



Sodium Chlorite Chlorine Dioxide Treatment for Industrial & Municipal Pollutants

Chlorine Dioxide - A Versatile Non-Chlorinating Oxidant

Chlorine dioxide (ClO₂) oxidation of wastes is a critical part of water and air treatment programs being driven by environmental regulations and stricter limitations on chemical contaminant emissions. Chlorine dioxide is a powerful oxidizing agent which can be generated efficiently from sodium chlorite. Its selective reactivity makes chlorine dioxide useful in many of the water and air treatment applications for which chlorine and other oxidizing agents are unsuitable.

Chlorine dioxide was first used in municipal drinking water treatment in 1944, to control taste and odor at the Niagara Falls, NY water treatment plant. Chlorine dioxide was found to be superior to chlorine for destroying phenols, algae, sulfides, iron, and manganese contaminants.

From that initial drinking water application, chlorine dioxide has been commercialized for a wide variety of potable, process, and wastewater applications, as well as for air treatment. A number of these applications are shown in Table 1 and are discussed briefly in later sections.

Summary of Water and Air Regulations

Federal Water Pollution Control Act (Clean Water Act)

The Clean Water Act (CWA) regulates pollutants entering surface waters. 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.

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.

If a user of sodium chlorite discharges wastewater effluent, not to waters of the US but to a Publicly Owned Treatment Works (POTW) or Sewer Authority, then the user will need a permit from the POTW which will set maximum concentration levels for pollutants in the wastewater stream. Effluent levels are based on a variety of standards but must meet established EPA Pretreatment Standards. Pretreatment Standards are set on an industry specific basis.

Safe Drinking Water Act

The Safe Drinking Water Act protects potable water sources and sets drinking water standards. Among other things, drinking water regulations set maximum contaminant levels for microorganisms and chemical contaminants such as total trihalomethanes (TTHM), chlorates and chlorites.

Clean Air Act

The Clean Air Act (CAA) provides the regulatory vehicle for prevention and control of emissions into the air of substances that may harm public health or natural resources. This Act contains provisions to reduce or minimize emissions of volatile organic compounds (VOCs) in ozone non-attainment areas, and to control hazardous air pollutants. Sodium chlorite itself is not considered a VOC or hazardous air pollutant and is not regulated under the Clean Air Act.





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Additional provisions of the Act are designed to control emissions of nitrogen oxides (NO_x) and sulfur oxides (SO_x).

Chlorine dioxide is on the EPA's List of Regulated Toxic Substances and Threshold Quantities, and is regulated in the Accidental Release Prevention provision of Title III of the Act, 40 CFR §68.130. A 1,000-pound threshold quantity currently serves as the trigger for the application of this program to chlorine dioxide.

Chemical Oxidation by Chlorine Dioxide

Introduction

Chlorine dioxide possesses a chemical reactivity which differs markedly from other oxidants (such as chlorine). Commercial applications have shown chlorine dioxide can effectively oxidize many compounds considered to be waste and water pollutants. Table 1 lists a number of pollutants found in various industries. Chlorine dioxide has been shown to be an effective treatment option for those designated by a "•". The following sections briefly summarize reaction conditions and stoichiometries to oxidize various common pollutants.

Sulfides

Many industrial processes produce sulfide-containing gases and waste products. These are generated, for example, during petroleum refining, coal coking, black liquor evaporation in kraft pulping, viscose rayon manufacture and natural gas purification. These gases and wastes are frequently scrubbed with alkaline solutions and require treatment before discharge.

Between pH 5 and 9, an average of 5.2 parts by weight of chlorine dioxide instantaneously oxidizes 1 part by weight of hydrogen sulfide (expressed as sulfide ion) to the sulfate ion (1).



Reduced Sulfur

Reduced sulfur compounds are a broad class of oxy-sulfur compounds, such as sulfite (SO₃⁼) and thiosulfate (S₂O₃⁼), that have an oxidant demand. These compounds are found in the waste streams of the petroleum, steel, pulp & paper,

and most chemical industries. Their high oxidant demand can cause eutrophication of natural waters and excessive chlorine demand in wastewaters treated by POTWs (Publicly Owned Treatment Works).

Chlorine dioxide effectively oxidizes these species to sulfate ions over a broad pH range (5-9). Below a pH of 4, sodium chlorite may be used without the generation of chlorine dioxide.

