




SODIUM CHLORITE HANDBOOK

OxyChem
Technical Information
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Dallas-based Occidental Chemical Corporation is a leading North American manufacturer of basic chemicals, vinyls and performance chemicals directly and through various affiliates (collectively, OxyChem). OxyChem is also North America's largest producer of sodium chlorite.

As a Responsible Care® company, OxyChem's global commitment to safety and the environment goes well beyond compliance. OxyChem's Health, Environment and Safety philosophy is a positive motivational force for our employees, and helps create a strong culture for protecting human health and the environment. Our risk management programs and methods have been, and continue to be, recognized as some of the industry's best.

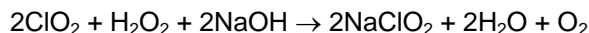
OxyChem offers an effective combination of industry expertise, experience, on line business tools, quality products and exceptional customer service. As a member of the Occidental Petroleum Corporation family, OxyChem represents a rich history of experience, top-notch business acumen, and sound, ethical business practices.

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Manufacture of Sodium Chlorite

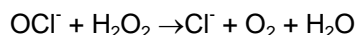
Sodium chlorite is made by the partial reduction of sodium chlorate to chlorine dioxide and the chlorine dioxide's subsequent conversion to sodium chlorite in an alkaline solution in the presence of hydrogen peroxide. An excess of both peroxide and caustic is present to ensure complete reaction as follows:



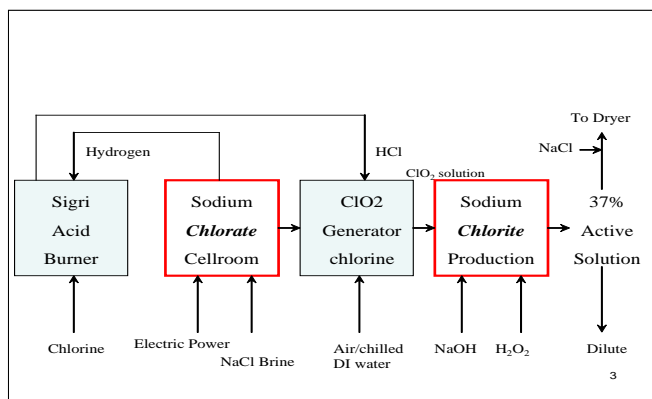
Under these conditions, ClO_2 is also known to disproportionate to chlorite and chlorate as follows:



Any chlorine present would be in the hypochlorite form because of the strong alkaline conditions. Hypochlorite will react with hydrogen peroxide to yield chloride as follows:



Because there is no source of ammonia or nitrogen in the process, chloramines will not be present as an impurity.



Sodium Chlorite Process

Sodium Chlorite End Uses

Sodium Chlorite Applications

Hydrogen Sulfide Odor Control. In the ionic form, sodium chlorite is almost exclusively reactive to hydrogen sulfide, and does not react with ammonia or form other chlorinated compounds.

Copper Cyanide Destruction. Copper Cyanide, which is formed in the recovery process for copper metal, is the only cyanide that catalyzes sodium chlorite making it an effective option.

Chlorine Dioxide Applications

Chlorine dioxide has a variety of commercial uses. In all of the following applications, sodium chlorite is used in the generation of the chlorine dioxide.

Treatment of Potable Water. Chlorine dioxide has long been used to remove tastes and odors in potable water. It is also used in the disinfection of water, particularly where trihalomethanes are of concern. And it oxidizes soluble manganese and iron compounds, eliminating a major cause of stained sinks and fixtures.

Bacterial Control in Oil Wells and Petroleum Systems. A patented use for chlorine dioxide is to treat water that is or will be contaminated with petroleum oil. Many such mixtures contain sulfite-reducing bacteria that form undesirable sulfide compounds. Chlorine dioxide oxidizes these sulfides to sulfates, while preventing or substantially retarding the formation of colloidal sulfur.

Bacterial Slime Control in Paper Mills. Some of the major operational problems in paper and paperboard production are caused by proliferation of microbiological organisms in white water and stock systems. An oxidizing biocide, chlorine dioxide can control microbiological growths, which cause paper malodors and discoloration, deterioration of felts, equipment corrosion, fouling of pipes and showers, and paper quality problems such as spots, specks and holes.

Food Processing. Chlorine Dioxide is highly effective for microbiological control in organically contaminated flume waters. Control of microbiological growths is necessary to ensure food product safety and quality. Chlorine dioxide has also found application in cherry bleaching.

Algae Control in Cooling Towers. Chlorine dioxide efficiently and economically controls microbiological growths in industrial cooling waters under conditions unfavorable to chlorine. It is the primary microbiological control agent in systems with high pH, ammonia-nitrogen contamination, or persistent slime problems.

Treatment of Wastes. Chlorine dioxide is used to disinfect sewage and plant wastes. It destroys phenolics, simple cyanides and sulfides by oxidation.

Stripping Dyestuffs from Textiles. Chlorine dioxide removes dyestuffs from textiles with a minimum of fiber degradation. However, its effectiveness depends upon the dyestuff and the type of fabric.

This method also provides a good bottom for redying.

Upgrading of Fats and Oils. Chlorine dioxide is effective in the bleaching of fats and oils. The process is simple and low cost and since it eliminates the need for a filter medium, it produces a higher yield than other methods. (About 30% of the weight of the filter residue, which is generally discarded, is tallow.) Problems such as storage and handling of the filter medium and disposal of filter residues are eliminated as well.

Sodium Chlorite Product Grades

OxyChem sodium chlorite is an excellent source of chlorine dioxide. Sodium chlorite is available in both dry and liquid forms. The dry forms contain approximately 80% Sodium chlorite and the solutions range from 7.5% to 40% sodium chlorite. Both forms are available for EPA registered and non-registered applications. Some of the chemical and physical properties of eight of Oxy's most popular products are given below.

Properties of Technical Sodium Chlorite

Sodium Chlorite, min (%)	77.5
Sodium Chloride, (%)	5-16
Appearance	white flakes
Bulk Density (lb/ft ³)	Loose 53 Packed 69
Inert Ingredients, sodium salts, and water	6.0%

Properties of Technical Sodium Chlorite Solution 50

Sodium Chlorite, min (%)	35.9
Sodium Chloride, max (%)	3.5
Water (%)	56.4 – 64.1
Density @ 35°C (lb/gal)	~11.1
Crystallization Point (°C)	~15
Appearance	slightly cloudy, pale yellow
Inert Ingredients, mixture of sodium salts.	

Properties of 31% Active Sodium Chlorite Solution

Sodium Chlorite, min (%)	30.5
Sodium Chloride, max (%)	3.5
Inert Ingredients, mixture of other sodium salts(%)	3 - 4
Water, (%)	61 – 69.5
Appearance	clear, pale yellow
Density @ 25°C (lb/gal)	10.7
Crystallization Point (°C)	3

Properties of Technical Sodium Chlorite Solution 31.25

Sodium Chlorite, min (%)	24.25
Sodium Chloride, max (%)	3.0
Inert Ingredients, mixture of other sodium salts (%)	3 - 4
Water, (%)	68 - 76
Appearance	clear, pale yellow
Density @ 25°C (lb/gal)	10.2
Crystallization Point (°C)	-7

Properties of Technical Sodium Chlorite Solution 18.75

Sodium Chlorite, min (%)	15
Sodium Chloride, max(%)	3.0
Inert Ingredients, mixture of other sodium salts(%)	3 - 4
Water, (%)	80 - 85
Appearance	clear, pale yellow
Density @ 25°C (lb/gal)	9.4
Crystallization Point (°C)	-8

Properties of Textone

Sodium Chlorite, min (%)	74
Sodium Chloride, (%)	9 - 22
Inert Ingredients, mixture of sodium salts and water balance	
Appearance	white flakes
Bulk Density (lb/ft ³)	Loose 53 Packed 69

Properties of Textone XL

Sodium Chlorite, min (%)	30.5
Sodium Chloride, max(%)	3.5
Inert Ingredients, mixture of other sodium salts(%)	3 - 4
Water, (%)	61 – 69.5
Appearance	clear, pale yellow
Density @ 25 °C (lb/gal)	10.7
Crystallization Point (°C)	3

Properties of Textone L

Sodium Chlorite, min (%)	24.25
Sodium Chloride, max(%)	3.0
Inert Ingredients, mixture of other sodium salts (%)	3 - 4
Water, (%)	70 - 76
Appearance	clear, pale yellow
Density @ 25°C (lb/gal)	10.2
Crystallization Point (°C)	-7

Safety and Handling

Shipping Information

OxyChem Technical Sodium Chlorite is registered with the EPA for a number of pesticidal applications as governed by FIFRA. Technical Sodium Chlorite is available in 100-lb drums. Technical Sodium Chlorite, Solution 50, Technical Sodium Chlorite Solution 31.25 and 31 percent Active Sodium Chlorite Solution are available in 55-gallon drums, 330 - gallon non-returnable totes, and tank trucks.

Safety and Handling

The following summary of health and safety information is not intended to be complete. For complete information, read the current Material Safety Data Sheet (MSDS). To obtain a MSDS, contact OxyChem's HESS Department or go to www.oxychem.com.

Toxicological Properties

Sodium chlorite is toxic by ingestion. Sodium chlorite may cause anemia by oral exposure and has low toxicity by dermal exposure. Dry sodium chlorite and sodium chlorite solutions have an oral LD₅₀ (rat) of 389 mg/kg. Sodium chlorite dry and solution products have a dermal LD₅₀ (rabbit) of greater than 2 g/kg. Sodium chlorite can produce severe irritation or burns to the skin and eyes. Corneal damage can occur if not washed immediately from the eyes.

