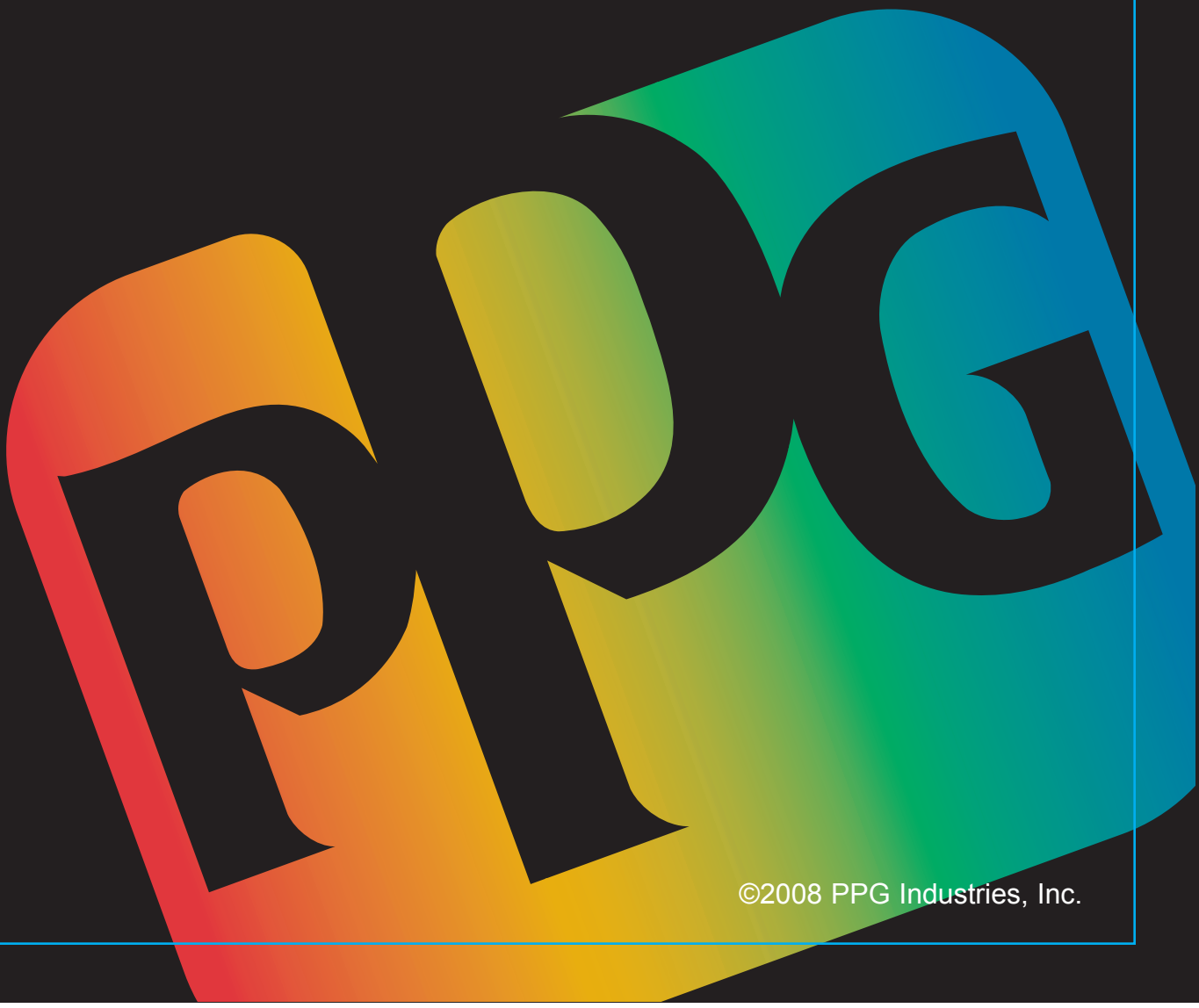


# NaOH

## Caustic Soda

PPG Industries, Inc.



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**DANGER!**

### **Caustic Soda Can Cause Severe Burns to Skin and Eyes**

Caustic soda (also called lye or sodium hydroxide) attacks the skin and eyes rapidly. Even a small quantity of a dilute solution can severely injure the eyes or cause blindness. Overexposure to caustic soda by way of skin burns or swallowing can cause death.

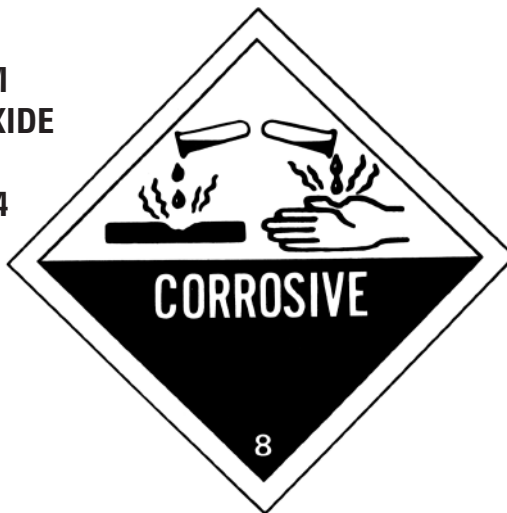
Persons working with caustic soda should wear protective clothing and close-fitting goggles at all times.

Caustic soda is a reactive chemical and can react with certain other chemicals and metals to produce explosions.

Read Chapter 3 for more information on the hazards of caustic soda, on protective devices and on first aid.

In case of emergency, call PPG Industries Emergency Response Center at (412) 434-4515. This telephone is answered 24 hours a day.

**SODIUM  
HYDROXIDE  
LIQUID  
UN 1824**



**SODIUM  
HYDROXIDE  
SOLID  
UN1823**

EMERGENCY TELEPHONE NUMBER

PPG INDUSTRIES, INC. **(412) 434-4515**

In Canada call CANUTEC (613) 996-6666

NaOH



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NaOH

©PELS is the registered trademark of PPG Industries for its brand of beaded caustic soda.

# Introduction



**C**autistic soda is a hazardous, reactive chemical. Before persons work with caustic soda, they should be instructed in safe handling practices and first aid, and should wear the recommended protective clothing and equipment (see chapter 3).

PPG Industries, one of the world's largest producers and suppliers of caustic soda, has three caustic plants strategically located in the United States and Canada. These plants serve a large and highly diversified group of consumers located throughout North America and worldwide. In addition, PPG is the only domestic producer of beaded caustic soda .

The combination of high-volume caustic soda production plus orientation as a supplier of caustic soda to North America enables PPG to back up its product with a coordinated system of services important to all caustic soda consumers.

- **A network of bulk terminals** for shipment of liquid caustic soda is operated in North America. Overnight service to many customers is made possible by this system of bulk terminals.
- **Versatile shipping capabilities** include tank car, tank truck, barge and ocean-going tanker transportation of caustic soda. PPG owns hundreds of tank cars designed and used exclusively for caustic soda solution service.

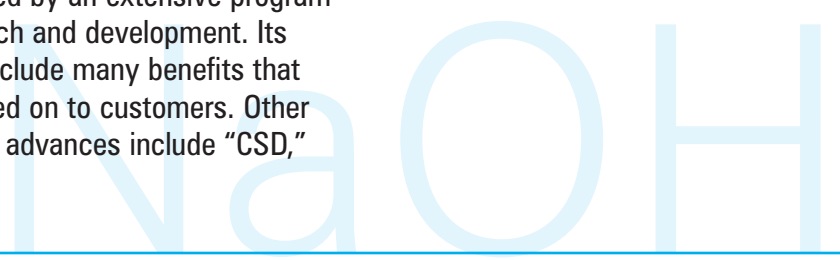
Other PPG rail cars and truck trailers are available for delivery of PELS® caustic soda beads. PPG barges serve customers on rivers and inland waterways, while coastal terminals are supplied by ocean-going tankers.

- **Stocks of PELS** caustic soda beads are maintained at strategically located distribution points throughout the nation.
- **PPG Industries sales representatives**, thoroughly trained to serve caustic soda customers, operate from offices located in major cities throughout the United States, Puerto Rico and Canada.
- **PPG engineers** provide assistance to customers by offering a full range of technical information and consultation. Their services include: sharing information on systems for unloading, handling and storing various caustic soda forms; assistance in startups and trouble-shooting; advice on form and grade selection.
- **Other activities** encompass technical service engineering assistance in selecting and installing handling equipment and protective devices as well as help with safety training programs. These include providing safety wall charts, booklets and other literature. PPG's position as a front-running caustic producer is maintained by an extensive program of research and development. Its results include many benefits that are passed on to customers. Other technical advances include "CSD,"

closed system delivery of bulk, dry caustic soda in pressure-differential hopper cars and truck trailers, and PELS caustic soda beads.

PPG is positioned to meet its customers' needs for material—and service.

A network of caustic soda distribution points assures quick delivery.



# Shipping Points



## SHIPPING POINTS FOR CAUSTIC SODA SOLUTIONS:

### Tank Cars and Tank Trucks

- Illinois: Lemont
- Louisiana: Lake Charles
- Missouri: St. Louis
- New Jersey: Bayonne
- West Virginia: Natrium
- Canada: Beauharnois, Quebec
- Puerto Rico: Guayanilla (tank trucks only)

### Barges

- Louisiana: Lake Charles
- West Virginia: Natrium

### Ocean-going Tankers

- Louisiana: Lake Charles



- Tank Cars and Tank Trucks
- Barges
- \* Ocean-going Tankers

NaOH

# Purchasing Information



**C**autistic soda is a hazardous, reactive chemical. Before persons work with caustic soda, they should be instructed in safe handling practices and first aid, and should wear the recommended protective clothing and equipment (see chapter 3).