Nitrogen Compounds

Nitrogen oxides are hazardous and corrosive. Nitrous oxide (NO) and nitrogen dioxide (NO₂) are industrial effluents which result from fuel combustion, nitric acid manufacture and use, and from metal finishing operations which use nitrates, nitrites, or nitric acid. Other sources include chemical processes in which nitrogen compounds are used as reagents.

Chlorine dioxide and/or sodium chlorite have been used to scrub these contaminants. Nitric oxide contained in gas discharges from coke kilns may be eliminated by chlorine dioxide oxidation. The process is particularly convenient for continuous operation.

Cyanides

Cyanide compounds originate from processes such as metal plating, steel case hardening, pickle liquor neutralizing, gold and silver ore refining and blast furnace stack gas scrubbing. Chlorine dioxide oxidizes simple cyanide to cyanate (a less toxic substance) and/or carbon dioxide and nitrogen. The end products depend on reaction conditions.

In neutral and alkaline solutions below pH 10, an average of 2.5 parts by weight of chlorine dioxide oxidizes 1 part by weight of cyanide ion to cyanate (2). Above pH 10, an average of 5.5 parts by weight of chlorine dioxide oxidizes 1 part by weight of cyanide ion to carbon dioxide and nitrogen (3). Chlorine dioxide does not react with cyanate ion, nor has it been observed to form cyanogen chloride during the oxidation of cyanide.

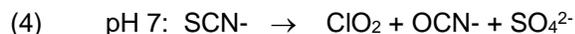




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Chlorine dioxide also oxidizes thiocyanate to sulfate and cyanate. In neutral solutions, an average of 3.5 parts by weight of chlorine dioxide oxidizes 1 part by weight of thiocyanate ion (4).



Phenols

Surface water often contains phenols from industrial effluents. Undesirable phenolic wastes are produced in the chemical, plastics, coke, and petroleum refining industries. If chlorine is used for oxidation, highly toxic chlorophenols are formed.

These chlorophenols can also cause taste and odor problems in drinking water. Ortho-chlorophenol is the most offensive of the phenol compounds. It is objectionable at concentrations as low as 102 ppb.

Treatment with chlorine dioxide can destroy chlorophenols. Below pH 10, 1.5 parts by weight of chlorine dioxide oxidizes 1 part by weight of phenol to benzoquinone. Above pH 10, an average of 3.3 parts by weight of chlorine dioxide oxidizes 1 part by weight of phenol to a mixture thought to be low molecular weight, nonaromatic, carboxylic acids (such as oxalic and maleic acids). At pH 7, the phenol reaction is rapid and complete; all phenols are consumed.

Aldehydes

Aldehydes are produced by a number of industrial processes. Their treatment is a common problem, and particularly so in the photographic industry. In general, chlorine dioxide can oxidize aldehydes to the corresponding carboxylic acid.

Formaldehyde is a major component in the formulations used in photo processing. Chlorine dioxide can oxidize formaldehyde initially, to formic acid and finally, to carbon dioxide. Paraformaldehyde can be depolymerized and eliminated completely by oxidation with chlorine dioxide.

Amines and Mercaptans

The major sources of odorous substances such as mercaptans and substituted amines include the chemical and petroleum industries, coking and sanitary processes, animal feedlots and rendering plants.

Between pH 5 and 9, 4.5 parts by weight of chlorine dioxide instantaneously oxidizes 1 part by weight of a mercaptan (expressed as sulfur) to the respective sulfonic acid/sulfonate compound, destroying the mercaptan odor. Similarly, chlorine dioxide reacts with organic sulfides and disulfides, destroying the original odor.

Secondary and tertiary amines are also present in many wastewaters, causing their own unique odor problems. The oxidation of amines with chlorine dioxide depends on the pH of the reaction mixture and the degree of substitution of the amine.

Between pH 5 and 9, an average of 10 parts by weight of chlorine dioxide oxidizes 1 part by weight of a tertiary aliphatic amine (expressed as nitrogen), destroying the amine odor.

At pH above 7, an average of 5 parts by weight of chlorine dioxide oxidizes 1 part by weight of a secondary aliphatic amine (expressed as nitrogen), removing all traces of amine odor. The higher the pH of the reaction mixture (chlorine dioxide and tertiary and/or secondary aliphatic amines), the more rapidly oxidation proceeds.

THM Precursors

The key to understanding why chlorine dioxide is so effective can be found in the differences in the reactions of chlorine dioxide and chlorine with trihalomethane (THM) precursors such as humic and fulvic acids.