Personnel Protection

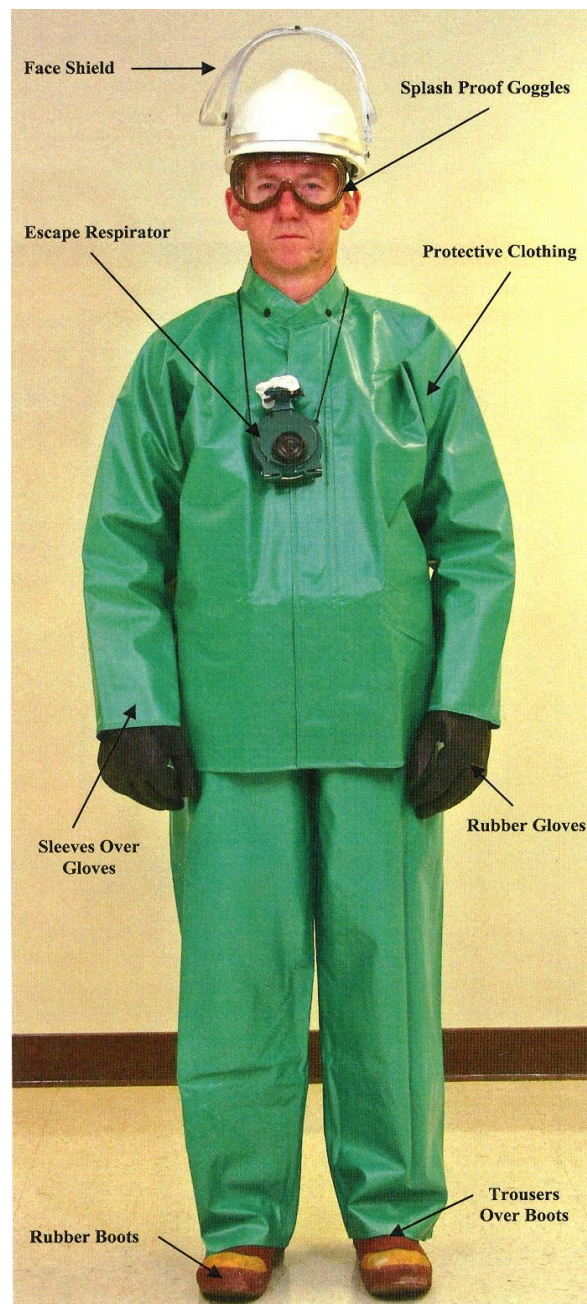
When there is a potential for contact while handling sodium chlorite solutions, chemical goggles, face shield, neoprene gloves, apron, and boots should be worn. A respiratory protection program that meets 29 CFR 1910.134 must be followed whenever workplace conditions warrant the use of a respirator.

Wear a NIOSH approved acid gas respirator with a dust/mist filter if any exposure is possible. Additionally, for dry sodium chlorite, wear a chemically impervious suit. Local exhaust is required where exposure to dust or mist might occur. If sodium chlorite is spilled on clothing, remove and wash contaminated clothing at once to avoid the potential of fire.

First Aid

Eyes: Immediately flush eyes with large amounts of water for at least 15 minutes while frequently lifting the upper and lower eyelids. Consult a physician immediately.

Skin: Remove contaminated clothing. Immediately flush exposed skin areas with large amounts of water for at least 15 minutes. Consult a physician if burning or irritation of the skin persists. Contaminated clothing must be laundered before reuse.



Personal Protective Equipment

Ingestion: DO NOT induce vomiting. Drink large quantities of water. Consult a physician immediately. DO NOT give anything by mouth if the person is unconscious or having seizures.

Inhalation: Move patient to fresh air and monitor for respiratory distress. If coughing or difficulty in breathing develops, administer oxygen, and consult a physician immediately. In the event that breathing stops, administer artificial respiration and obtain emergency medical assistance immediately.

Notes to Physician: Chlorine dioxide vapors are emitted when this product contacts acids or chlorine.

If these vapors are inhaled, monitor patient closely for delayed development of pulmonary edema which may occur up to 48-72 hours post-inhalation.

Following ingestion, neutralization and use of activated charcoal is not indicated.

Storage and Handling

Do not contaminate sodium chlorite with incompatible materials such as dirt, organic matter, oxidizers, reducing agents, chemicals, soap products, solvents, acids, paint products, or combustible materials. Do not store or transport sodium chlorite with incompatible materials. Contamination may start a chemical reaction with generation of heat and emission of chlorine dioxide (a poisonous, explosive gas). A fire or explosion may result. Rinse empty containers thoroughly with water and dispose of in accordance with label instructions.

Dry sodium chlorite. Do not expose to moisture during storage. Store in the original container, in a cool, dry, well ventilated area away from direct sunlight. Always replace cover tightly. Mix only into water using a clean, dry metal scoop reserved for this product alone.

Keep away from flame or any burning material (such as a lighted cigarette). If fire occurs, extinguish with plenty of water. Cool any unopened drums near the fire by spraying water on them.

Sodium chlorite solutions. Store in clean, closed, non-translucent containers. Exposure to sunlight or ultra-violet light will reduce product strength.

Do not allow solution to evaporate to dryness; this product becomes a fire or explosion hazard if allowed to dry and can ignite in contact with combustible materials.

Spill and Leak Procedures

In the event of a spill or leak, remove all sources of ignition. Wear NIOSH approved positive pressure, self contained breathing apparatus with a chemically impermeable, fully encapsulated suit. Follow OSHA regulations for respirator use (see 29 CFR 1910.34).

Sodium chlorite, dry, is a fire or explosion hazard if contaminated with combustible material. Clean up in a manner to avoid contamination. Spilled material should be picked up, by using a clean, dry, scoop or shovel and placed into a clean, dry, container. Do not return spilled material to the original container. Isolate the recovery container outside or in a well ventilated area and hold for proper waste disposal. Do not seal the container. Flush any residual material with large quantities of water.

Sodium chlorite solution also becomes a fire or explosion hazard if allowed to dry and can ignite on contact with combustible material. Continue to keep damp. Contain spilled material by diking or absorbing with clay, soil or non-flammable commercial absorbents. Do not return spilled material to original container. Place in a clean container and isolate outside or in a well ventilated area. Do not seal the container. Flush any residual material with large quantities of water.

Disposal

Spill residues may be a hazardous waste as defined in 40 CFR 261. The EPA hazardous waste designation for dry sodium chlorite waste would be D001 and sodium chlorite solution waste would have the waste designation of D002. As a hazardous waste, it will be subject to the Land Disposal Restrictions under 40 CFR 268 and must be managed accordingly. As a hazardous waste solution or solid, it must be disposed of in accordance with local, state, and federal regulations in a permitted hazardous waste treatment, storage and disposal facility.

All persons transporting, storing, handling and using sodium chlorite must comply with applicable federal, state and local regulations. The following summaries of some of the current federal OSHA, DOT, FDA, USDA and EPA requirements are not intended to be complete or to represent summaries of all relevant requirements. Rather, these summaries describe the areas creating most questions from involved parties. These summaries also do not touch upon any similar state or local regulations, some of which may impose additional or different obligations from those imposed by federal regulations. All parties are responsible for complete review of applicable regulations pertaining to their own operation. Contact our Technical and Environmental Services group for additional information.

OSHA Hazard Communication

The Occupational Safety and Health Administration (OSHA) requires employers to develop, implement and maintain a written hazard communication program. The written hazard communication program must be available, upon request, to employees, their designated representatives and OSHA.

This written program must cover container labeling, material safety data sheets, and employee training. It must also include a list of hazardous chemicals in each work area and explain the methods for communicating hazards to employees.

The Material Safety Data Sheet (MSDS) is the main vehicle for communicating the hazards, safe handling and emergency procedures for each hazardous chemical in the workplace. Employers are required to obtain and maintain an MSDS for each hazardous chemical which they use in the workplace.

The MSDS includes information on the physical and chemical characteristics of the chemical; acute and chronic health effects; information on exposure limits; emergency and first aid procedures; and information on whether OSHA, the International Agency for Research on Cancer or the National Toxicology Program considers the chemical a carcinogen.

For the complete requirements of the hazard communication rule, refer to OSHA's Hazard Communication Standard (29 CFR 1910.1200).

Emergency Response

Facilities expecting employees to engage in emergency response must develop an emergency response plan as specified by OSHA in 29 CFR 1910.1200. Where employees are not expected to assist in handling emergencies, an emergency plan is not required. However, in this case an emergency action plan in accordance with 29 CFR 1920.38(a) is required.

Employees participating in emergency response must be trained and have competency in the following subjects:

- Knowing and implementing the emergency response plan;
- Classifying and identifying known and unknown materials;
- Selecting and using proper personal protective equipment;
- Understanding hazard and risk assessment procedures;
- Performing containment and/or confinement operations;
- Understanding and implementing decontamination procedures, and
- Understanding basic chemical and toxicological terminology and behavior

Complete training requirements are found in 29 CFR 120.

Some of the OSHA regulations that may have a direct impact on the use of sodium chlorite and/or chlorine dioxide are: Process Safety Management, Permit-required Confined Spaces, Toxic and Hazardous Substances (Air Contaminants), and Hazard Communication.