## GRADES AND FORMS

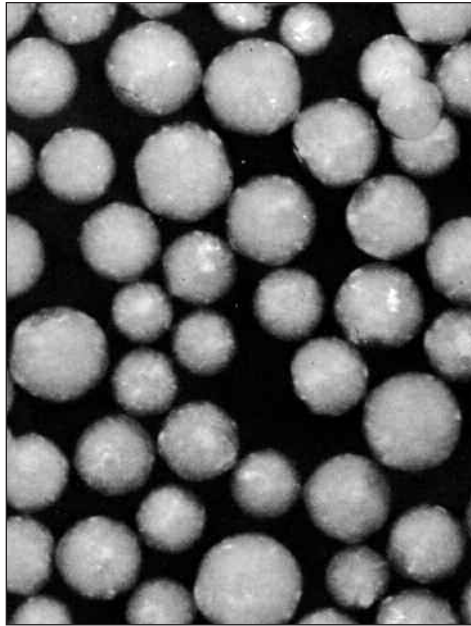
To meet the needs of various industrial processes, the Chemicals Group of PPG Industries produces caustic soda in a wide range of grades and forms. Caustic soda solutions are available in these grades:

- standard
- purified
- diaphragm
- membrane
- mercury cell

The most common commercial form is the solution containing 50% actual sodium hydroxide by weight. A 25% form is also available from the Natrium plant. PPG Industries also produces the more concentrated solution form, approximately 73% actual sodium hydroxide by weight.



Liquid caustic soda available in 25%, 50% and 73% solutions.



PELS caustic soda beads show little or no dust.

PPG Industries manufactures anhydrous caustic soda in the modern beaded form bearing the trademark *PELS*. The beads come in one optimum size averaging 0.7 millimeter in diameter. Their size uniformity is an advantage for repacking and compounding. Their one optimum size eliminates the need to store numerous grades of anhydrous caustic soda.

## SELECTING THE OPTIMUM GRADE AND FORM

Among the factors in selecting the most economical grade and form of caustic soda for a particular application, besides purchase price, are the costs of: transportation, unloading, handling within plant, preparing caustic soda for use, and the investment in equipment for unloading, storage, handling and preparation.

Representatives of PPG Industries will gladly cooperate in determining the most economical and best-suited grade and form for any operation.

## CAUSTIC SODA SOLUTIONS

### Shipping

Caustic soda solutions are shipped from PPG manufacturing plants as 50 percent solutions and 73 percent solutions by weight. The company's nationwide network of terminals stocks 50 percent solutions in strategic geographic locations for prompt delivery to many parts of the United States.

PPG Industries ships 50 percent caustic soda solution in ocean-going tankers to terminals on the east and west coasts for domestic users as well as for export. The company's large fleet of barges carries caustic soda solutions to inland and coastal terminals and customers' docks.

PPG engineers will assist companies in evaluating new unloading facilities for tank cars, tank trucks or barges.



Loading caustic soda solution into tank car.



# Purchasing Information



Loading caustic soda solution into an ocean-going tanker at PPG plant in Lake Charles, Louisiana.

To prevent metallic contamination and maintain the high purity of caustic soda shipments, PPG lines all cargo tanks of tank cars and barges.

## Tank Cars

PPG maintains a fleet of many hundreds of tank cars for caustic soda service only. They are specifically designed for the unloading of caustic soda solutions. Every PPG tank car is equipped for either top or bottom unloading.

PPG tank cars are well insulated, so that freezing is unlikely except under non-routine conditions such as pro-



Caustic soda tank car.

longed transit delays. The cars are equipped with channel-type heaters if heating is required.

Shipments are usually made in tank cars with nominal capacities of 10 and 16 thousand gallons.

## Typical Weights of Tank Car Contents

	Nominal Capacity, Gallons	
	10,000	16,000
Liquid basis		
50 percent	128,000 lb	194,000 lb
73 percent	142,000 lb	200,000 lb
Anhydrous basis		
50 percent	33 tons	50 tons
73 percent	52 tons	73 tons

## Dimensions of Tank Cars

	10,000-gallon	16,000-gallon
Length	41' 2"	43' 3 3/8"
Height		
Empty	14' 7 3/4"	14' 10 5/8"
Loaded	14' 5 3/4"	14' 8 5/8"
Width	10' 7 1/2"	10' 0"

## Tank Trucks

Tank trucks provided by common carriers for the shipment of caustic soda solutions must conform to applicable specifications. PPG will not load into low-pressure trailers. Tanks are generally made of stainless steel. Maximum permissible loads may be limited by regulations of the different states.

The capacities of tank trucks in caustic soda service have not been standardized, but vary between 2,000



Caustic soda tank truck at loading platform.

to 4,000 gallons. For shipments of 50 percent caustic soda solutions, the corresponding weights would range from 25,000 to 50,000 pounds of liquid. On an anhydrous basis, these shipping weights would range from 6 to 12 tons.



PPG barge dedicated to shipping caustic soda solution.

## Barges

The barges in the fleet of PPG Industries are built specifically for transporting caustic soda solutions. They are equipped with diesel unloading pumps.

Barge capacities range from a minimum of 1,200 liquid tons or approximately 200,000 gallons up to a nominal 3,000 liquid tons or approximately 475,000 gallons. Since almost all barge shipments consist of 50 percent caustic liquor, the actual dry caustic soda content ranges from 500 to 1500 tons.

# Purchasing Information



## Shipping Points for Caustic Soda Solutions

See list with map on pages 4 and 5.

## Billing Procedure

Caustic soda solutions are billed on a basis that is standard in the United States. However, this basis is rather complicated because of the way it originated. Since the days when alkalis were first manufactured, caustic soda (NaOH) and soda ash (Na<sub>2</sub>CO<sub>3</sub>) were compared on the basis of their sodium oxide (Na<sub>2</sub>O) content. The anhydrous forms of caustic soda in those days were about 98 percent pure. According to the molecular weights of NaOH and Na<sub>2</sub>O, 100 pounds of pure NaOH are calculated to contain 77.48 pounds of Na<sub>2</sub>O. But the 98 percent purity factor reduces this value to 76 pounds. Today, liquid caustic soda is still sold as "NaOH on a 76 percent Na<sub>2</sub>O basis."

Another factor in billing is that the Na<sub>2</sub>O content is determined by a laboratory method which includes the Na<sub>2</sub>O in both NaOH and Na<sub>2</sub>CO<sub>3</sub>. The latter is present as a fractional percentage.

For example, assume that a shipment of nominal 50 percent caustic soda solution contains the following percentages by weight of NaOH and Na<sub>2</sub>CO<sub>3</sub>:

% Actual NaOH = 50.00%  
 % Actual Na<sub>2</sub>CO<sub>3</sub> = 0.16%

Based on their relative molecular weights, NaOH contains 77.48% Na<sub>2</sub>O while Na<sub>2</sub>CO<sub>3</sub> contains 58.48%.

Therefore, the % Na<sub>2</sub>O content of the NaOH and Na<sub>2</sub>CO<sub>3</sub> in the shipment is as follows:

$$\begin{aligned} \% \text{ Na}_2\text{O in NaOH} \\ = 0.16\% \times 0.5848 = 0.09\% \end{aligned}$$

$$\begin{aligned} \text{Therefore, the total \% Na}_2\text{O is:} \\ 38.74\% + 0.09\% = 38.83\% \text{ Na}_2\text{O} \end{aligned}$$

The billing analysis (% NaOH on a 76% Na<sub>2</sub>O basis) is calculated as follows:

$$\frac{\text{Total \% Na}_2\text{O} \times 100\%}{76\%}$$

or,

$$\frac{38.83 \times 100}{76\%}$$

$$= 51.09\% \text{ NaOH (76\% Na}_2\text{O basis)}$$

The pounds of NaOH (76% Na<sub>2</sub>O basis) used for invoicing are calculated as follows:

Pounds Caustic Soda Solution

$$\frac{\% \text{ NaOH (76\% Na}_2\text{O basis)}}{100}$$

$$= \text{Pounds NaOH (76\% Na}_2\text{O basis)}$$

## PELS CAUSTIC SODA BEADS

PPG Industries supplies anhydrous caustic soda in the beaded form bearing the trademark *PELS*. Drums and bags are designed to be moisture-resistant because anhydrous caustic soda readily absorbs moisture from atmospheric air.

## Bulk Shipments

*PELS* caustic soda beads are shipped in rail cars and truck trailers. The recommended method of handling is the "CSD" closed system delivery method developed by PPG Industries. It utilizes a closed-loop, pneumatic system for pressure conveying.

PPG maintains its own fleet of pressure differential trailers that can make shipments up to approximately 21 tons. These trailers carry equipment for drying the pneumatic conveying air so that the receiving system does not need to have dryers.

PPG Industries owns the pressure differential rail cars used for CSD shipments. The rail cars are lined with a caustic soda compatible protective coating. These cars have a capacity of 3000 to 4000 cubic feet, sufficient for shipments up to 100 tons.

More detailed information on "CSD" closed system delivery appears on page 24 in the chapter on *PELS* Caustic Soda Beads Unloading and Handling.

## Bag Shipments

PPG Industries can supply *PELS* caustic soda beads on sturdy pallets protected by a shrink-wrapped polyethylene film. This method of shipment can reduce damage in transit, permits outdoor storage, facilitates stacking pallet loads and protects the bags from chemicals, moisture and dirt.

The bag has six walls including two moisture barriers: a heat-sealed polyethylene liner and a Valeron®



# Purchasing Information



The entire pallet load, (foreground) 1 1/2 tons, consisting of sixty 50-pound bags, is protected by a shrink-wrapped polyethylene film. PELS Caustic Soda beads are also packaged in steel and fiber drums (background).

cross-laminated polyethylene ply (see photo). This bag exceeds Department of Transportation drop test standards for multiwall packages. Accelerated tests in a "jungle room" showed that moisture vapor did not reach the caustic soda. Drop tests performed after the "jungle room" exposure showed virtually the same results as before.

In comparison with older types of pillow-shaped bags, the rectangular bags have better sag resistance and fewer loose edges that provide easier handling. The bags occupy less space than older types of bags and permit more compact unitizing and more stable pallet loads.

Rectangular bags take up only 73% of the space occupied by round drums.

