Empty Drums

Once the Hypo Product containers are empty and cleaned, they should be rendered useless. Do not reuse containers as product residue may still remain. All labeled precautions must be observed. Empty drums are normally crushed or cut up and any polyethylene liners are sliced to render them useless. They may then be disposed of as solid waste in a secure landfill or incinerated. If landfilled, make sure that the waste hauler and disposal site operator are fully informed of the operation. In addition, contact your state and local authorities to determine if more stringent regulations apply in your area. Some states require specific waste classifications and record keeping for empty containers.

The plastic Hypophosphorous Acid drums may also be recycled. There are organizations that can provide drum disposal/recycle assistance. The SPI is one such organization which may be able to identify drum disposal or reconditioning companies in your area that handle plastic drums.

Another organization is the Reusable Industrial Packaging Association. They have established a program to respond to today's strict standards of environmental responsibility. This trade group for the United States and Canada can provide a directory of reconditioners and may be reached at:

Reusable Industrial Packaging Association  
Phone: 800/533-DRUM  
Web site: <http://reusablepackaging.org/>

# Safety Precautions

## Flammability

### Unusual Fire & Explosion Hazards:

OxyChem's Hypo Products are classified as nonflammable. However, when in contact with metals, Hypophosphorous Acid can break down to liberate phosphine and flammable hydrogen gas. **DO NOT** allow the use of open flames, open lights, matches or smoking in or around areas where the Hypo Products are handled.

At high temperatures or under fire conditions, the Hypo Products will decompose to generate toxic and irritating gases, such as phosphine, oxides of phosphorous and acidic residues. Solutions of Hypophosphorous Acid may become unstable upon evaporation of water resulting in concentrations above 50% H<sub>3</sub>PO<sub>2</sub>.

### Extinguishing Media:

Use water spray, fog, foam, dry chemicals, CO<sub>2</sub> or other reagents as may be appropriate for materials in the surrounding fire. Water may be used to cool the product containers.

### Special Fire Fighting Precautions:

Use self-contained, positive pressure breathing apparatus and full protective equipment.

## Reactivity

All three Hypo Products are reducing agents and if uncontrolled, will react violently with strong oxidizing agents such as chromic acid, and even explosively with nitrates and chlorates.

The Hypo Products are not combustible but yield toxic fumes when heated to decomposition. At elevated temperatures, Sodium Hypo (>545°F) and Manganese Hypo (>662°F) can violently decompose, liberating toxic and flammable phosphine gas. They will also react in hot alkali solutions (>110°C) to form phosphine.

The maximum recommended temperature for Hypophosphorous Acid is 50°C. It should not be

exposed to heat conditions that might result in concentrations above 50% H<sub>3</sub>PO<sub>2</sub> as this can yield thermally unstable solutions.

Hypo Acid is strongly acidic. When in contact with metals, it will start to break down considerably below its boiling temperature. Phosphine and flammable hydrogen are liberated and phosphorus and phosphonic acids are formed.

## Employee Training

Safe handling of the Hypo Products depends a great deal on the effectiveness of employee education, proper training in safe practices, the use of safety equipment and good supervision. Training classes and drills for both new and veteran employees should be conducted at least annually to maintain a high degree of safety awareness in handling procedures. Employees should be informed of the potential hazards that may result from improper handling practices. They should be cautioned to prevent spills and thoroughly instructed in emergency spill response plans in the event that an incident should occur. Employee training and drills should include (but not limited to) the following:

1. The location, purpose and use of fire fighting equipment.
2. The location, purpose and use of personal protective equipment.
3. The location of safety shower and eye wash.
4. Instructions to avoid the inhalation of vapors/dust and direct contact with these products.
5. Instructions to properly report all cases of equipment failure.
6. Procedural instructions on the company's Spill Response Plan.
7. The location of appropriate Material Safety Data Sheets (MSDS). Review their content so the information is **understood**.