29 CFR, Occupational Safety and Health Admin., Labor (OSHA)

29 CFR 1910.119, **Process Safety Management of Highly Hazardous Chemicals** (chlorine dioxide)

29 CFR 1910.146, **Permit-Required Confined Spaces**

29 CFR 1910.1000 Table Z-1, **Toxic and Hazardous Substances, Air Contaminants** (chlorine dioxide)

29 CFR 1910.1200, **Hazard Communication** This section is to ensure that the hazards of all chemicals produced or imported are evaluated and that information concerning their hazards is transmitted to employers and employees.

Department of Transportation (DOT)

The following information covers some of the most frequently asked questions on transportation of hazardous materials including specifically questions regarding the transportation of sodium chlorite.

Sodium chlorite (dry) is classed by DOT as an "Oxidizer" hazardous material in 49 CFR 172.101. Transporting vehicles must be placarded with DOT "Oxidizer" 5.1, yellow placards and marked with the UN identification number "1496" during all modes of transportation.

Sodium chlorite (solution) is classed by DOT as a "Corrosive" hazardous material in 49 CFR 172.101. Transporting vehicles must be placarded with DOT "Corrosive" 8, black and white placards and marked with the UN identification number "1908" during all modes of transportation.

Non-bulk packaging must be marked with proper shipping name, ID number, name and address of consignor or consignee (with some exceptions, see 172.301), and have a DOT "Corrosive" (liquid) or "Oxidizer" (dry) label affixed to each container. These requirements are found in 49 CFR 172.300 and 172.400.

Additionally, 49 CFR 172.602 and 172.604 require the following emergency response information be provided and maintained during transportation and at facilities handling and storing hazardous materials:

- Description of the hazardous material
- Immediate hazards to health
- Risks of fire and explosion
- Immediate precautions to be taken in the event of an incident
- Immediate methods for handling fires
- Immediate methods for handling spills or leaks
- Preliminary first aid measures
- A 24 hour emergency response telephone number

The 24 hour number must be on the shipping paper; and when using a third party for emergency response information provider, the person registered with the provider must be clearly identified. The remaining information may be presented on the shipping paper or on a separate document.

The proper shipping descriptions for dry and liquid sodium chlorite, according to current regulations are:

UN1496	UN1908
Sodium Chlorite	Chlorite Solution
5.1	8
PG II	PG II
	Contains Sodium Chlorite

DOT 49 CFR 171.15 requires notice to the National Response Center for certain types of transportation incidents and a written report to DOT. 49 CFR 171.16 also requires a written report to DOT for the unintentional release of any quantity of hazardous material during the course of transportation, which includes loading, unloading or temporary storage.

The DOT requires all hazmat employees to be trained as detailed in 49 CFR 172.700. Some of the key provisions of this rule are:

- Covered "hazmat" employees include those who are employed full-time, part-time, or are self employed and who directly effect hazardous material transportation safety; design, manufacture, fabricate, inspect, maintain, prepare hazardous materials for transportation; load, unload or handle hazardous materials; test, recondition, repair, modify or mark packages for transport; or operate a vehicle used for hazardous materials transportation.
- Training must include general awareness/familiarization (training to recognize and identify hazardous materials), function specific training (applicable to the functions that employee performs), modal specific training (for the individual modes of transportation), safety training (emergency response information and measures to protect the employee from the hazards of the material being transported), security threats, and in-depth security training (for those persons required to operate under a transportation security plan).
- Depending on the nature and extent of the instruction, training completed to comply with OSHA, EPA, or security training programs to comply with federal or international agency requirements may be used to satisfy DOT regulations.

DOT 49 CFR 107.601 requires any person who transports or causes to be transported or shipped in commerce the classes and quantities of hazardous materials defined in the rule, to register and pay applicable fees no later than June 30th each year. The annual fee, including a processing fee ranges from \$275 to \$2,600 based on business size and non-for-profit status.

Some of the DOT regulations that may have a direct impact on the use of sodium chlorite and/or chlorine dioxide are:

49 CFR, Parts 105 – 180, Pipeline and Hazardous Materials Safety Administration, Department of Transportation (PHMSA/DOT)

49 CFR 172.101 (subpart B) **Table of Hazardous Materials and Special Provisions:** All shipments of hazardous materials must be conducted under one of the proper shipping names as specified in this table. The table lists the proper name, hazard class, UN number, packaging group, labels, quantity limitations, and other special information.

49 CFR 172.200 (subpart C), **Shipping Papers:** Specifies requirements for proper shipping descriptions, the shipper's certification and hazardous waste manifests.

49 CFR 172.300, 400, and 500 (subparts D, E, and F), **Marketing, Labeling, and Placarding:** Specifies

requirements for marking, labeling and placarding for hazardous material packages, freight containers, and transport vehicles

49 CFR 172.600 (Subpart G), **Emergency Response Information:** Specifies the type of information that must be present while transporting hazardous materials. The information can be in the form of a Material Safety Data Sheet, Transportation Emergency Guide or separate document.

49 CFR 172.700 (Subpart H), **Training Requirements:** Specifies requirements for training of Hazmat Employees. The type of training required includes general awareness, function specific, safety and emergency response. "Hazmat Employees" are those that prepare, load and unload hazardous materials, qualify packaging for hazardous materials, operate motor vehicles transporting hazardous materials, or are responsible for the safety of transporting hazardous materials.

49 CFR 172.800 (Subpart I), **Safety and Security Plans:** Prescribes requirements for development and implementation of plans to address security risks related to the transportation of hazardous materials in commerce.

In addition to the general requirements outlined above, persons involved in the transportation of hazardous materials must also be aware of requirements under the following sections: 173 (General Requirements for Shipments and Packagings), 174 (Carriage by Rail), 175 (Carriage by Aircraft), 176 (Carriage by Vessel), and 177 (Carriage by Public Highway).

EPA Registration

When used as the parent chemical for on-site production of chlorine dioxide in pesticidal applications, sodium chlorite is governed by the Federal Insecticide, Fungicide and Rodenticide Act (FIFRA) (40 CFR 152, Pesticide Registration & Classification Procedures). This means that the sodium chlorite sold for this purpose must be registered with the EPA and must be used in accordance with the directions listed on the labels.

Pesticidal uses of chlorine dioxide generated by sodium chlorite are biocidal, disinfective or sterilizing in nature. Examples of such uses are: as a bactericide or slimicide in treatment of drinking water, processing plant flume water, and rinse water in produce packing facilities; as a slimicide in recirculating cooling waters; and as a microbiocide in oil recovery operations.

OxyChem technical sodium chlorite products are registered for these applications under the following EPA Registration Numbers:

5382-41 Technical Sodium Chlorite Solution 50
5382-42 Technical Sodium Chlorite

5382-43 Technical Sodium Chlorite Solution 31.25
5382-44 Technical Sodium Chlorite Solution 18.75
5382-45 31% Active Sodium Chlorite
21164-3 Dura Klor
21164-6 Akta Klor 25
21164-9 Akta Klor 7.5

EPA SARA Title III: Emergency Planning and Community Right To Know Act (EPCRA)

The provisions under EPA SARA Title III do not specifically regulate sodium chlorite.

Food and Drug Administration (FDA) – 21 CFR Parts 1 to 1499

The Federal Food, Drug, and Cosmetic Act (FFDCA) authorizes the assessment of the safety of ingredients in food, drugs, and related products. The present FFDCA was passed in 1938. Later amendments have been added to develop the current food and drug law.

FDA regulations can be found in 21 CFR Parts 1 to 1499. The application of sodium chlorite and chlorine dioxide is a complex issue. It is often difficult to identify the regulatory agency that is responsible for overseeing the use of sodium chlorite and/or chlorine dioxide in a particular segment of the food processing industry. Please contact OxyChem's Technical Services Department to assist with determining which agency has jurisdiction over your specific use of sodium chlorite and/or chlorine dioxide.

United States Department of Agriculture (USDA)

USDA authorizes the inspection and regulation of meat, poultry, dairy products and eggs. Meat and meat products are outside FDA's jurisdiction except for product labeling where meat products are commingled with fabricated food. USDA's role in other food products is production oriented only. The regulatory aspects, such as monitoring pesticide residues, are left to FDA. In addition to the inspection programs administered by USDA, it also establishes and administers several food and nutrition related programs.

Pursuant to the Meat Inspection Act and Poultry Inspection Act, USDA is extensively involved in the inspection and regulation of meat and poultry products at all states of production. After the food products have left the USDA inspection plant, FDA and USDA have concurrent authority for establishing safeguards and regulating the meat and poultry products. [FDA Compliance Policy Guide](#) No. 7111.01. Besides the initial inspection of meat and poultry, the USDA approves new plant construction and equipment, develops and monitors plant sanitation standards, and conducts technical training of inspection personnel. Additionally, to insure compliance with USDA procedures, the USDA review shipping, handling, and all other functions performed by allied industries engaged in the transportation, storage, and

distribution of meat and poultry products to assure that the products remain unadulterated and truthfully labeled. References: Federal Publications Inc., "Practical Food Law", Course Manual, 22, 1993.

USDA regulations can be found in 7 CFR Parts 1 to 5099.

Clean Air Act

The federal Clean Air Act was enacted in 1970, with several amendments, including a major overhaul in 1990. The Act provides the regulatory vehicle for prevention and control of discharges into the air of substances that may harm public health or natural resources. This Act consists of eleven titles including Title I (non-attainment provisions) and Title III (hazardous air pollutants). Sodium chlorite is not considered a VOC or hazardous air pollutant and is not regulated under the Clean Air Act.