## Drum Shipments

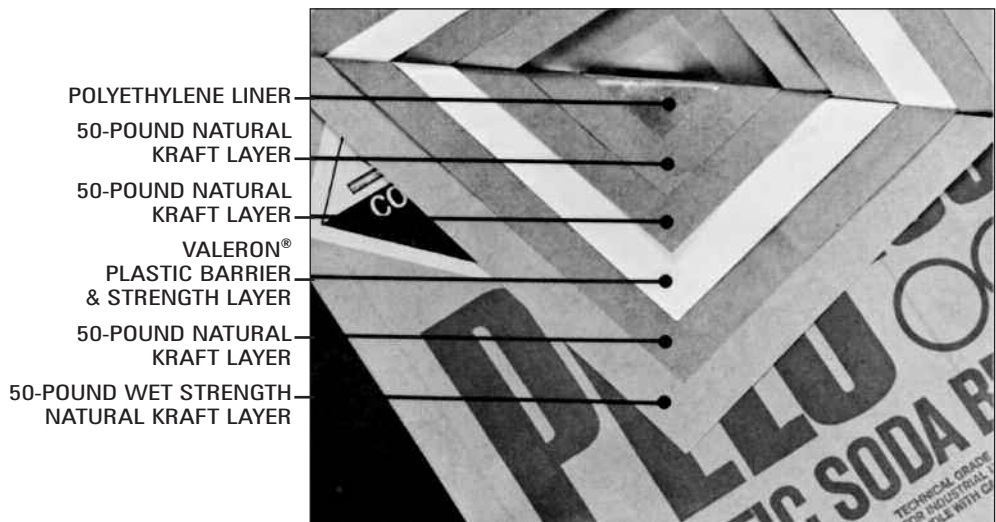
The common type of full open head steel drum has a flowed in or a rubber gasket to make the container moisture-resistant.

### PELS Caustic Soda

Container	Net Weight, pounds
Steel drums	100, 500
Bags	50, 2000
Fiber drums	500

### Shipping Points

For PELS Caustic Soda  
 Louisiana: Lake Charles  
 West Virginia: Natrium



Super-strong bag has six walls including two moisture barriers.



Drumming PELS caustic soda beads.

# Protective Devices and First Aid



**O**verconfidence can be responsible for serious personal injuries from caustic soda, especially to persons who have worked around caustic soda for many years without accident. Caustic solutions or particles in contact with the skin can cause severe burns. Even a small quantity in the eye may damage vision permanently.

Persons working around caustic soda should wear protective clothing and close-fitting monogoggles at all times.

## Prevent Accidents

The prevention of accidents is the best protection against injury. Equipment should be checked on a regular maintenance schedule to prevent leaks and spraying of caustic soda liquor. Personnel should be trained in proper handling procedures.

Liquid spills should be cleaned up right away. They make a floor slippery, and a person falling into a puddle of caustic liquor can suffer extensive body burns.

## Treat Immediately

**Speed of treatment in case of contact with caustic soda is extremely important.** Caustic soda reacts chemically with body tissue and will cause severe burns unless it is washed off the skin with plenty of water immediately. Clothing should be removed. Refer to Material Safety Data Sheet for caustic soda.

A deceptive aspect of caustic soda is that a burning sensation is not always immediately experienced on contact with the skin. Serious damage may therefore happen before a person is aware of contact with caustic soda. If contact with caustic soda is suspected, flush with water right away even though there is no burning sensation.



*If caustic soda gets in the eye, flush eye immediately with large quantities of water.*

## FIRST AID

All persons who handle caustic soda should be familiar with proper first aid procedures.

Fast action is imperative because caustic soda attacks the skin and eye tissues rapidly. Delay greatly increases the severity of a caustic burn; prompt and thorough treatment can reduce the danger of serious permanent damage. **Get the injured person to an eye wash or spray shower immediately! Every second is critical. Be sure that medical attention is obtained as soon as possible if caustic soda has contacted an eye, also if skin contact has resulted in burns, reddening or excessive irritation.**

## Eyes

Eyes are particularly sensitive to caustic soda. Even tiny particles of caustic soda dust, or a small quantity of dilute solution, can injure the eye or result in blindness or permanent eye damage.

**If caustic soda gets in the eye, flushing the eye immediately with large quantities of water will do more good than the best of medical services can provide later on.**

**Continue washing for at least 15 minutes. Call a physician.**

To make sure that water contacts all surface tissues of the eye and lid, hold the eyelids apart while flushing. The best way to irrigate the under surfaces of the upper eyelid is to raise the eyelid and roll it back. Prolonged irrigation with plenty of water—and only water—is the proper treatment for eyes. **Neutralizing solutions must not be used in the eyes.** Chemical neutralization generates heat and may introduce another burn hazard. Diluting caustic soda also generates heat; so use plenty of water to conduct the heat away.

**Call a physician**, preferably an eye specialist, as soon as possible. **Continue eye irrigation until the physician arrives.**

## Eye-Washing Fountains and Spray Showers

The operation of eye wash fountains and safety showers should be checked

## Protective Devices and First Aid



*Eyewash fountain.*

before wash begins. Spray showers and eye-washing fountains or a bubbler fountain should be located in the immediate working area where they can be reached **in seconds**.

Out-of-doors, a hose that is not under high pressure can serve temporarily in place of a shower or eye-washing fountain.

Prominent markings to make the location of safety showers and fountains distinctly visible by means of a sign or conspicuous background color are recommended. Showers and fountains should be tested daily and kept clean and in perfect working order at all times.

### Protective Clothing

All persons working around caustic soda should wear protective clothing as well as **close-fitting safety goggles**. Do not wear contact lenses when working around caustic soda. Face shields may be worn in addition to—but not instead of—goggles. Gloves, boots aprons, and other clothing made of rubber or rubber-covered cloth give good protection because rubber is resistant to caustic soda.

Clothing made of polyvinyl chloride also gives good protection. Under the protective clothing, wear cotton clothing rather than woolens, because animal fibers such as wool are rapidly

destroyed by caustic, whereas cotton is more resistant. Leather, like wool and animal tissues, is also attacked by caustic. Wear shoes, boots or over-shoes made of rubber. Also, wear a plastic safety hard hat. The wide brim style provides additional protection from overhead drips or leaks. Set the front of hat to slope down over eyes to provide more cover for the eyes. When boots are worn, trouser legs should be on the outside to reduce the possibility that caustic soda might enter the top opening. Wear a long-sleeved shirt and button the collar.



*Proper protective clothing reduces chances of injury.*

shirt or coveralls should fit snug at neck and wrists.

### Respirator

Persons working where caustic soda dust or mist is present should wear NIOSH/MSHA-approved dust-type respirators. Inhalation of dust or mist will irritate the respiratory tract.

The OSHA permissible exposure limit (PEL) for caustic soda is a ceiling value of 2 milligrams per cubic meter of air. Work areas where caustic soda dust or mist is present should be well ventilated.

### Skin

Flush skin immediately and continuously with plenty of water for at least

15 minutes—longer if a soapy feeling persists, which indicates that some caustic soda is still present. **Do not sponge or rub or use small amounts of water.** Large quantities of water are necessary to carry away the heat generated by diluting caustic soda. Do not touch your eyes with your hands or fingers—they might be contaminated with caustic soda.

Caustic soda burns can be most deceiving. A grave danger lies in stopping the flushing with water too soon. What may first appear to be a superficial skin irritation can become a serious burn that may take a long time to heal. The greater the amount and concentration of the caustic soda and its temperature, the more imperative it is to get to a source of water immediately.

Under the safety shower, remove clothing that has been in contact with caustic. Continue flushing while removing clothing. Do not remove safety goggles unless caustic soda has been washed off completely under the shower. Anyone helping a person with caustic soda on their body or clothing should wear safety goggles to make sure that their own eyes don't get splashed with water contaminated by caustic soda.

A less serious condition, stinging due to caustic soda mist or dust on exposed skin, can be stopped by rinsing with a 10% solution of ammonium chloride. Diluted vinegar may also be used to stop stinging. Do not use neutralizing solutions in the eye.



## Protective Devices and First Aid

### Swallowing

If a person swallows caustic soda, give plenty of water to drink. Afterwards, citrus juices or weak vinegar solutions may also be given. **Do not induce vomiting** because passing the caustic soda through the food pipe, throat and mouth a second time will result in more damage to these tissues. Caustic soda causes severe damage to mucous membranes. If a person has swallowed caustic soda, call a physician at once. **Don't give anything by mouth to an unconscious person.**

### Shock

If a patient suffers from shock, place them on their back and keep them warm until a doctor arrives.

NaOH



# Caustic Soda Liquor Handling and Storage Equipment



**C**austic soda is a hazardous, reactive chemical. Before persons work with caustic soda, they should be instructed in safe handling practices and first aid, and should wear the recommended protective clothing and equipment (see chapter 3).

## MATERIALS OF CONSTRUCTION

Iron and steel are the usual materials of construction for equipment handling 50 percent solutions of caustic soda below 140° F. Nickel, nickel alloys and stainless steel are required at higher temperatures, for higher concentrations, or for operations where iron pickup of a few parts per million cannot be tolerated.