## Vapor Exposure Limits

At the time of this writing, exposure limits for the Hypo Products have not been established by either the ACGIH<sup>a</sup> or OSHA<sup>b</sup>. (Refer to a current OxyChem MSDS for updates.) However, work areas should be well ventilated to minimize worker exposure to potentially hazardous conditions. Provide good general room ventilation plus local exhaust at points of potential emission. Personnel handling the Hypo Products must be trained and provided with protective equipment to limit exposure. Exposure monitoring for Hypophosphorous Acid should follow NIOSH Analytical Method Number 7903 for inorganic acids. As a point of reference, the ACGIH has set a recommended TWA<sup>c</sup> exposure limit of 1 mg/m<sup>3</sup> and STEL<sup>d</sup> of 3 mg/m<sup>3</sup> for phosphoric acid.

a **ACGIH** – American Conference of Governmental Industrial Hygienists (1999), 1330 Kemper Meadow Drive, Cincinnati, OH 45240-1634

b **OSHA** – Occupational Safety and Health Administration: Refer to current Exposure Standards (29 CFR, Part 1910.1000)

c **TWA** – Time Weighted Average

d **STEL** – Short Term Exposure Limit

## Protective Equipment

- Respirator:** Use a NIOSH/MSHA approved respirator following manufacturer's recommendation where airborne contaminants may occur.
- Eye Protection:** Face shield and chemical splash-proof goggles should be worn where appropriate to protect against Hypophosphorous Acid splashing (ANSI Z87.1). Safety glasses with side shields or goggles are generally adequate when handling Sodium and Manganese Hypophosphite.
- Gloves:** Wear protective gloves such as rubber, neoprene, or vinyl.
- Other Clothing:** Impervious or chemical resistant clothing and rubber shoes or boots should be used when handling Hypophosphorous Acid. The use of coveralls is suggested for Sodium and Manganese Hypophosphite.
- Other Equipment:** Emergency shower and eyewash facilities should be readily accessible in accordance with ANSI Z358.1.

## Effects of Overexposure

### **Sodium & Manganese Hypophosphite**

- Acute:** May cause irritation of the skin, eyes, respiratory and digestive tracts. Not highly toxic if swallowed.
- Chronic:** No known effects.

### **Hypophosphorous Acid**

- Hypophosphorous Acid is a strong acid with corrosive properties. Exposure to liquid, mist or fumes may cause irritation and corrosion to the skin, eyes, respiratory and digestive tracts.
- No known chronic health effects.

## Routes of Exposure

- Eyes:** Exposure to dust from Sodium or Manganese Hypophosphite may cause moderate irritation of the eyes, while contact with Hypophosphorous Acid liquid or fumes may cause irritation or burns.
- Skin:** Exposure to Sodium or Manganese Hypophosphite may cause moderate irritation of the skin, while contact with Hypo Acid liquid or fumes may also cause burns.
- Inhalation:** Inhalation of Sodium or Manganese Hypophosphite dust may cause irritation of the respiratory tract, while inhalation of Hypophosphorous Acid mist or fumes may also cause burns.
- Ingestion:** Sodium and Manganese Hypophosphite are not highly toxic if swallowed and are excreted unchanged in the urine. They may cause irritation to the mouth and gastrointestinal tract. Hypophosphorous Acid ingestion may cause irritation and/or burns to the mouth and gastrointestinal tract.

## First Aid

- Eyes:** The object is to flush material out immediately, then seek medical attention. Immediately flush eyes with large amounts of water for at least 15 minutes, forcibly holding lids apart to ensure complete irrigation of all eye and lid tissue. For Hypophosphorous Acid, washing eyes within one minute is essential to achieve maximum effectiveness. Seek medical attention immediately thereafter.
- Skin:** Seek medical attention immediately. Flush thoroughly with cool water under shower while removing contaminated clothing and footwear. Continue to flush until medical attention arrives. Discard non-rubber shoes. Wash clothing before reuse.
- Inhalation:** Remove to fresh air as quickly as possible; if breathing is difficult, have trained person administer oxygen. If respiration stops, give mouth-to-mouth resuscitation. Get medical attention immediately.
- Ingestion:** Never give anything by mouth to an unconscious person. Give large quantities of water or if available, several glasses of milk. Seek immediate medical attention.
- If **Hypophosphorous Acid** is swallowed, **Do Not Induce Vomiting**. If vomiting occurs spontaneously, keep airway clear and give more water.
  - If **Sodium or Manganese Hypophosphite** are swallowed, **Induce Vomiting** by having patient tickle back of throat with finger. Keep airway clear.

\*\*\*\* **IMPORTANT** \*\*\*\*

Assistance should be given to anyone that has been exposed. These first aid procedures must be performed immediately to gain their full effectiveness. When significant exposure occurs, seek professional medical treatment immediately after the first aid. Refer to the proper MSDS before handling any chemical and take the appropriate safety steps.