Chlorine dioxide is on the EPA's List of Regulated Toxic Substances and Threshold Quantities as proposed to be regulated in the Accidental Release Prevention provision of Title III of the Act. A 1,000 pound threshold quantity currently serves as the trigger for the application of these rules in reference to chlorine dioxide.

Federal Water Pollution Control Act (Clean Water Act)

Sodium chlorite is a controlled substance in water media. The primary statutory mechanism for controlling water pollution is provided by the Federal Water Pollution Control Act (FWPCA). The FWPCA was amended in 1972 by the Federal Water Pollution Control Act Amendments, in 1977 by the Clean Water Act, and in 1978 by the Clean Water Act Amendments. Most people refer to this growing statute simply as the Clean Water Act (CWA). Regulations promulgated pursuant to the CWA can be found throughout 40 CFR Parts 104-140.

Users of sodium chlorite who generate a wastewater stream should be aware that regulations promulgated pursuant to the Clean Water Act most likely affect them. The basic prohibition of the CWA is that it is unlawful for any person to discharge, without a permit, any pollutant from a point source into navigable waters of the U.S. As discussed under the Community-Right-to-Know Section of this manual, sodium chlorite is considered a hazardous substance.

As a hazardous substance, any discharge of sodium chlorite to waters of the U.S. requires a permit. Sodium chlorite users should contact their state regulatory agency to obtain information on applying for a permit consistent with the Clean Water Act requirements. These permits operate under a CWA program called the National Pollutant Discharge Elimination System (NPDES) and are generally

referred to as NPDES permits. Pollutant limitations contained in these permits are, in part, based on federal or state effluent limitations.

Effluent limitations are set by industry category and are contained in 40 CFR Parts 405 through 471. Pollutant limitations may also be set by Best Management Practices (BMPs), Best Practicable Control Technology (BPT), Best Conventional Control Technology (BCT), Best Available Technology Economically Achievable (BAT), Health Based Toxic Effluent Standards, Water Quality Standards, or Water Quality Management Plans.

If a user of sodium chlorite discharges wastewater effluent, not to waters of the U.S. but to a Publicly Owned Treatment Works (POTW) or Sewer Authority, then the user will need a permit from the POTW which may set permit limits for pollutants in the wastewater stream. Effluent limits are based on a variety of standards, but must meet established EPA Pretreatment Standards. Pretreatment Standards are set on an industry specific basis and can be found in 40 CFR Parts 401-471.

Generators planning to discharge to the ground (which is considered a groundwater discharge) or to underground injection wells, must also obtain a permit. Such discharges have to meet the minimum standards and restrictions for these media. These standards and restrictions can be found in 40 CFR Parts 144-149.

Regulations promulgated under the CWA include the Stormwater Regulations. Under these regulations, an owner/operator must apply for a Stormwater Permit under NPDES if the facility is being operated under a Standard Industry Classification Code (SIC) as a manufacturer (codes 2000 - 3999), and if the facility in question discharges stormwater directly to waters of the U.S.


For more information on Stormwater Permitting, call your state water authority or review the requirements on the EPA and state web sites.

Resource Conservation and Recovery Act (RCRA)

Persons who generate, transport, treat, store or dispose of waste sodium chlorite must comply with EPA regulations concerning hazardous waste management. Those regulations are published beginning at 40 CFR Part 260.

Sodium chlorite solutions are classified in 40 CFR 261 as a characteristically hazardous (*i.e.*, corrosive) waste due to a pH of greater than or equal to 12.5. The EPA waste code for corrosive wastes is D002.

Sodium chlorite dry products are classified in 40 CFR 261 as a characteristically hazardous (*i.e.* ignitable)



waste.. The EPA waste code for ignitable wastes is D001.

Other hazardous waste codes may apply, and it is the waste generator's responsibility to properly classify each waste.

RCRA Land Disposal Restrictions in 40 CFR 268.9, establish treatment standards for hazardous wastes before they can be placed in a RCRA landfill. This rule requires that the hazardous characteristic must be removed from the waste before it is disposed of. If this hazardous characteristic remains, then the hazardous waste must be manifested for disposal or treatment at a permitted hazardous waste treatment, storage, or disposal facility.

The following information on materials and equipment for handling and storing sodium chlorite liquid is meant to serve as a general guideline only. A number of factors will influence the selection of materials and equipment. Physical properties of the solution being handled will be of primary importance. All equipment purchased for handling and storing sodium chlorite liquid should be verified by the manufacturer or vendor to be suitable for use under the conditions specified.

The primary concern when handling and storing liquid sodium chlorite is freeze-up and solidification which can occur with certain concentrations of material. Solidification will plug pipelines, valves and other equipment.

If the customer plans on using Solution 50, a heating system is required. Other diluted solutions, such as Solution 31.25, should not require any heating except in very cold environments.

Materials Of Compatibility

Tanks

Tanks should be of closed top construction to prevent product contamination. The tank vapor space should be adequately vented to prevent chlorine dioxide gas accumulation above explosive decomposition limits.

Tank materials of construction:

1. Fiberglass reinforced polyester with Hetron 922 resin or equivalent, color natural, with UV protection.
2. High density polyethylene (HDPE)
3. Titanium

Fiberglass Reinforced Plastic (FRP) construction with no internal insulation. Typical size would be 10' in diameter by 14' straight side height holding approximately 8,000 gallons. One typical full truck load would be 4,200 gallons. Nozzles should include truck unloading vent, and pump suction as a minimum. Nozzles should be provided to allow a local level and temperature indication. Manways should also be provided to allow access to the interior of the tank.

Pumps

Pumps are a typical source of leakage around glands and packing. This type of spillage should be promptly cleaned up and the area washed down with water. The water should then be drained into an industrial sewer in accordance with the regulations that govern wastewater discharges in your area. Greaseless lubricants should be used in areas where spilled material or dust from dried material may come in contact with the lubricants or the housings containing those lubricants.

Pump should not be run against closed valves. This may result in heating the sodium chlorite solution to above its decomposition temperature.

The pump should be a centrifugal type with all 316 SS wetted parts. The seal for the pump should be a double mechanical type. Motor for the pump should meet local requirements and codes.

Pump materials of construction (Wetted Parts):

1. 316 SS
2. Polypropylene
3. Seal type:
Continual running = Dynamic
Start/Stop = Double Mechanical

Piping

Design the piping system to avoid space which may trap gases. The piping system should be designed to accommodate thorough flushing or complete drain down especially in cold climates where low temperatures may induce crystallization of the liquor in the piping system.

The recommended material is CPVC piping. Other materials such as vinyl esters and Teflon piping may be used. Carbon steel and stainless steel are not recommended. Minimum pipe size should be 1 1/2 inches.

Piping materials of construction:

1. CPVC
2. Vinyl Ester FRP
3. Teflon lined pipe

Heat tracing and insulation (calcium silicate or equivalent) may be appropriate where solution freezing point is exceeded. However, if that approach is used, a temperature controller should be used to keep from generating any "hot" spots in the piping. Heat tracing temperature settings should be specific for the product being used.

Equipment Sources

The equipment suppliers listed here are believed to be reliable. This is, however, only a partial listing as space does not permit a listing of every supplier of each type of equipment.

Listings in this section is for information only and should not be considered as a recommendation for one supplier's products over another.

Pumps

Flowserve Corporation
5215 N. O'Conner Blvd.; Suite 2300
Irving, TX 75039
(972) 443-6500
www.flowserve.com

Goulds Pumps, Inc.
(FRP*, Teflon-lined, Ceramic)
240 Fall Street
Seneca Falls, NY 13148
(315) 568-2811
<http://www.gouldspumps.com/>

March Manufacturing, Inc.
(Polypropylene)
1819 Pickwick Avenue
Glenview, IL 60025
(847) 729-5300
www.marchpump.com

Met-Pro Corporation (FRP*)
Fybroc Division
700 Emlen Way
Telford, PA 18969
(215) 723-8155
www.fybroc.com

Ansimag Inc.
14845 W. 64th Ave.
Arvada, CO 80007-7523
(303) 425-0800
www.ansimag.com

Wilden Pump and Engineering, LLC
22069 Van Buren Street
Grand Terrace, CA 92313-5607
(909) 422-1730
www.wildenpump.com

Piping

Fiber Glass Systems
2425 S.W. 36th Street
P.O. Box 37389
San Antonio, TX 78237
(210) 434-5043
<http://www.starfiberglass.com/index.htm>

P.E.P
50 Tannery Rd., Bldg #3
Branchburg, NJ 08876
(800) 407-3726
<http://www.pep-plastic.com>

Crane Resistoflex (Plastic Lined Steel)
One Quality Way
Marion, NC 28752
(828) 724-4000
<http://www.resistoflex.com/>

Hoses

Birmingham Rubber & Gasket Company Inc.
200 Industrial Drive
Birmingham, AL 35211
(205) 942-2541
www.bragusa.com

Goodyear Engineered Products
(888) 899-6354
<http://www.goodyearep.com/>

Rubber Belting & Hose Supply Inc.
1850 North Ohio
Wichita, KS 67214
(800) 835-0226
<http://www.rbhinc.com>

Valves

Flowserve Corporation
5215 N. O'Conner Blvd. Suite 2300
Irving, TX 75039
(972) 443-6500
www.flowserve.com