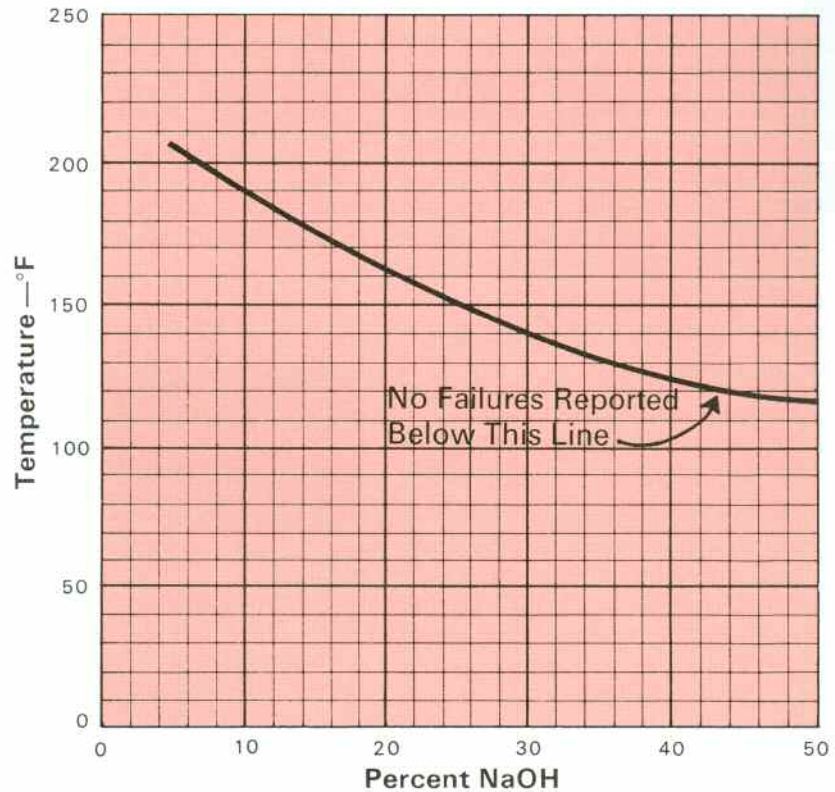
To prevent metallic contamination in transit, more than one type of liner is used on the interior of tank cars and barge tanks. Tank trucks are ordinarily constructed of stainless steel but their interiors may also be lined.

Designers of equipment for handling and storing caustic soda may want to evaluate other materials and new products. PPG engineers will share information on request regarding the selection of construction materials for individual applications.

### Stress-Corrosion Failure

Commonly called “caustic embrittlement,” stress-corrosion failure occurs at areas of mechanical stress or where fabricating operations produce high residual stress such as at bends in piping and at welds.

Stress-Corrosion Cracking of Mild Steel



Generally accepted practices suggest temperatures to 140°F.

(National Association of Corrosion Engineers Technical Practices Committee Report, Publication 51-3)

### Corrosion Rates in 50% NaOH

Metal	Corrosion Rate, Mpy		
	At 100°F <sup>a</sup>	At 135°F <sup>b</sup>	At 131 to 167°F <sup>c</sup>
Titanium	<0.1	0.5	....
Zirconium	<0.1	<0.1	....
Nickel	<0.1	<0.1	<0.1
Monel <sup>d</sup> alloy 400	<0.1	<0.1	<0.1
Inconel <sup>d</sup> alloy 600	<0.1	<0.1	<0.1
Ampco <sup>e</sup> 8	....	1	....
Mild steel	0.7	5	8
Copper-nickel (70-30)	....	....	<0.1
18-8 stainless steel	....	....	0.1
Ni-Resist <sup>f</sup> Type 1	....	....	2
Cast iron	....	....	10.5

<sup>a</sup>Duration of test: 162 days. <sup>b</sup>Duration of test: 135 days. <sup>c</sup>Duration of test: 30 days.

<sup>d</sup>Trademark of The International Nickel Co., Inc. <sup>e</sup>Trademark of Ampco Metal, Inc.

<sup>f</sup>Trademark of Thomas Foundries, Inc.



## Caustic Soda Liquor Handling and Storage Equipment



As shown in the graph, mild steel is subject to stress-corrosion failure under conditions that depend upon temperature and also upon concentration. These conditions would exist in the area above the curve in the graph. To prevent stress-corrosion failure, temperatures of 50 percent caustic soda solutions should not be permitted to go above 140° F.

### MATERIALS ATTACKED BY CAUSTIC SODA

Certain metals, such as aluminum, magnesium, zinc, tin, chromium, brass, and bronzes made with zinc or tin are attacked by caustic soda. They should be avoided as parts of equipment; for example, brass bearings. Also items such as safety hats, buckets, drums and ladders made of aluminum should not be used with caustic soda.

Since galvanizing is done with zinc—which is attacked by caustic soda—keep the liquor away from galvanized iron surfaces. The reaction of caustic soda with zinc is vigorous and—under some conditions—may be dangerous because hydrogen is generated and may introduce an explosion hazard.

Silica-containing materials such as glass, brick and tile are attacked by caustic liquor. The action is slow and will at first only contaminate the caustic with silica, but failure of the material will eventually follow.

Copper can seriously contaminate caustic soda even though it may not be severely attacked. Caustic handling equipment made with copper or copper alloys can be harmful to certain manufacturing processes, such as the production of hypochlorite bleaches, because of their great sensitivity to copper contamination.

### PIPE LINES

Wrought iron or mild steel is suitable for pipelines and fittings to convey caustic soda solutions below 140°F; nickel is customary for hotter solutions. Monel alloy or stainless steel may be used under certain conditions. Polypropylene-lined steel pipe has been used with solutions up to 225°F. Outdoor pipes should be insulated and, if air temperatures fall below 65°F, electrically-traced. Welded or flanged joints are preferable to screwed fittings.

Many different types of plastics and elastomers may be used with caustic liquor but the upper temperature limit, of course, is different for each plastic. Do not use rubber gaskets. **Materials whose capabilities for caustic service have not been established by experience should be adequately tested in advance.**

Pipe lines should slope so as to be self-draining. Caustic liquor left in pipe lines has a passivating effect and retards corrosion; however, it should not be left in pipe lines if temperatures may reach the freezing point of the solution. Water should be available for washing out sections of line where

there is danger that caustic liquor might drip and contact personnel.

New piping installations should **always** be tested with water for leaks before caustic liquor is introduced into the piping. Plastic covers on flanged joints, also guards over pump packing glands, prevent spraying of caustic liquor if leaks develop.

Process piping under 1-inch diameter is not recommended. Larger sizes permit greater flow-through and minimize the possibility of plugging or freezing.

### Unloading Lines

Tank car unloading lines are usually 2-inch diameter pipe because this is the standard fitting size on caustic soda tank cars.

Flexible hoses are preferred for connecting a tank car to the unloading pipe line because the car rises during unloading. Spiral-wire-wound hoses made of alkali-resistant material are suitable. These hoses should have clamped "combination nipple" ends made of cadmium-plated malleable iron. Hoses may also be made of stainless steel.

### PUMPS

For transferring caustic liquor from tank car to storage or from storage to point of use, centrifugal pumps are recommended. All-iron construction is generally suitable, although nickel and nickel-cast-iron pumps give longer service life. Above 140°F, a nickel or nickel alloy pump must be used. Brass

## Caustic Soda Liquor Handling and Storage Equipment



fittings and bearings should be avoided, especially at high temperatures or concentrations of 50% or above. To prevent leakage at the pump shaft, deep stuffing glands with high-grade TFE impregnated carbon fiber packing are preferred to mechanical seals. Mechanical seals are only recommended after consultation with the seal manufacturer.

### VALVES

Use Teflon® brand fluoropolymer resin-lined plug valves, which are gear-operated above the 4" size. Iron or steel plug valves of the lubricated type give good service for normal use. For high-temperature use, nickel-cast-iron plug valves or nickel gate valves with deep packing glands should be used. Stainless steel can also be used in some cases.

Other types of valves commonly used include non-lubricated ball valves and gate valves made entirely of iron or steel.

Brass valves should be avoided, especially at high temperatures or concentrations of 50% or above, because they are corroded by caustic soda and will leak. They can also introduce copper contamination that can be harmful to certain manufacturing processes.

### METERS

There are several types and designs of highly accurate meters with excellent reproducibility that are made of mate-

rials resistant to caustic soda solutions. **Meters with brass parts, such as used in water meters, would be corroded by caustic soda.** Suitable meters are available with attachments to compensate for changes of specific gravity with temperature where this degree of accuracy is required.

The selection includes automatic and semi-automatic meters for continuous or batch operations. They range in capacity from 0.1 gallon per minute to hundreds of gallons per minute.

PPG engineers will share information on request regarding the type and size of meter required for any installation.

### STORAGE TANKS

The most popular construction material for storage tanks holding caustic soda solutions is mild steel because of its lower cost and satisfactory performance under most conditions.

The 50% solution should be stored at 75 to 100°F. Below 75°F, viscosity increases rapidly and pumping becomes difficult. Above 140°F, iron pickup increases and stress-corrosion cracking may become a problem.

#### Preventing Caustic Attack and Iron Pickup

Even localized high temperature can accelerate caustic attack. For example, heating coils carrying steam will have a surface temperature of at least 212°F. Coils made of nickel or nickel alloys such as Monel and Inconel are highly resistant to attack even at tem-

peratures far above 290°F, the boiling point of a 50% solution of caustic soda. Coils located too near the side of the tank are known to have caused stress-cracking as well as accelerated corrosion of the tank walls. Piping and storage systems should be checked throughout to make sure that local overheating will not occur.

Both metallic contamination and stress-corrosion can be eliminated by a special coating applied to the inside of the tank, such as epoxy-phenolic (Carboline Plasite 9570) or novalac-epoxy (PPG Amercoat 253). It should be applied by licensed applicators only.

Areas of unrelieved high stress, such as welds, are susceptible to caustic attack. Many tanks can be completely stress-relieved by the fabricator; the cost may be considered as insurance against failure. Many welds made in the field have been locally stress-relieved with satisfactory results.

#### Preventing Freezing

The storage temperature of 50% caustic soda solutions should not go below 75°F. At lower ambient temperatures, tanks should be insulated. If these solutions are stored out-of-doors in cold climates, the tanks should be insulated and heated. Steam coils made of nickel submerged in the solutions are the usual method of heating. A small steam or hot water heat exchanger and circulating pump can also be used.

If storage capacity is available, dilution is an effective way to avoid freezing

# Caustic Soda Liquor Handling and Storage Equipment



problems. For example, a 20% solution freezes at approximately—17°F.

## Tank Capacity and Strength

Storage capacity should exceed the volume of the largest shipment planned by at least 50%. A stronger tank is needed than for water because the weight of a 50% caustic soda solution is around 1½ times the weight of water or approximately 12¾ pounds per gallon.