**Seek Medical Attention Immediately for any over exposure.**

# Emergency Response Program

Due to its corrosive characteristics, users of Hypophosphorous Acid are subject to Emergency Response requirements under OSHA. Although Sodium and Manganese Hypophosphite are not subject to these same requirements, every company dealing with chemicals should develop and maintain such a written program. Each employee should be thoroughly trained in this program on a regular basis. Such a program should address, but is not limited to the following:

- SAFETY AWARENESS
  - Communication & employee training
  - Inspection of premises & equipment
  - Stock rotation
- PLANNING
  - Form a HAZMAT Team
  - Team coordination
  - Practice and revise until system works
- RELEASE (or) INCIDENT
  - Evacuate and avoid contact in all forms
  - Implement your Spill Response Plan
  - Address Post-Emergency cleanup
- EVALUATION
  - Upgrade safety program
  - Medical surveillance
- EMERGENCY ASSISTANCE

**OxyChem 24 hr Emergency customer assistance:**

**800-733-3665**

or

**972-404-3228**

**In cases of Transportation Emergencies, call CHEMTREC:**

**1-800-424-9300**

## Spill Response Plan

### General

- Keep Recovery/Over Pack drums on hand for spill cleanups or to enclose leaking drums.
- Evacuate - Keep unnecessary people away and upwind of the spill area.
- Do an evaluation from a safe position. Consider factors: quantity, location, weather.
- All responding personnel should be equipped with appropriate protective equipment. For additional information on safe handling, refer to the appropriate OxyChem MSDS.
- Once the emergency is addressed, notify proper authorities and supply complete appraisal of the situation.

### Sodium & Manganese Hypophosphite

- In the case of dry powder type products, sweep and scoop up all spilled product and other contaminated material, and place in marked disposal containers.
- If possible, clean up spill on a dry basis and then flush the area with plenty of water.
- That product which cannot be reclaimed or chemically reprocessed should be disposed of in a licensed landfill in accordance with federal, state and local regulations.

### Hypophosphorous Acid

- For a liquid, minimize the spill by shutting off pumps and valves, invert leaking drums.
- Isolate or contain spill/release in as small an area as possible. Prevent flow to sewers and streams.
- Neutralize small spills with soda ash, then transfer into marked containers for disposal in a licensed landfill or treatment facility.
- For larger spills, pump as much freestanding liquid as possible into well marked containers for future reclamation or disposal.
- Spread soda ash over spill area and wet down. Let stand for 2 hr., then flush with plenty of water.

## Reporting Requirements

(Also see the appropriate section of the Material Safety Data Sheet for additional information.)

Determine if the release should be reported:

- The three Hypo Products have not been assigned a CERCLA Reportable Quantity (RQ); however, releases may be reportable.
- Report any release of any amount if it could cause harm to people or the environment, or as State or Local regulations require.
- **It is best to report a spill if there is any uncertainty.**

When it has been decided that a release is reportable, report immediately to the National Response Center [for CERCLA], in addition to appropriate State and Local agencies as required according to SARA.

<p><b>National Response Center</b> <b>1-800-424-8802</b></p>
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Depending on specific conditions, the material resulting from cleanup operations may be classified as hazardous waste and therefore subject to specific regulations.

Package, store, transport and dispose of all cleanup materials and any contaminated equip-

ment in accordance with all applicable federal, state and local health and environmental regulations. Shipments of waste material may be subject to manifesting requirements per applicable regulations. Appropriate disposal will depend on the nature of each waste material and should be performed by competent and properly permitted contractors. Ensure that all responsible Federal, State and Local agencies receive proper notification of spill and disposal methods.

## Waste Disposal

Any material that can not be used or chemically reprocessed should be disposed of in accordance with all Federal, State and Local regulations.

The three Hypo Products are not listed as substance or source wastes under Federal RCRA Hazardous Waste Classifications. However, if disposed of, Hypophosphorous Acid would be classified as D002 hazardous waste due to its corrosive characteristic.

Although all three products would be expected to pass, they may require TCLP testing for hazardous constituents. It is important to be familiar with State and Local waste disposal regulations as they may be more stringent than the Federal RCRA requirements.

### Registered Trademarks

OxyChem® is a registered trademark of Occidental Chemical Corporation

Responsible Care® is a registered trademark of the Chemical Manufacturers Association, Inc.

Teflon® is a registered trademark of E. I. duPont de Nemours and Company