France Compressor Products
Division of Goodrich
104 Pheasant Run
Newtown, PA 18940
(800) 675-6646
www.francecomp.com

ITT Engineered Valves
33 Centerville Road
Lancaster, PA 17603-2064
(800) 366-1111
<http://www.engvalves.com>

Xomox Corporation
4444 Cooper Road
Cincinnati, OH 45242
(513) 745-6000
www.xomox.com

Crane Resistoflex (Teflon)
1 Quality Way
Marion, NC 28752
(828) 724-4000
www.resistoflex.com

Fiberglass-Reinforced Plastic Tanks

Ershigs, Inc.
742 Marine Drive
Bellingham, WA 98225
(360) 733-2620
www.ershigs.com

Heil Process Equipment Company
34250 Mills Road
Avon, OH 44011
(440) 327-6051
www.heilprocessequipment.com

Composites USA, Inc.
One Peninsula Drive
North East, MD 21901
(410) 287-2700
<http://www.compositesusa.com>

Plastico Industrial Plastics
32 Flicker Street
Memphis, TN 38104-5918
(901) 452-6527
www.plastico-memphis.com

Gaskets

Garlock Sealing Technologies
1666 Division Street
Palmyra, NY 14522
(800) 448-6688
www.garlock.com

O'Brien Corporation
1900 Crystal Industrial Court
St Louis, MO 63114
(314) 423-4444
www.obcorp.com

Gauges

The Foxboro Company
33 Commercial Street
Foxboro, MA 02035
(866) 746-6477
<http://www.foxboro.com>

Meriam Instrument Division
Scott & Fetzer Company
10920 Madison Avenue
Cleveland, OH 44102
(216) 281-1100
<http://www.meriam.com>

Uehling Instrument Company
473 Gatty Avenue
Paterson, NJ 07503
(973) 742-8710
www.uehling.com

Jogler, Inc.
9715 Derrington
Houston, TX 77064
(800) 223-8469
www.jogler.com

Unloading Sodium Chlorite Tank Trucks

Preparation

The unloading area should be arranged so that any spillage that might occur will be properly contained for recovery. The unloading area should be level and constructed to allow the truck to be driven away in a forward direction.

The truck's parking brakes must be set. When the unloading operation is performed in an area accessible to untrained personnel, a safety rope bearing "DANGER - Sodium Chlorite" signs should be set up around the unloading area.

Unloading operations must be carried out under adequate supervision and by properly trained employees.

Precautions

Trained, properly equipped personnel are required for safe unloading of sodium chlorite tank trailers. The unloading operation should be continuously watched and if it should be necessary for the operator to leave, the operation should be shut down.

If a tank trailer sample is required, all pressure must be bled off the tank trailer before opening the manway.

Emergency showers and eye wash fountains should be located in the unloading area and easily accessible in case of accidental exposure.

Smoking should not be allowed in the unloading area.

Unloading should be done during daylight hours. If it is necessary to unload after dark, adequate lighting must be provided.

Unloading with Compressed Air

Before starting the unloading operation, it must be verified that the storage tank is adequately vented and that the tank will hold the entire contents of the tank trailer. The tank vent should be properly sized for the air surge which occurs at the end of the product transfer which is approximately 1350 SCFM (Standard Cubic Feet per Minute). **(See Figure 1)**

Follow these instructions:

1. Connect the air line from the trailer compressor to the trailer air inlet valve.
2. Connect one end of the sodium chlorite unloading hose to the product discharge valve on the trailer and the other end to customer's storage tank inlet valve.


3. Open the product outlet valve on the sodium chlorite discharge piping on the trailer and open the inlet valve on storage tank.
4. Slowly apply air pressure until there is a steady flow of product into the storage tank. The air pressure in the trailer must not be allowed to exceed 30 psig.
5. When the trailer is empty, shut off compressor or other source of air pressure.
6. Allow the unloading line to be blown empty into the storage tank; then close the product outlet valve on the tank trailer.
7. Immediately close the product inlet valve to the storage tank.
8. Bleed the pressure off the product hose by slowly opening the drain valve at the trailer discharge or at the storage tank inlet. Any residual product remaining in the hose should be drained to a suitable chemical sewer. Disconnect the unloading line from the trailer and the storage tank. OxyChem recommends that the customer provide a drain valve between the hose connection and inlet valve to depressure and drain the product unloading hose prior to disconnection.
9. If any sodium chlorite has spilled on the outside of the truck or on the ground, it should be washed off with plenty of water and drained into an appropriate chemical sewer.

Unloading by Pump

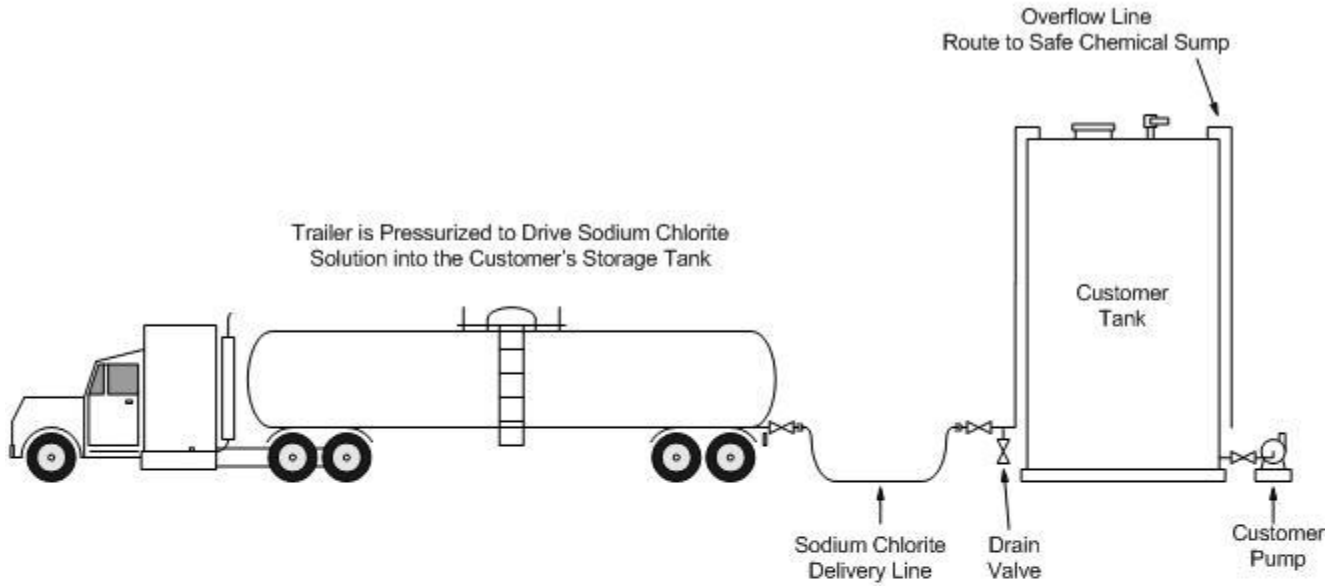
Before starting the unloading operation, it must be verified that the storage tank will hold the entire contents of the tank trailer. Either a centrifugal or rotary pump proven suitable for sodium chlorite must be provided by the customer for this type of unloading.

Follow these instructions:

1. Be sure the manway cover is open and remains open during the unloading operation to ensure the tank trailer is properly vented.
2. Connect one end of the sodium chlorite unloading hose to the product discharge valve on the trailer and the other end to the customer's pump inlet valve.
3. Open the product discharge outlet valve on the trailer and check for leaks.
4. Open the pump product inlet and outlet valves.
5. Start the pump and continue pumping until the tank trailer is empty.

- 
6. After the tank trailer has been emptied, shut off the pump and immediately close the valve on the pump inlet piping and close the trailer discharge valve.
 7. Bleed the pressure off the product hose by slowly opening the drain valve at the trailer discharge or at the pump inlet. Any residual product remaining in the hose should be drained to a suitable chemical sewer. Disconnect the unloading line from the trailer and the pump inlet. OxyChem recommends that the customer provide a drain valve between the hose connection and pump inlet valve to depressure and drain the product unloading hose prior to disconnection.
 8. Close and secure the top manway cover.