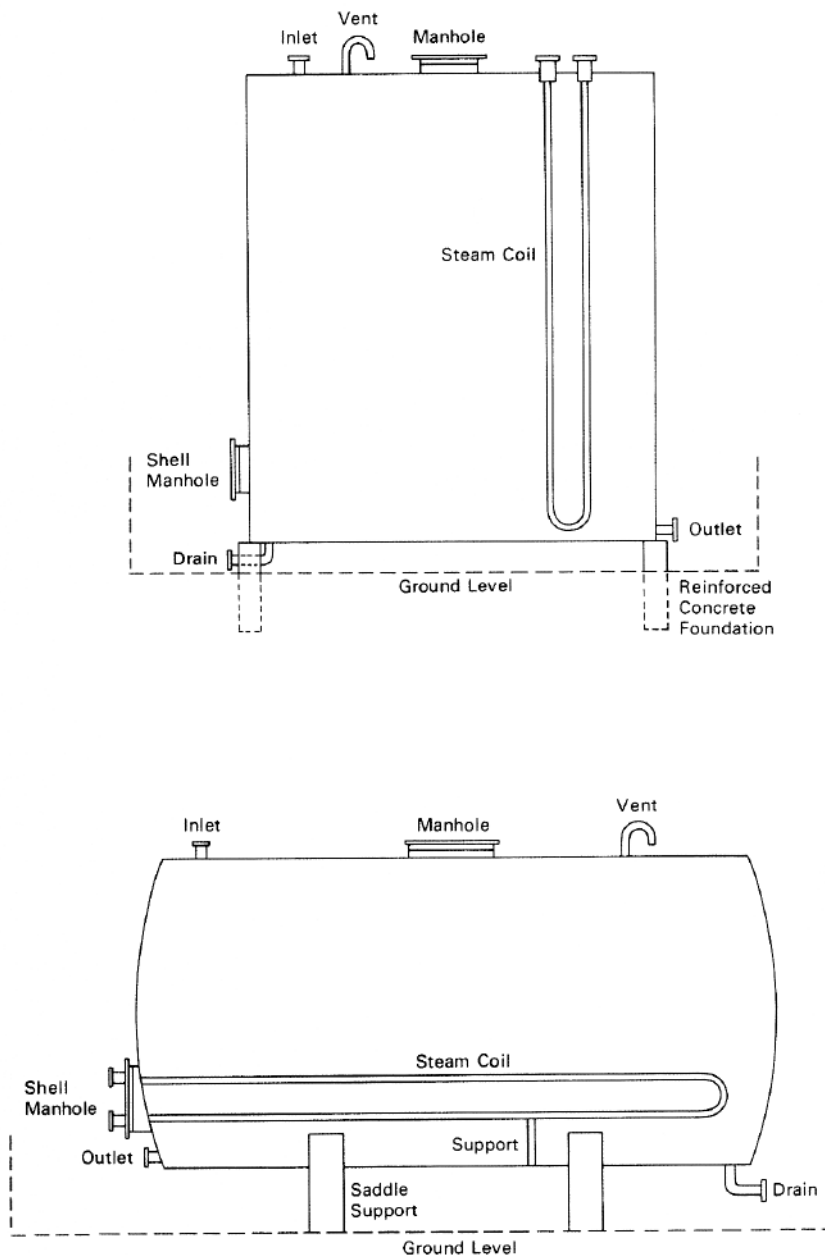
## Curbings and Spills

As a safety measure in case of spills, curbings should be erected around a tank. These structures permit recovery of spilled caustic liquor in some cases; in others, they reduce the hazard of disposing of spills. Regulations should be checked to determine if there are any containment requirements. When most of a spill has been removed, the rest should be cleaned up with lots of water. Final traces of caustic soda can be neutralized with dilute acid or sodium bicarbonate solutions.

Do not dump caustic soda solution into streams or waterways.

Disposal of caustic soda solution should be done in accordance with all applicable laws and regulations.

Typical Storage Tanks for 50-Percent Caustic Soda Solutions



NaOH

## Caustic Soda Liquor Unloading and Handling



**C**austic soda is a hazardous, reactive chemical. Before persons work with caustic soda, they should be instructed in safe handling practices and first aid, and should wear the recommended protective clothing and equipment (see chapter 3).

This chapter describes the unloading of tank cars and the dilution of 73 percent caustic soda solutions.

When requested, PPG will assist companies in evaluating and designing new unloading and handling systems for bulk caustic soda in solution forms.

### CAUSTIC SODA SOLUTIONS

Although the procedures described in this section refer to unloading tank cars, the principles apply in general to tank trucks and barges. On request, PPG will share information on procedures for unloading and handling, since no two installations are alike.

Procedures for unloading 50 or 73 percent caustic soda solutions are the same up to the point where the flexible connection from the tank car ends. From there on they differ because the 73 percent solution is usually diluted before storage.

### PREPARING TANK CARS FOR UNLOADING

The suggestions that follow are based on procedures developed over the years. While certain steps appear

obvious, the procedure would not be complete without them. A detailed listing of steps is helpful from the safety standpoint, too.

### Placing Car for Unloading

1. After the car is properly spotted, set handbrake and block wheels.
2. Caution signs must be so placed on the track of the car as to give necessary warning to persons approaching the car from open end or ends of siding and must be left up until after the car is unloaded and disconnected from discharge connection. Signs must be of metal or other suitable material, at least 12 by 15 inches in size and bear the words "STOP—TANK CAR CONNECTED," the word "STOP" being in letters at least 4 inches high and the other words in letters at least 2 inches high. The letters must be white on a blue background.
3. Derails should be placed at the open end or ends of the siding approximately one car length from the car being unloaded.



Wheel chock for tank car.



Derail. Stop sign stands between tracks.

### SPECIAL PRECAUTIONS

**Storage tank inspection:** Before connecting the tank car to the unloading line, make sure the storage tank is vented and the vent is open. Air displaced by the caustic soda solution must be permitted to escape, otherwise the pressure could rupture a tank. **Also make sure there is room for the contents of the car so that no caustic soda solution will overflow the tank.**

**Lighting:** During the hours of darkness no attempt should be made to connect or disconnect a car or to open or close any attachment unless adequate lighting is provided.

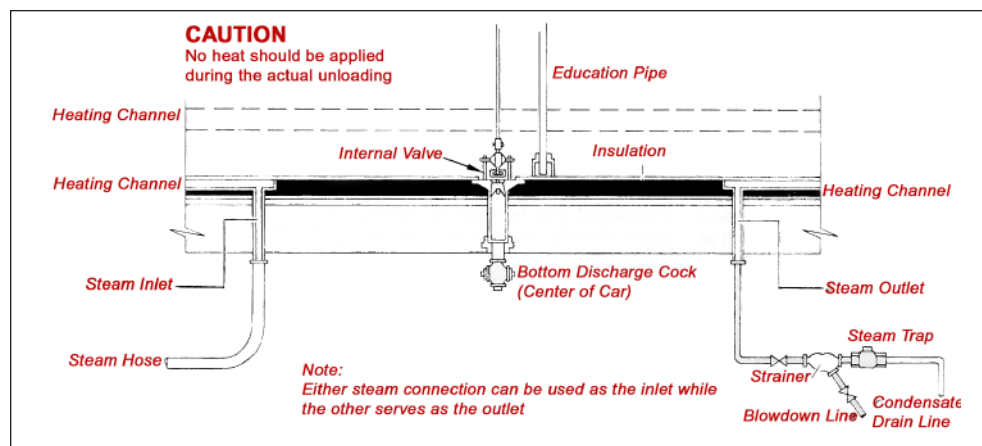
Do not enter cars under any circumstances because of the possibility of caustic burns or suffocation due to insufficient oxygen.

**Moving partially unloaded car:** The movement of partially unloaded cars should be avoided. However, if it should be absolutely necessary to move a partially unloaded tank car, make sure the outlet valve is closed and all connections to the car have been disconnected. The manway cover should be closed and all bolt closures tightened securely. Unloading lines should be drained.

**Safety check:** To prevent caustic soda from contacting personnel, make sure that all unloading connections, piping, joints, valves and storage tanks are sound and tight before unloading. After flow starts, check all around again for leaks.



# Caustic Soda Liquor Unloading and Handling



Preheating Liquid in Tank Car.

## Sampling

Since a sample is taken at the time of loading, many users accept the shipper's analysis. However, on request, PPG will share information on procedures for sampling tank car shipments.

## Preheating Liquid in Tank Car

All PPG tank cars are insulated so that caustic soda liquor is unlikely to freeze—but it may if the tank car delivery is delayed. The 50% caustic soda solution is entirely fluid and can easily be unloaded when its temperature is above 80°F.

The same goes for the 73% solution when its temperature is above 185°F. Difficulty may be encountered in attempting to unload at lower temperatures since viscosity is then greater and there is a hazard of freezing in the unloading lines. Crystals start to form well above the freezing point.

Also, caustic soda solution near the top of a tank car may be as much as 20 degrees hotter than liquor near the

bottom. In fact, the bottom layer may have solidified even though the temperature at the top is well above the solution's freezing point.

If a crust has formed on top of the caustic soda solution, break it up before heating the car contents so the solution can expand without exerting dangerous pressure on the crust. You can break it by inserting a U-tube through the manway onto the crust and applying steam through the U-tube. **Never apply live steam directly onto the surface of caustic soda because of the danger of eruption.**

To apply steam to preheat the tank car contents before unloading, attach a steam line to one of the connections. Install a steam trap on the other connection (see diagram).

The steam connections shown in the diagram on both sides of the discharge leg are for cars with heating channels.

**In extremely cold weather**, heating channels should be drained and blown

out with compressed air to prevent freezing. These channels should also be left open to permit any condensate still remaining after air-blowing to drain out.

Wet or saturated steam at maximum 15 psig and 250°F must be used **because steam at higher temperatures will damage the car lining** and may result in contamination of the caustic liquor. **Do not use superheated steam.** Apply steam gradually to prevent mechanical stresses. Heat should never be applied to a car by blowing steam into the caustic liquor.