Chlorine reacts with THM precursors by oxidation and electrophilic substitution to yield both volatile and nonvolatile chlorinated organic substances (THMs).

Chlorine dioxide, however, reacts with THM precursors primarily by oxidation to make them nonreactive or unavailable for THM formation. This means that pretreatment with chlorine





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dioxide has an inhibiting effect on THM formation when chlorine is subsequently used.

Pesticides

Some pesticides can be oxidized to fewer toxic materials by chlorine dioxide. Specifically, DMDT (methoxychlor) and aldrin react with chlorine dioxide. With parathion, the reaction is slow near neutral pH; however, when pH is above 8, less biodegradable herbicides such as paraquat and diquat are eliminated within a few minutes.

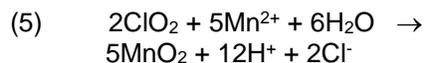
Algae/Slime

Chlorine dioxide has been shown to be effective in controlling algae growth. In one study, chlorine dioxide was found to be more effective than copper sulfate, at comparable treatment costs. Chlorine dioxide is believed to attach to the pyrrole ring of the chlorophyll. This cleaves the ring and leaves the chlorophyll inactive. Since algae cannot function without chlorophyll metabolism, they are destroyed. The reaction of chlorine dioxide with algae and their essential oils forms tasteless, odorless substances. Algae control is carried out by adding chlorine dioxide to the reservoir at night (to prevent decomposition of chlorine dioxide by sunlight). The algae-killing action is fast enough to be effective before the sun rises. A dosage of 1 mg/l has been reported to control algae populations.

Manganese

Manganese ions in water supplies can cause stained clothes, "black" water, water main incrustation, deposits on paper machines, and debris at water customers' taps.

Though chlorine can be used to control these problems, it reacts so slowly that manganese ions may still be in the water distribution system after 24 hours. Chlorine dioxide reacts much more rapidly with manganese, oxidizing it to manganese dioxide (5):



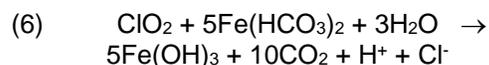
After five minutes contact time, 99+% of the manganese oxide may be removed through a

0.45-micron filter. To remove 1 part of manganese requires 2.45 parts by weight of chlorine dioxide. Favorable results are obtained when the pH is higher than 7.

Iron

Iron from acid waters cannot be tolerated in certain industrial wet processes commonly used in the textile, pulp and paper, and beverage industries. Iron can be a problem, either from the effects of its presence in the water alone, or because iron-bearing water has promoted the growth of iron bacteria.

Above pH 5, an average of 1.2 parts by weight chlorine dioxide oxidizes 1 part by weight soluble iron (ferrous) to insoluble iron (ferric) (6).



Above pH 5, the resulting ferric form of iron is 99% removable by a 0.45-micron filter after five minutes.

Chlorine dioxide has been reported to oxidize organically bound iron. Excess free chlorine residuals (>5 mg/l) could not control the iron bacteria, presumably because the organically bound iron was chlorine nonreactive, and yet remained bioavailable to the bacteria in the attached biofilms. Chlorine dioxide has been used to control these biofilms. It removes the attached bacteria and exposes them to the disinfectant while the iron is being oxidized.

Nonreactions

Perhaps of equal, and sometimes greater, importance are the types of compounds with which chlorine dioxide will not react. Many compounds that will not react with chlorine dioxide will react with chlorine, thereby creating a "chlorine demand" that vastly increases the amount of chlorine that must be added to reach a given level of disinfection.

For example, chlorine will react with ammonia to form mono-, di- and trichloramines. At pH 7-8, approximately 10 parts by weight of chlorine react with each part of ammonia present. Chlorine dioxide will not react with ammonia.