Unloading with Compressed Air



Unloading by Pump

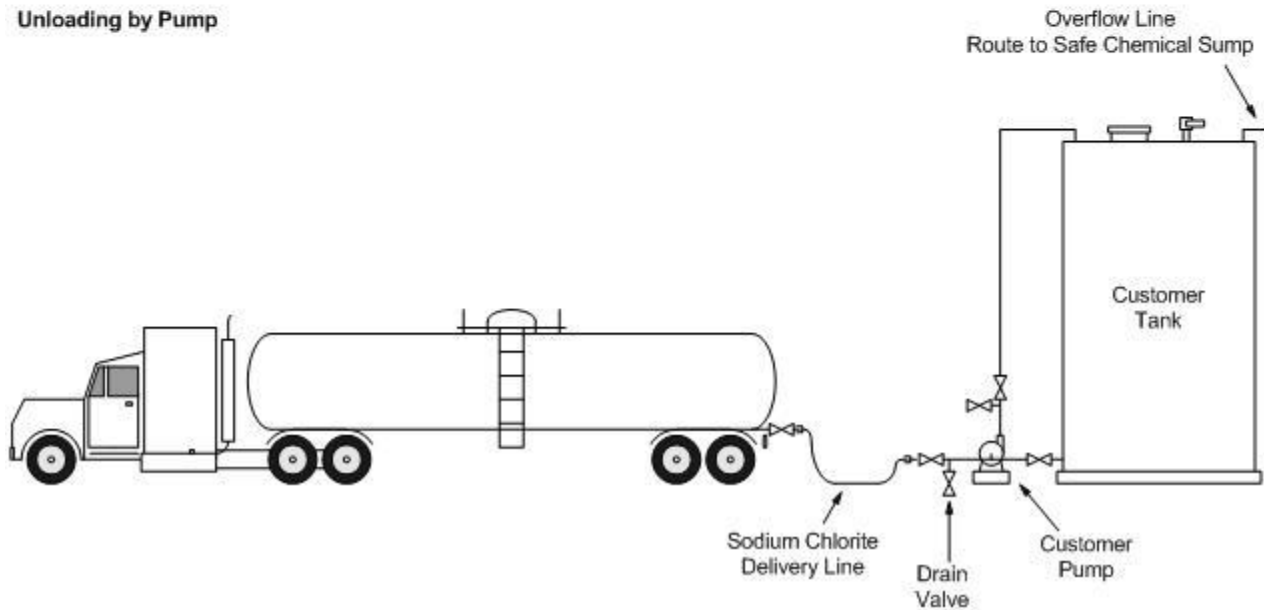


Figure 1: Unloading Sodium Chlorite Tank Trucks

Preparing a 25% sodium chlorite solution from OxyChem Technical Sodium Chlorite (dry) product.

The following conversions will enable you to blend a 25% sodium chlorite solution from OxyChem Technical Sodium Chlorite (dry) product.

To determine the amount of OxyChem Technical Sodium Chlorite and water needed to prepare a specified number of gallons of 25% sodium chlorite solution you would follow the following steps:

1. Multiply the desired number of gallons of 25% sodium chlorite solution by the weight per gallon of a 25% sodium chlorite solution prepared from OxyChem Technical Sodium Chlorite dry product (10.27 lbs./gal.). This gives you the total weight of the desired volume of sodium chlorite solution.
2. Multiply the total weight of the sodium chlorite solution by 0.25 (25%) to determine the weight of sodium chlorite required to prepare that solution, and then divide the sodium chlorite weight by 0.80 (80%) to determine the amount of dry product (80% NaClO₂ by wt.) required.
3. Now that you know the weight of OxyChem Technical Sodium Chlorite dry product in the desired volume of 25% sodium chlorite solution, subtract that weight from the total solution weight to determine the weight of the water required to prepare the 25% solution. The weight of the water can be converted to gallons by dividing by 8.34 lbs./gal.

Example: How much OxyChem Technical Sodium Chlorite dry product and water is required to prepare 50 gallons of 25% sodium chlorite solution?

$$50 \text{ gals.} \times 10.27 \text{ lbs./gal.} = 513.5 \text{ lbs.}$$

513.5 lbs. \times 0.25 = 128.38 lbs. of sodium chlorite required to prepare 50 gallons of 25% active sodium chlorite solution.

128.38 lbs. NaClO₂ / 0.8 = 160.48 lbs. of OxyChem Technical Sodium Chlorite dry product required to prepare the desired volume of 25% sodium chlorite solution.

513.5 lbs. - 160.48 lbs. = 353.02 lbs. of water is required to prepare 50 gallons of 25% sodium chlorite solution.

353.02 lbs / 8.34 = 42.33 gallons of water is required to prepare the 50 gallons of 25% sodium chlorite solution.

Sodium Chlorite Solutioning Instructions:

1. To aid in the solutioning process, the water should be between 100 to 120 degrees F.
2. Mix as you add the 80% Technical Sodium Chlorite dry product.
3. Add sodium chlorite in free flowing flake form.
4. Mix for 30 minutes and sample to verify proper strength.
5. Allow to cool, then drum.

Figure 2 illustrates how crystallization temperature varies with the solution concentration. Crystallization temperature is not necessarily the temperature at which the solution freezes, but instead the temperature at which the sodium chlorite begins to form crystals and fall out of solution. This curve is based on OxyChem solution products and not solutions made from the dry product.

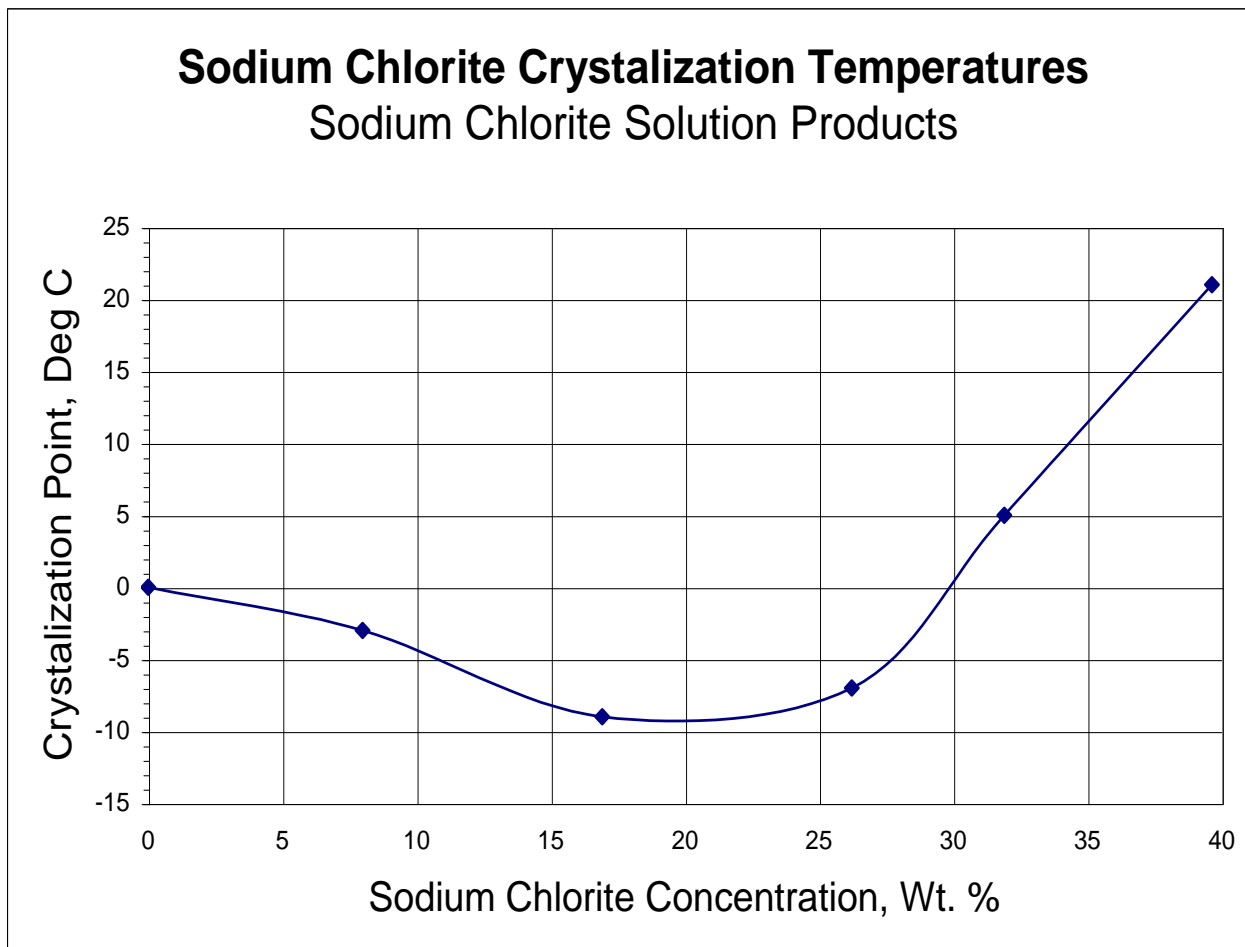


Figure 2: Sodium Chlorite Crystallization Curve

Methods of Analysis

Analytical Methods presented here shall be limited to those methods used for quality control; not for field testing. The following OxyChem Standard Methods of Analysis are equivalent to AWWA Standards for Sodium Chlorite ANSI/AWWA B303-88.

Determination of Sodium Chlorite Strength in Sodium Chlorite Products

Method Summary

Sodium chlorite assay is determined in sodium chlorite product samples by iodometric titration of the iodine released when the samples are treated with potassium iodide and hydrochloric acid. The samples are titrated with a standard sodium thiosulfate solution.

Apparatus:

1. 250 ml Erlenmeyer flask.
2. 500 ml Class A Volumetric flask.
3. 20 ml Class A Volumetric pipet.
4. Analytical balance, 3 place.
5. 50 ml buret.

Reagents and Solutions:

1. Potassium Iodide, 5% solution, LaMarKa # VW-13.
2. 1.0 N Hydrochloric Acid, LaMarKa # VW-9.
3. 0.1 N Sodium Thiosulfate standard solution, Fisher cat # SS368-20.
4. Starch indicator solution, 5 gpl amylose in water, LaMarKa # VW-20.
5. Deionized water.

Procedure

1. Weigh sample (to the nearest 0.001 grams) into a 500 mL volumetric flask, according to the following table:

PRODUCT GRADE	TARGET WEIGHT
Solution 31.25	10. grams
31 % Active	8. grams
Solution 50	6.5 grams
Solid Flake	3. grams

2. QS to 500 ml and mix well.
3. Rinse a 20 ml Class A pipet with the above solution. Pipet 20 ml of the solution into a 250 ml erlenmeyer flask containing 100 ml of DI water.
4. Add 40 ml of 5% KI and MIX WELL. (KI MUST be added before HCl.)