**Disconnect all steam lines as soon as the proper unloading temperature has been obtained. No heat should be applied during the actual unloading because the car lining will be damaged.**

## Thawing Frozen Bottom Discharge Cock and Internal Valve at the Bottom of a Car

The bottom discharge cock can be freed by steaming internally through the valve opening or externally on the surface of the cock.



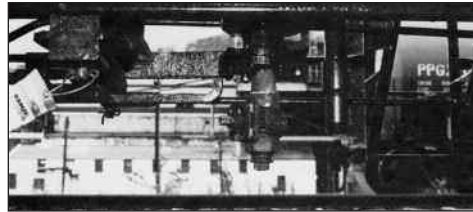
Testing internal valve handle.



## Caustic Soda Liquor Unloading and Handling



If the internal valve cannot be opened with reasonable pressure on the valve handle, it indicates that caustic soda around the valve is frozen and heat should be applied to free it. **Do not attempt to force the valve by applying extreme pressure to the valve handle as this may break the pin connecting the valve to the valve rod and make bottom unloading impossible.**



Above: Steam lance helps loosen frozen valves.

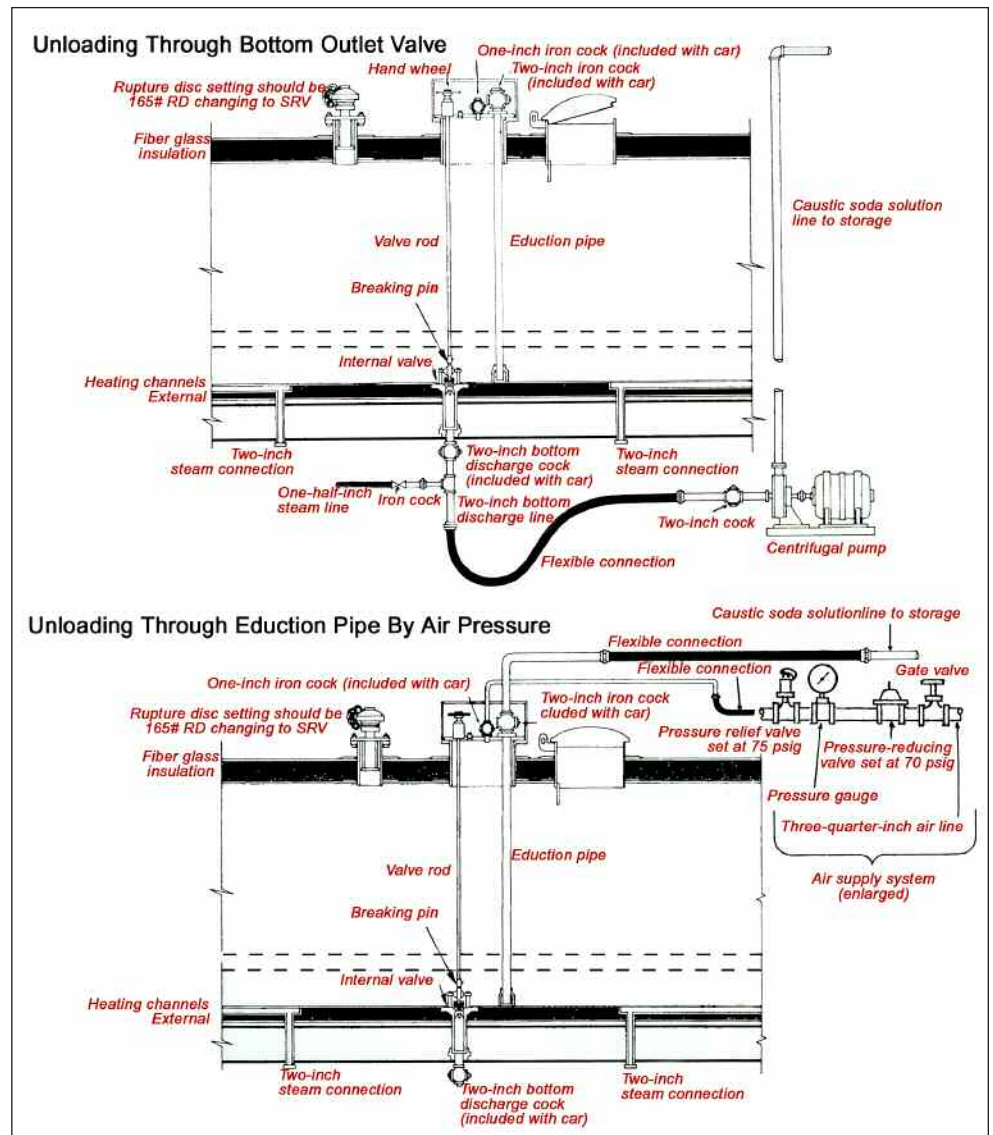


Right: Steam line should always be removed prior to unloading.

The internal valve can be freed by inserting a steam lance of 1/4" copper tubing up through the bottom discharge cock and playing live steam directly onto the internal valve. After a period of 10 to 15 minutes, the frozen caustic soda will have melted. Be sure to withdraw lance and close bottom discharge cock before again trying to open internal valve. The internal valve can now be opened.

### Preheating Unloading Lines

In cold weather it is good practice to preheat unloading lines to prevent cooling and solidifying of caustic soda solutions at the start of unloading. Preheating may be done by passing steam directly through the line. Sections of the unloading line exposed to temperatures below 60°F should be insulated.



# Caustic Soda Liquor Unloading and Handling



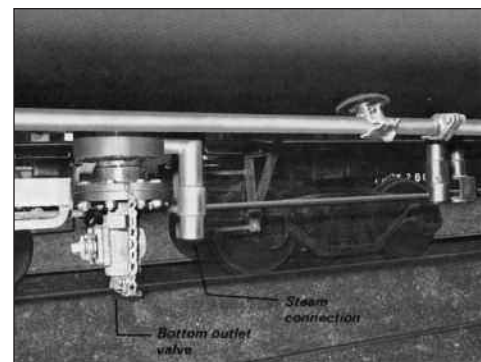
## METHODS OF UNLOADING

Two methods are preferred for unloading tank cars:

1. Through the bottom outlet valve with a pump.
2. Through the top by way of the eduction pipe with air pressure.

### Unloading Through Bottom Outlet Valve

1. Check the inside valve handle on top of the car and make sure it is closed. If it is not, then the internal valve is not completely closed. Vibration in transit can loosen the internal valve and permit caustic liquor to leak by and become trapped above the bottom cock.
2. Carefully open the one-inch cock on the air connection at the top of the car to release any pressure or vacuum that might be in the car. Slicker suit with face shield should be worn.
3. Open the manway cover and support it in a partially open position during unloading. Underneath the manway cover handle are two eyebolts which have the nuts mounted in such a way that they cannot be removed and the eyebolts cannot be swung out of the way until the cover has been lifted slightly. This is a safety device to keep the cover from being blown open.
4. On the manway cover nut-and-bolt fastenings, use only end wrenches—not pipe wrenches.
5. Removing plug requires two wrenches, one to remove plug and one to hold valve so that it remains stationary. Caustic liquor that might have leaked through the internal valve and become caught between the internal valve and bottom cock could come out as a spray if the bottom cock is not closed before the plug is removed.
6. Connect unloading line to the 2-inch bottom cock.
7. See that all valves on the unloading line are in the proper position for unloading.
8. Open the bottom cock.
9. Open internal valve. If this cannot be done with reasonable pressure, see earlier section entitled "Thawing Frozen Bottom Discharge Cock and Internal Valve...".
10. Start pump.
11. After flow starts, check piping valves and hose for leaks.
12. Check unloading progress from time to time by looking into the manway.
13. When the car is empty, stop the pump and close the internal valve. Inspect the car through the manway to make sure it is empty.
14. Prepare empty car for return (see final section of this chapter for instructions).



Tank fittings at bottom of car.

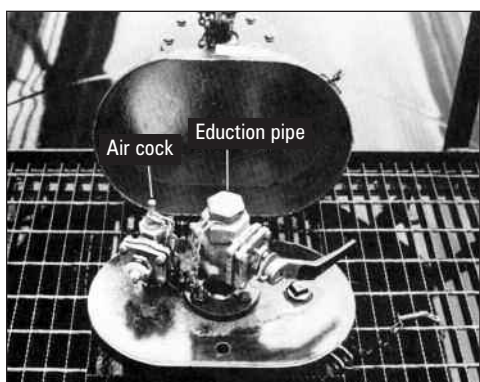
### Top Unloading Through Eduction Pipe by Air Pressure

1. Make sure the manway cover is securely fastened in place and all nuts on the cover are tightened.
2. Inspect all fittings at top of car for defects before unloading to avoid possible injury from caustic spray after the car is put under air pressure.
3. Every PPG car has a safety rupture disc set at 165 pounds per square inch or a safety relief valve (SRV) at 150 pounds per square inch. The air pressure should never exceed 75 pounds per square inch (maximum). Higher pressures may fracture the rupture disc in the safety vent. If a disc is ruptured while unloading, call the PPG Industries Emergency Response Center at (412) 434-4515 for further instructions.
4. The air supply line should be equipped with a pressure relief valve set at 75 pounds per square inch (maximum); a pressure-reducing valve set at 70 pounds per square inch (maximum); a pressure gage; and a shut-off valve.

## Caustic Soda Liquor Unloading and Handling



5. Remove the plug from the 1-inch air valve on top of the car. Open the 1-inch cock to relieve air pressure or vacuum. Cup palm of rubber-gloved hand over cock to prevent being sprayed.



6. Check to see that the 2-inch cock on top of the car is closed. Remove the 2-inch plug and connect the education pipe to the unloading line with a flexible hose. Open the cock.

Use of a flexible hose for connecting tank car to unloading line is recommended. This will facilitate making connections and prevent a possible rupture of the line because the car rises during unloading as the weight on its springs decreases. This rise may be as much as 6 inches.