Table 1

Chlorine Dioxide Applications

Industry/Application	Sulfides	Reduced Sulfur	Nitrogen Compound	Cyanides	Phenols	Aldehydes	Amines & Mercaptan	THM Precursor	Pesticides	Algae & Slime	Metals
Chemicals - Agricultural	•	•			•				•		
Chemicals - Industrial Inorganic	•	•	•	•							•
Chemicals - Industrial Organic	•	•	•	•	•	•	•	•			
Chemicals - Miscellaneous	•	•	•		•						
Chemicals - Photographic		•				•					
Electroplating/Electronics	•			•							
Food Processing	•	•			•		•		•		•
Iron and Steel	•	•	•	•	•		•				
Leather Tanning	•	•								•	
Machinery and Metal Products	•			•							
Mining - Coal		•			•						
Mining - Metals		•		•	•						
Paving and Roofing					•						
Petroleum Refining	•	•	•		•		•			•	
Plastics and Synthetics	•			•	•	•					
Pulp and Paper	•		•				•			•	
Rubber Processing		•									
Soil Remediation	•	•				•			•		
Steam and Power Plants	•	•	•					•		•	
Textiles	•	•			•	•		•		•	•
Waste Treatment - Commercial	•	•	•	•	•	•	•	•			
Water - Groundwater Contamination	•				•	•	•		•	•	
Water - Potable	•					•	•		•	•	•
Water - Municipal Wastewater	•						•	•		•	
Wood and Wood Products					•	•		•			





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), as amended. This means that the sodium chlorite sold for this purpose must be registered as a pesticide with the United States Environmental Protection Agency, under a label or labels containing these uses.

Pesticidal uses of chlorine dioxide generated by sodium chlorite are biocidal, disinfecting or sanitizing 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 microbicide in oil recovery operations.

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

5382-42 Technical Sodium Chlorite Dry
5382-45 31% Active Sodium Chlorite Solution
5382-43 Technical Sodium Chlorite Solution 31.25
5382-44 Technical Sodium Chlorite Solution 18.75
21164-6 Akta Klor 25
21164-9 Akta Klor 7.5

Generation of Chlorine Dioxide

Chlorine dioxide can be generated by activating sodium chlorite (NaClO_2) with an oxidizing agent or an acid source.

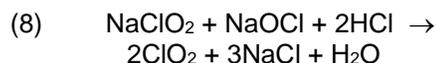
OxyChem produces a dry technical sodium chlorite, nominally 80% active. The most commonly available oxidant is chlorine, which reacts with sodium chlorite as follows (7):



From the above equation, 1.34 lb. of pure sodium chlorite will react with 0.5 lb. of chlorine to produce 1.0 lb. of chlorine dioxide. However, since dry technical sodium chlorite is 80% active, the

reaction takes 1.68 lb. of technical sodium chlorite. Usually, a slight excess of chlorine should be used to ensure that the reaction solution has a pH value between 2-4. This will produce chlorine dioxide more efficiently.

In some applications, a two-fold excess of chlorine can be used for maximum yield of chlorine dioxide. However, for treatment of potable water where the formation of trihalomethanes is a concern, only an amount of chlorine slightly in excess of the theoretical should be used. Alternatively, the addition of a small amount of muriatic acid (HCl) or other mineral acid may be used for pH adjustment. If chlorine is not readily available, chlorine dioxide can also be prepared by mixing sodium hypochlorite bleaching solution with sodium chlorite and an acid (8):



The reaction with hydrochloric acid as shown in the above reaction, produces the most efficient generation of chlorine dioxide. Other inorganic and organic acids may be used, and sulfuric acid produces the highest purity chlorine dioxide. Regardless of the acid used, a slight excess is needed to maintain a pH of 2-4.

Chlorine dioxide is generated by acidifying a solution of sodium chlorite as shown below with hydrochloric acid:



This reaction represents the easiest method of generating chlorine dioxide. It can often be used by simply mixing sodium chlorite into water systems which are slightly acidic (pH 4-5).

Commercial generators are available based on the chemistry described in these equations. The ease of generation of chlorine dioxide in a closed system is shown in Figure 1, using activation by chlorine as an example.

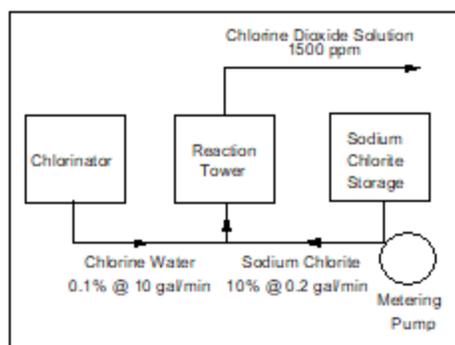


Figure 1. Chlorine Dioxide Generator

A 0.1% Cl_2 solution is fed from the chlorinator at 10 gal/min and mixed with a 10% NaClO_2 solution pumped at 0.2 gal/min into a reaction tower filled with Raschig rings. The resulting product contains about 1500 ppm of chlorine dioxide, which can be introduced in the system for water treatment.