5. Add 6.5 ml of 1 N HCl and mix.
6. Titrate with 0.1 N $\text{Na}_2\text{S}_2\text{O}_3$ to a faint yellow color.
7. Add Starch indicator and continue the titration to a colorless endpoint. Record titer to the nearest 0.01 ml.

Calculations:

$$\text{Wt \%NaClO}_2 = \frac{\text{ml of Na}_2\text{S}_2\text{O}_3 \times \text{N} \times \text{Dilution Factor} \times 2.2612}{\text{Sample Weight in grams}}$$

$$\text{Dilution Factor} = (500/20) = 25$$

$$\text{N} = \text{normality of Na}_2\text{S}_2\text{O}_3$$

simplified calculation:

$$\text{Wt \%NaClO}_2 = \frac{\text{ml Na}_2\text{S}_2\text{O}_3 \times 56.53 \times \text{N}}{\text{Sample Weight}}$$

$$\text{gpl NaClO}_2 = \text{Wt\% NaClO}_2 \times \text{Specific Gravity} \times 10$$

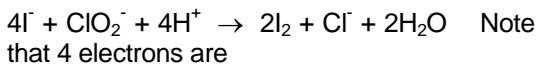
Notes

1. Sodium chlorite is a very strong oxidizing reagent and as such is incompatible with strong reducing agents, finely powdered metals, phosphorus and acids!
2. Keep solid sodium chlorite away from combustible materials, heat, sparks and open flame!
3. Since potassium iodide will oxidize when exposed to air in acidic conditions, the starch endpoint color may return after the titration is actually completed. Therefore, record the ml of sodium thiosulfate used at the first clear endpoint.
4. KI **MUST** be added and mixed before 1.0 N HCl. If the HCl is added first, chlorine dioxide will gas off, and the strength will be reported lower than actual.
5. A reagent blank should be run periodically, to insure the purity of reagents. This will mainly be a check on the KI solution. It could possibly become oxidized with chlorine (turning it yellow) which will give erroneously high results.
6. If the alkalinity of the sample is unusually high, 10 ml of 1 N HCl may be needed, rather than 5 ml. The pH needs to be between 1 and 2 for the ClO_2^- to oxidize the I^- completely.

Following is a table showing the relationship between volume of 1.0 N HCl and pH, for a Solution 50 assay:

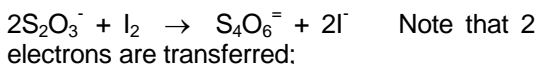
ml of 1.0 N HCl Added	pH	Assay: NaClO ₂	%
0	9.0		
0.5	6.0		
1.0	5.6		
1.5	5.6		
2.0	5.3	15.1%	
2.5	5.2		
3.0	5.1	23.3 %	
3.5	5.0		
4.0	4.9	31.6 %	
4.5	4.2		
5.0	2.5	37.4 %	
5.5	2.1		
6.0	1.9	37.4 %	
6.5	1.8	Recommended Target	
13.0	1.2		
20.0	1.0		
30.0	0.9		

7. Reaction that occurs when acid is added (KI already present):



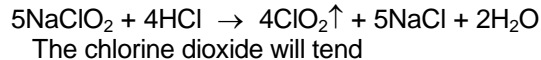
transferred; thus, equivalent weight of sodium chlorite is molecular weight divided by 4.

8. Reaction that occurs during titration:



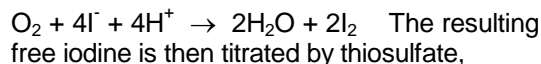
thus, equivalent weight of sodium thiosulfate is molecular weight divided by 2.

9. Reaction that occurs if acid is added BEFORE KI is added:



to gas out of solution, and thus will not be titrated. Results for NaClO₂ strength will be falsely low.

10. Reaction that occurs if titration is done too slowly, or if too much acid is added:



giving falsely high results.

References

1. Mathieson Chemical Corporation, Niagara Falls Quality Control Lab.
2. Aieta et al. "Detn of Chlorine Dioxide, Chlorine, Chlorite, and Chlorate in Water," Journal of AWWA, January, 1984.

Determination of Sodium Hydroxide and Sodium Carbonate in Sodium Chlorite Products

Method Summary

Sodium hydroxide and sodium carbonate are determined in sodium chlorite product samples by an acid/base titration of two samples (one of which has been treated with barium chloride to precipitate barium carbonate), using hydrochloric acid titrant. The difference between the two titrations determines the sodium carbonate present.

Apparatus

1. Two 250 ml Erlenmeyer flasks, with stoppers.
2. Analytical balance, 3 place.
3. Buret, 50 ml.

Reagents

1. 0.1 N Hydrochloric Acid, LaMarKa # VW-8.
2. Phenolphthalein indicator, 5 gpl in Methanol, LaMarKa # VW-12.
3. Deionized water.
4. Barium Chloride, 100 gpl, LaMarKa # VW-2.

Procedure

- 1A. Solid Sodium Chlorite Sample:
Weigh 10.0 gm into each erlenmeyer flask, record the weights to the nearest 0.001 gm, and label the flasks A and B.

- 1B. Sodium Chlorite Solutions:
Pipet 25 ml of sample into each Erlenmeyer flask, record the weights to the nearest 0.001 gm, and label the flasks A and B.
- To flask A add 50 ml of DI water. Stopper and mix well.
 - To flask B add 40 ml of 10 % BaCl₂. Stopper, mix well, and allow to sit for at least 3 minutes.
 - Add phenolphthalein indicator to each flask.
 - While swirling vigorously, titrate flask A with 0.1N HCl to the absence of pink; record titer to the nearest 0.01 ml.
 - While swirling vigorously, titrate flask B with 0.1N HCl to the absence of pink; record titer to the nearest 0.01 ml. The B titer will be smaller than the A titer.

Calculations:

$$\% \text{ NaOH} = \frac{\text{ml B} \times \text{N} \times 4.000}{\text{Weight of Sample B in grams}}$$

$$\% \text{ Na}_2\text{CO}_3 = \frac{(\text{ml A} - \text{ml B}) \times \text{N} \times 10.5999}{\text{Average Weight of Samples A and B in grams}}$$

$$\% \text{ Total Alkalinity} = (\% \text{ Na}_2\text{CO}_3 \times 0.7547) + (\% \text{ NaOH})$$

Notes

- Sodium chlorite is a very strong oxidizing reagent and as such is incompatible with strong reducing agents, finely powdered metals, phosphorus and acids!
- Keep solid sodium chlorite away from combustible materials, heat, sparks and open flame!
- NaClO₂ may degrade the indicator, and more may need to be added when the endpoint is approached.
- Swirl flask vigorously during titration to minimize localized reaction of acid with chlorite:

$$5\text{NaClO}_2 + 4\text{HCl} \rightarrow 4\text{ClO}_2\uparrow + 5\text{NaCl} + 2\text{H}_2\text{O}$$

Determination of Hydrogen Peroxide in Sodium Chlorite Products

Method Summary

Hydrogen peroxide is determined in sodium chlorite product samples by an iodimetric titration of the sample using a 0.1 N sodium hypobromite titration solution.

Apparatus:

- 500 ml Erlenmeyer flask.
- 10 ml volumetric pipet.
- Analytical balance, 4 place.
- 50 ml buret.

Reagents:

- 0.1 Normal Sodium Hypobromite solution, LaMarKa # VW-76.

Preparation:

- Add 1 Liter DI water to a 2 Liter volumetric flask.
- Add 17 ml of 50% membrane NaOH.
- Add 5.8 ml reagent liquid Bromine.
- Stopper and mix well, until reaction ceases.
- QS to 2 Liters, mix well, and store in amber bottle.

Standardization:

- dd 150 ml DI water to a 500 ml erlenmeyer flask.
- Pipet 25 ml of NaOBr solution into flask.
- Add 40 ml of 5% KI (or 2 gm KI crystals).
- Add 8 ml glacial acetic acid, and 5 drops starch indicator.
- Titrate with standard 0.1 N Na₂S₂O₃ to a clear endpoint.
- Record titer, and calculate as follows:

$$\text{Normality of NaOBr} = \frac{\text{ml Na}_2\text{S}_2\text{O}_3 \times \text{Normality}}{\text{ml NaOBr}}$$

- If the normality of the NaOBr is more than 1% too strong (over 0.101 N), adjust it as follows:

$$\text{ml DI water to add} = (1975 \times \text{N}) - 1975$$

- Starch indicator solution, 5 gpl, LaMarKa # VW-20.
- Deionized water.
- Potassium Iodide, 5% (w/v), LaMarKa # VW-13.

Procedure

Blank Titration:

- Add approximately 150 ml of deionized or equivalent water to a 500 ml erlenmeyer flask.
- Add 20 ml of 5% KI solution (or 1 gram of KI crystals).
- Add 2 ml of starch indicator.

4. Titrate the sample using the 0.1 N NaOBr titration solution by adding it dropwise.
5. When a light blue color appears and is permanent the titration is complete.
6. Record the number of mls of 0.1 N NaOBr used to titrate the blank.