7. Connect the air supply line to the 1-inch cock. This connection, which should also have flexibility, can be made by using pressure hose or flexible tubing.

8. Apply air pressure slowly until there is a normal flow of caustic liquor to the storage tank. Use of minimum air pressure is recommended.

9. If, after applying air pressure, no caustic flows out of the car, contact PPG Industries Customer Service.

10. Check fittings and line for leaks.

11. Maintain the air pressure until the tank car is completely empty. A drop in the pressure and the sound of air rushing through the discharge pipe tell you that the tank car is empty.

12. Shut off the air supply and 1-inch cock on the car. Disconnect the air supply piping. Cupping your rubber-gloved hand for protection, open the 1-inch cock on the car to make sure pressure has been released from the car before opening the manway cover. Then open the manway cover and inspect the car to make sure it is empty.

13. Prepare empty car for return (see section on page 22 for instructions).

### Unloading and Diluting 73 Percent Caustic Liquor

Since 73 percent caustic soda solution has a freezing point of approximately 145°F, it is shipped in lined, insulated

tank cars at temperatures calculated to arrive at 175 to 200°F. The hot concentrated solution is usually diluted to 50 percent and cooled before storage to reduce handling problems, embrittlement of storage tanks, and iron contamination from tanks.

PPG Industries developed a system that has been used since 1939 for unloading and diluting 73 percent solution. Many years of service in numerous installations have shown the system to be practical and economical, and it must be supervised at all times.

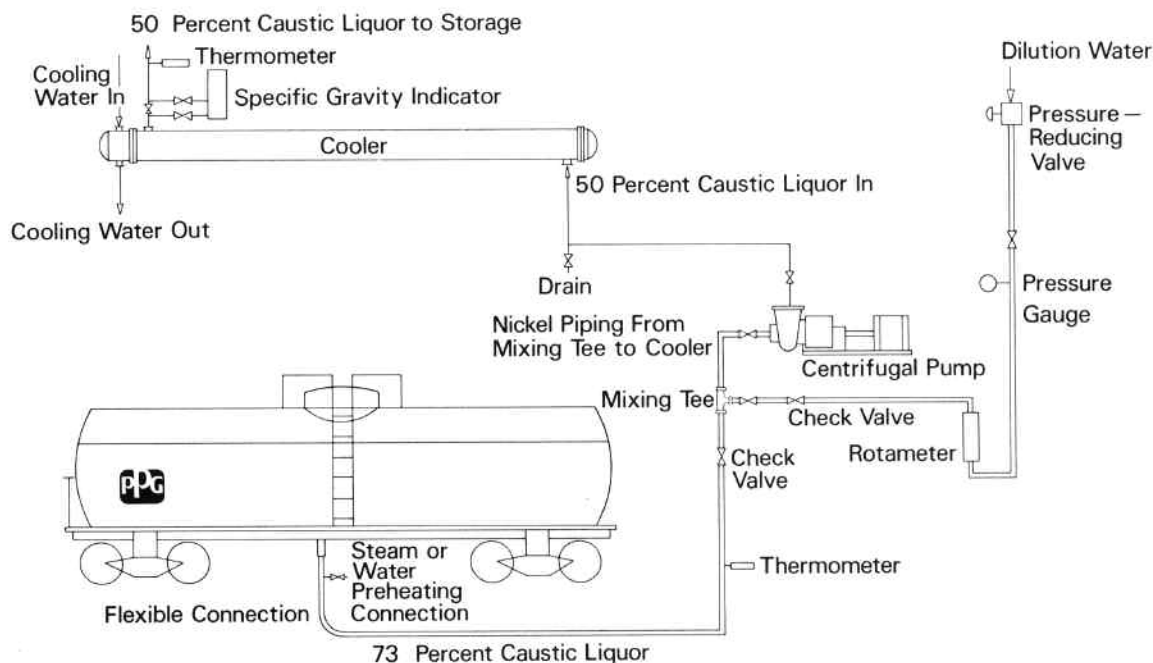
As shown in the schematic diagram, the system discharges 73 percent solution from the tank car to a centrifugal pump. Dilution water at constant pressure is also fed to the suction side of the pump. A rotameter indicates the flow rate of dilution water. A valve controls the amount of water needed to give the 50 percent strength or any other desired concentration.

Heat evolved by the diluting reaction can raise the temperature of the diluted 50 percent solution to as high as 270°F. For safe storage in a steel tank, solution temperature should be less than 140°F. To cool the caustic liquor, it is pumped through a heat exchanger before going to storage. The liquor concentration can be determined by simple measurements of specific gravity and temperature, and can be used to control dilution.

On request, PPG will share information on planning and installing the dilution system.



## Caustic Soda Liquor Unloading and Handling



### Preparing Empty Cars for Return

1. Clean out unloading lines with steam, water or air before disconnecting. When disconnecting and draining the unloading line, be careful not to spill caustic soda solution on personnel, the tank car or the ground. Wash off with water any accidental spill on the car to minimize damage to car and to protect trainmen who will be handling the empty car.
2. If steam line and steam trap were used, remove them from car. **In extremely cold weather**, blow out car's heating coils or channels with compressed air.
3. Close all valves. Replace the bottom outlet plug, or the eduction pipe and air inlet plugs. Close and tighten the manway cover and valve housing cover at top of car.

### Unloading Tank Trucks

Tank trucks are almost always unloaded by the driver. Trailers are equipped with 30 feet of hose unless additional hose is specified on the order. The hose is fitted with a two-inch quick-connect coupling. The receiving installation should have a two-inch quick-connect fitting on the pipe to storage. This pipe should be marked with a caustic soda warning sign.

The trailer can be unloaded by air pressure from a compressor mounted on the truck. If a pump is needed on the truck for unloading, this should be specified on the order.

The unloading area must have a safety spray shower and eye-washing fountain for the driver's protection.



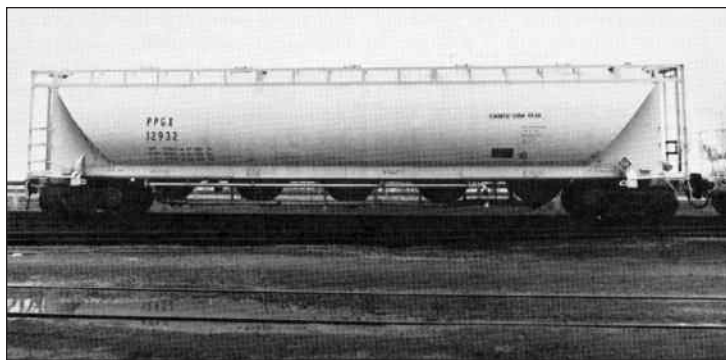
Hoses, valve and unloading connection at rear of tank truck.



# PELS® Caustic Soda Beads Unloading and Handling



**C** caustic soda is a hazardous, reactive chemical. Before persons work with caustic soda, they should be instructed in safe handling practices and first aid, and should wear the recommended protective clothing and equipment (see chapter 3).



Pressure differential rail car for delivery of PELS caustic soda beads in bulk.

## BAGS AND DRUMS

Take care to avoid breaking or puncturing bags and drums of PELS caustic soda beads. Since anhydrous caustic soda left exposed to the atmosphere absorbs moisture and reacts with carbon dioxide, containers must be kept closed. The contents of broken bags or drums must be promptly used or placed in suitable air-tight containers. Drums and bags should be stored in a dry place indoors.

**Dissolving PELS beads:** A short period of mechanical agitation is all that is needed to dissolve this form of caustic soda in water completely. PELS beads dissolve twice as fast as flakes.

**CAUTION:** Do not add water to caustic soda beads. The right way is to add the beads slowly to the surface of

cold water that is being stirred. If the water is not stirred, adding caustic soda beads is dangerous and can result in spattering and eruption. Don't raise the concentration of caustic soda by more than 5% with any single addition. Do not add caustic soda beads to hot water. The water should be at

ambient temperature. The high heat of solution of dry caustic soda may cause sudden evolution of steam and spattering and eruption. Also a layer of concentrated solution may form and suddenly mix with a layer of less concentrated

solution. In this case, the high heat of dilution may create steam and cause the solution to spatter and erupt.

## Disposing of Containers

Flush all traces of PELS caustic soda beads from drums. And then flush again to get rid of any dissolved caustic soda adhering to the metal before disposing of drums. This will protect people handling these drums later on who may not recognize the traces as being caustic soda or be aware of its dangers.

**Disposing of Bags:** Do not re-use bags. Dispose of waste materials according to approved procedures and regulatory laws/regulation regarding disposal.

## Cleaning up Spills and Disposing of Waste

When cleaning up a spill, wear proper protective clothing and equipment. Shovel up any spilled PELS caustic soda beads. Do not put unneutralized waste caustic soda into a sewer or stream. Follow an approved procedure for neutralizing the spilled material and getting rid of it in your waste disposal system.

Flush the area contaminated by the spill with huge quantities of water.

Waste material must be disposed of in an approved hazardous waste facility. Care must be taken when using or disposing of chemical materials and/or their containers to prevent environmental contamination. Chemical materials and/or their containers must be disposed of in accordance with the Clean Air Act, the Clean Water Act, the Resource Conservation and Recovery Act, as well as any other relevant federal, state, or local laws/regulations regarding disposal.

**Do not re-use bag.**

## Hazards

Avoid breathing dusts or mists from solutions.

Avoid contact with acids, aluminum, chromium, tin, zinc (galvanized metal), brass, bronze and organic materials. Such contact may generate explosive or toxic materials. Avoid contact with sugars. Such contact may generate toxic carbon monoxide.