Methods of Analysis

Chlorine Dioxide

Chlorine dioxide solutions can be analyzed by iodometric and amperometric titrations and spectrophotometrically with the standard DPD (N,N-diethyl-*p*-phenylenediamine) method. These methods are described in detail in *Standard Methods for the Examination of Water and Wastewater*.

The use of the iodometric method is questionable, even for the carefully controlled chlorine dioxide standards in research laboratories. For actual samples, which contain a large number of potential interferences, the method is almost certain to produce erroneous results. Any method that determines concentrations by difference is potentially inaccurate and subject to large accumulative errors in terms of both accuracy and precision. Newer, more species-specific methods are better choices.

Because the DPD method determines concentrations by difference, direct measurement by a more reliable and accurate method is needed to determine ClO_2 . Clearly the best choice for measuring ClO_2 is either the

amperometric method or a UV-based method¹. Amperometric systems have excellent utility in laboratories with highly trained personnel. Automated versions have the potential for field use if electrode stability and reliability are established. Ultraviolet spectrophotometry utilizing continuous-flow automated methods has a great potential for accurate and precise measurements, with the added advantage of ease of operation and high sample throughput². Flow injection analysis (FIA) and continuous flow methods exhibit superior selectivity.

Chlorite and Chlorate Ions

The iodometric and amperometric methods are used for indirect determination of ClO_2^- at high concentrations and cannot be recommended for drinking water measurements. The DPD method cannot be recommended for ClO_2^- because it is unreliable.

The iodometric sequential methods³ appear to be useful for samples containing >1 mg/l ClO_2^- or ClO_3^- , with good precision and accuracy. For samples containing <1 mg/l ClO_2^- or ClO_3^- , these methods require considerable operator skill and experience to obtain good precision and accuracy and should be field tested with other methods using both high and low ratios of ClO_3^- to ClO_2^- . They should be used with caution on low-level samples of drinking water or wastewater. Direct methods requiring less specialized skills are preferred.

A highly precise, automated Flow Injection Analysis (FIA) method has been developed for low-level ClO_3^- using various masking agents such as glycine, oxalic acid, malonic acid, or nitrite ion to initially remove other interfering oxyhalogen species. FIA and ion chromatography have been used to directly determine low-level ClO_3^- concentrations.

Safety and Handling of Sodium Chlorite

The following summary of health and safety information is not intended to be complete. For complete information, read the current Safety Data Sheet (SDS). To obtain an SDS, contact the OxyChem Technical Service Department.



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Toxicological Properties

Sodium chlorite is toxic by ingestion. Sodium chlorite may cause anemia by oral exposure and has low toxicity by dermal exposure. OxyChem sodium chlorite has an oral LD₅₀ (rat) of 165 mg/kg. Sodium chlorite has 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 handling sodium chlorite solutions, chemical goggles, face shield, neoprene gloves, apron, and chemically resistant boots should be worn. 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 re-use.

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 cough 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. The dry flakes will absorb moisture from the environment, resulting in clumping of the material. 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 ultraviolet 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.





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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 re

Sodium chlorite, dry, is a fire or explosion hazard if contaminated with combustible material. Clean up in a manner to avoid contamination. Keep product wet by sprinkling water on spill. Spilled material should be picked up, by using a clean 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 solutions 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-combustible 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

Dispose in accordance with all applicable regulations. Do not put product, spilled product, or filled or partially filled containers into the trash or waste compactor. Contact with incompatible materials could cause a reaction and fire. Contact OxyChem Technical Service to obtain neutralization instructions. Sodium chlorite is toxic to fish and aquatic organisms. Keep out of water supplies and sewers. If sodium chlorite is spilled or becomes a waste, it must be disposed of in accordance with local, state, and Federal regulations by a NPDES permitted out-fall or in a permitted hazardous waste treatment, storage, and disposal facility.

Shipping Information

Technical Sodium Chlorite is available in 100-lb drums. 31% Active Solution is available in tank trucks and railcars. Technical Sodium Chlorite Solution 31.25 is available in railcars, tank trucks, 330-gallon non-returnable totes, and 55-gallon drums.

For Further Information

More detailed information on sodium chlorite is available on request through:

OxyChem Technical Service Department
6200 S. Ridge Rd.
Wichita, Kansas 67215
800-733-1165 option #1
OxyChem_Tech_Service@oxy.com





References

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