Sample Titration:

1. Add approximately 150 ml of deionized or equivalent water to a 500 ml erlenmeyer flask and tare the flask.
2. Add 20 ml of 5% KI.
3. Add 2 ml of starch indicator.
4. Pipet 10 ml of sample into flask, holding the tip of the pipet below the surface of the liquid. If sample is a solid flake, weigh in 10 grams of sample. Record sample weight.
5. Titrate the sample using the 0.1 N NaOBr titration solution by adding it dropwise.
6. When a light blue color appears and is permanent the titration is complete.
7. Record the number of mls of 0.1 N NaOBr used.

Calculations:

10 ml Liquid Sample:

$$\% \text{H}_2\text{O}_2 = \frac{(\text{ml of NaOBr} - \text{ml Blank}) \times \text{Normality} \times 0.170074}{\text{Specific Gravity}}$$

Solid Flake Sample:

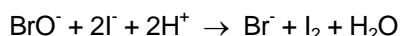
$$\% \text{H}_2\text{O}_2 = \frac{(\text{ml of NaOBr} - \text{ml Blank}) \times \text{Normality} \times 1.70074}{\text{Sample Weight}}$$

Notes

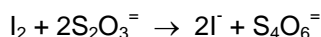
1. Sodium chlorite is a very strong oxidizing reagent and as such is incompatible with strong reducing agents, finely powdered metals, phosphorus and acids!
2. Keep solid sodium chlorite away from combustible materials, heat, sparks and open flame!
3. Hydrogen peroxide is unstable in sodium chlorite solutions and will react with other contaminants in the sodium chlorite sample. For this reason, the samples should be analyzed for hydrogen peroxide as soon as possible.

4. Handle liquid bromine under a fume hood using appropriate protection.

5. Reaction during standardization of NaOBr:



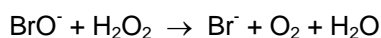
Hypobromite oxidizes iodide. A two electron transfer.



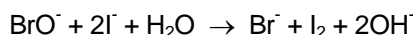
Thiosulfate reduces iodine. A two electrontransfer.

6. Reaction during analysis of H₂O₂:

$$\text{H}_2\text{O}_2 + \text{I}^- \rightarrow \text{No Rx in basic solution. Solution stays clear.}$$



Hypobromite oxidizes peroxide. A two electron transfer.



Hypobromite oxidizes iodide, forming iodine, which forms a blue complex with starch. A two electron transfer.

Determination of Sodium Chlorate and Sodium Sulfate in Sodium Chlorite by Ion Chromatorgraphy with Conductivity Detection

Method Summary

Chlorate and sulfate levels are determined in sodium chlorite by ion chromatography, using an IONPAC AS9-SC separator column. Sodium chlorite is injected into a DIONEX 2000i/sp ion chromatograph after dilution with DI water. Concentrations of chlorate and sulfate are determined by comparison with response curves and calculated as sodium chlorate and sodium sulfate.

Apparatus

1. Dionex Model 2000i/SP ion chromatograph or equivalent.
2. Dionex Ionpac AS-9 separator column.
3. Dionex Ionpac AG-9 guard column.
4. Dionex AMMS-II Micro Membrane Suppressor.
5. Nelson chromatography, electronic data handling system.
6. 100 ml volumetric flasks.
7. Disposable transfer pipet.
8. 3 ml plastic disposable syringes.
9. Analytical balance, 4 place.

Reagents

1. Milli-Q Deionized water. (Low conductivity, free from sulfate and chlorate.)
2. Sodium Carbonate, ACS Reagent grade. Na₂CO₃. Fisher cat # S263-500.
3. Sodium Bicarbonate, ACS Reagent grade. NaHCO₃. Fisher cat # S233-500.
4. Concentrated Sulfuric Acid, ACS Reagent grade. Fisher cat # A300S-212.
5. Stock Eluent Concentrate: Into a 1 liter volumetric flask, weigh 21.978 grams of Na₂CO₃ and 6.301 grams of NaHCO₃, qs to 1000 ml. Mix thoroughly, store in a labeled plastic container. (200 mM Na₂CO₃/75 mM NaHCO₃)
6. Working Eluent for chromatograph: Pipet 20 ml of stock eluent into a 2 liter volumetric flask (2mM Na₂CO₃ and 0.75 mM NaHCO₃). qs to 2000 ml with Milli-Q DI water.
7. 0.025 N sulfuric acid: To 4 liters Milli-Q deionized water, add 2.8 ml of concentrated sulfuric acid. Mix thoroughly.
8. Sodium Chlorate, ACS Reagent grade. NaClO₃. Baker cat # 3616-01.
9. Sodium Sulfate, ACS Reagent grade. Na₂SO₄. Fisher cat # S421-500. (Alternative: Standard solution 50.0 ± 0.5 mg/l as SO₄ Hach cat # 2578-49)

PRODUCT GRADE	TARGET WEIGHT
Solution 31.25	10 grams
31 % Active	8 grams
Solution 50	6.5 grams
Solid Flake	3 grams

3. QS to 500 ml and mix well.
4. Rinse a 0.5 ml Class A pipet with the above solution. Pipet 0.5 ml of the solution into a 100 ml Erlenmeyer flask containing 50 ml of DI water, qs to 100 ml. Mix well.
5. Prepare and inject a Milli-Q or equivalent water blank into the ion chromatograph.
6. Prepare and inject 5 ml of the diluted sample in to the ion chromatograph and press the start button on the Nelson box.
7. Quantitation of sodium chlorate/sodium sulfate will be accomplished by comparison of sample peak heights to standard peak heights OR via the Nelson integrator data system which also uses peak height. If using the Nelson, use a "Nelson dilution factor" of 100,000 and a "Nelson sample weight" of Wt (from step 2 above) divided by specific gravity. For flake, use the weight from step 2 above for "Nelson sample weight."

Procedure

Instrument parameters:

Sample size:	50 µl
Columns:	Ionpac AG-9 Guard column Ionpac AS-9 Separator column
Eluent:	2.0mM Na ₂ CO ₃ /0.75mM NaHCO ₃ Flow rate= 2.0 ml/min
Pressure:	850-1400 psig
Suppressor:	AMMS II Micromembrane suppressor
Regenerant:	0.025N H ₂ SO ₄ Flow rate = 7-10 ml/min
Sensitivity:	3 µs

1. Start the ion chromatograph and ensure that proper flows of eluent and regenerant are established. Allow the instrument to equilibrate and ensure that a stable baseline is established. Select the appropriate detector output range setting.
2. Weigh sample (to the nearest 0.001 grams) into a 500 ml volumetric flask, according to the following table:

Calculations

Peak Height Single Point Calibration:

$$\text{ppm NaClO}_2/\text{Na}_2\text{SO}_4 = \frac{\text{Sx Pk Ht} \times \text{Std Conc.} \times 100,000 \times \text{Sp Gr}}{\text{Std pk ht} \times \text{Sx wt}}$$

where: Sx Pk Ht = sample peak height,
Std Conc = standard concentration (ppm),
Sp Gr = specific gravity of sample,
Std Pk Ht = standard peak height,
Sx wt = actual sample weight in grams, and
100,000 = dilution factor.

Nelson Calibration Curve:

$$\text{ppm NaClO}_2/\text{Na}_2\text{SO}_4 = \frac{\text{Cal Conc} \times 100,000}{\text{Nelson Sx wt}}$$

where:

Cal Conc = ppm NaClO_2 or Na_2SO_4 concentrations
from calibration curve
100,000 = Nelson dilution factor,
Nelson Sx wt = actual sample weight / specific gravity
(for liquid samples), or
Nelson Sx wt = actual sample weight (for flake
samples).

Notes

1. Nitrate and other anions may elute at nearly the same time as chlorate. If the procedure is adapted to other samples, some sort of screening will be necessary to assure that no interfering substances are present.
2. Sodium chlorite is a very strong oxidizing reagent and as such is incompatible with strong reducing agents, finely powdered metals, phosphorus and acids!
3. Keep solid sodium chlorite away from combustible materials, heat, sparks and open flame!
4. Do not use hydroxide eluents or hydroxide to adjust the pH of any eluent higher than pH 11 to effect selectivity changes. Using eluents with pHs greater than 11 may cause irreversible damage to the IonPac AS9-SC/AG9-SC Columns.

Standard Preparation

1. Stock standard (500 mg/l NaClO_3 , 740 mg/l Na_2SO_4): On an analytical balance weigh 0.25 grams of dry sodium chlorate into a weighing boat. Then weigh 0.37 grams of dry sodium sulfate into a weighing boat. Transfer the salts to a 500 ml volumetric flask, rinse boats into flask, QS with Milli-Q DI water, and mix well. Store in a labeled plastic bottle.
2. Working standard: Into a 100 ml volumetric flask about 2/3 full of DI water, micropipet 100 μl of the stock standard. QS and mix well. This standard contains 0.50 mg/l Na_2ClO_3 and 0.74 mg/l Na_2SO_4 .

(1 ml of 50 mg/l Na_2SO_4 soln, qs to 100 ml, gives 0.739 mg/l Na_2SO_4 , if solid Na_2SO_4 is not available.)
3. Analyze the working standard three times, calculate and average the peak heights for both NaClO_3 and Na_2SO_4 . Store and save the averages in Nelson method SO_4 -9B.

FURTHER INFORMATION

More detailed information on OxyChem sodium chlorite or any specific applications is available on request through the OxyChem Technical Service Department.

Technical Service Department

OxyChem

PO Box 12283

Wichita, KS 67277-2283

800-733-1165 Ext. 1

www.OxyChem.com