## PELS® Caustic Soda Beads Unloading and Handling



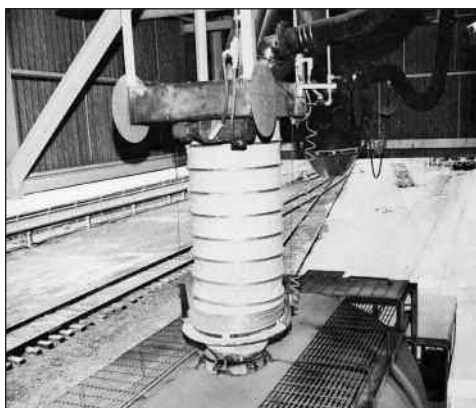
### CSD (CLOSED SYSTEM DELIVERY) BULK UNLOADING AND HANDLING

PPG engineers designed "CSD," a closed system delivery method for bulk shipping, handling and storing PELS caustic soda.

The company has a number of pressure differential rail cars designed for pneumatic unloading. Each has a caustic soda compatible protective coating. PPG also maintains its own fleet of lined pressure differential truck trailers.



Manifold piping of pressure differential rail car.



Automatic loading of piping of pressure differential rail car.

### Advantages of CSD Rail Car System

**1. HIGH SPEED:** Unloading rate ranges from 15 to 20 tons per hour. A 90-ton car can be unloaded in less than an 8-hour shift.

**2. REDUCED MANPOWER:** One man can carry out the entire unloading procedure. There is no need to unload drums from boxcar or truck; move them to storage area; and stack them.

**3. INCREASED SAFETY:** Closed loop pneumatic conveying system helps reduce the opportunity for human contact with caustic soda.

**4. IMPROVED HANDLING:** When exposed to the atmosphere, caustic soda readily absorbs moisture. The crystals become soft and tacky so that crusts and lumps can form in storage. By exposing caustic soda only to dry air in a closed loop system, CSD avoids caking and retains the original free-flowing characteristics of anhydrous caustic soda.

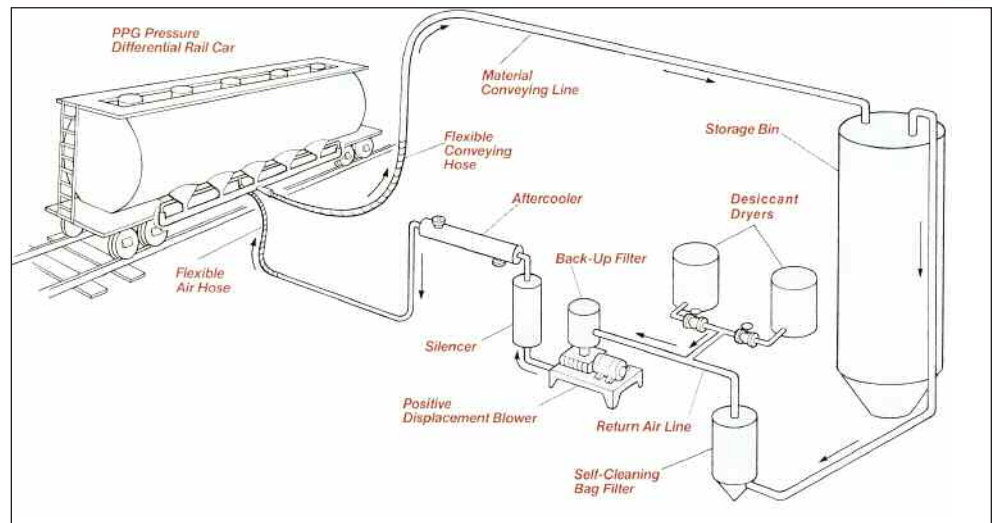
### 5. ALL-WEATHER OPERATION:

Rain and temperature extremes do not interfere with enclosed pneumatic conveying system; do not stop unloading operations.

### CSD operation

The diagram below shows the main components of a typical CSD closed loop pneumatic system.

When conveying air is admitted to the system, it passes over a desiccant to dry the air to a minimum dew point temperature of  $-40^{\circ}$  (measured at atmospheric pressure). The blower compresses the air at a rate of 600 cubic feet per minute to 15 psig. Upon leaving the blower, the air is cooled in a heat exchanger to a temperature less than  $140^{\circ}\text{F}$ . The air then enters a manifold on the underside of the car through piping and flexible hose. The air is distributed from the manifold to the car interior and to the conveying line. When sufficient pressure has built up in the car, a discharge valve is



CSD bulk unloading system for rail cars.

# PELS® Caustic Soda Beads Unloading and Handling



opened. Anhydrous caustic soda can now flow into the conveying line. The caustic soda is carried through the conveying line by a high velocity air stream to the top of the storage bin.

After the conveying air leaves the storage bin, any entrained dust is removed by an automatic self-cleaning bag-type filter. The filtered air is returned to the blower intake to complete the pneumatic loop. Since the recycled air has been in contact with highly hygroscopic caustic soda, this air is extremely dry. Any make-up air that is required enters the closed loop through the air dryers at the blower section. The air dryers reduce the dew point of the make-up air to -40°F.

Between unloading operations, the storage bin and other parts of the system are protected against moisture pickup by the same dryers that admit air to the system. The economical design plus the dual purpose of the dryers is a unique feature of CSD.

### Advantages of CSD Truck Trailer System

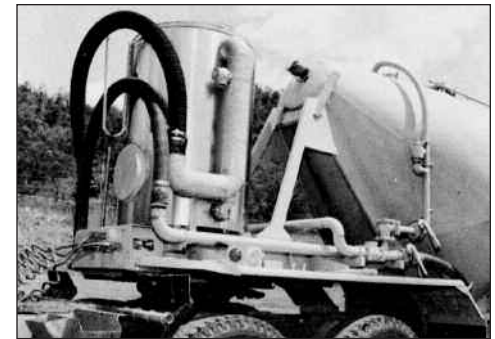
Trucks with hopper trailers deliver 22-ton shipments. These systems have the same advantages just described for rail car systems plus additional benefits.

A blower is mounted on the truck and a dryer is mounted on the trailer (see photo and diagram) so that the receiving installation needs to include only a dust filter, a storage bin fitted with a vent dryer and piping from the truck to the storage bin. Accordingly, the

investment for a system to receive truck trailer shipments is less than one designed for rail car deliveries.

### CSD System Design

Since anhydrous caustic soda is less corrosive than solutions, its demands upon materials of construction are less critical. Steel is the usual material. A smooth, continuous surface inside the

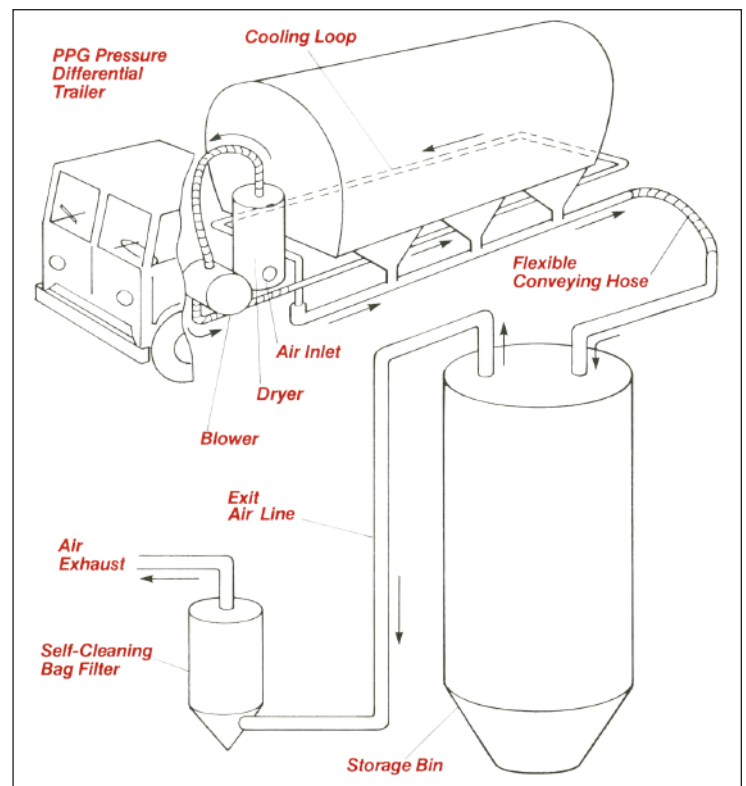


Dryer for closed system delivery is mounted on trailer.

Pressure differential hopper trailer for delivery of PELS caustic soda beads in bulk.

conveying line and a minimum of bends promote the efficiency of pneumatic conveying.

Each installation requires an individual design dependent upon existing plant facilities. On request, PPG will share information in evaluating, planning and designing the complete system.



CSD bulk unloading system for truck trailers.

# Technical Data



**C**austic soda is a hazardous, reactive chemical. Before persons work with caustic soda, they should be instructed in safe handling practices and first aid, and should wear the recommended protective clothing and equipment (see chapter 3).

## Chemical Reactivity

Caustic soda, either dry or in solution, is not combustible; has no flash point, no autoignition temperature, and no explosive limits.

Anhydrous caustic soda is deliquescent (dissolving in moisture absorbed from the atmosphere) and reacts with

carbon dioxide in the air to form sodium carbonate. Conversion is 100 percent after prolonged exposure. Therefore, minimum exposure to air is desirable in handling anhydrous forms. On the other hand, the hygroscopic nature of anhydrous caustic soda makes it useful as a drying agent.

Solutions of caustic soda are strongly alkaline and have a high pH or high concentration of negatively charged hydroxyl ions,  $\text{OH}^-$ . The high reactivity of caustic soda with many organic and inorganic substances, including certain metals is the main reason for its importance as a basic chemical.

## Hydration

Anhydrous caustic soda is a white translucent solid with a fibrous crystalline structure. Being hygroscopic, it attracts water molecules and forms hydrates. Six hydrates of caustic soda have been identified, each with its own distinctive crystalline structure. The number of water molecules attached to each molecule of sodium hydroxide, corresponding to the six hydrated forms, is 7, 5, 4,  $3\frac{1}{2}$ , 2 or 1. This degree of hydration depends upon the temperature and concentration of the solution in which the crystals form, as shown in the diagram on the facing page. The curve shows the freezing points of solutions with different concentrations; the eutectic solution is just less than 19 percent.

## Anhydrous Sodium Hydroxide

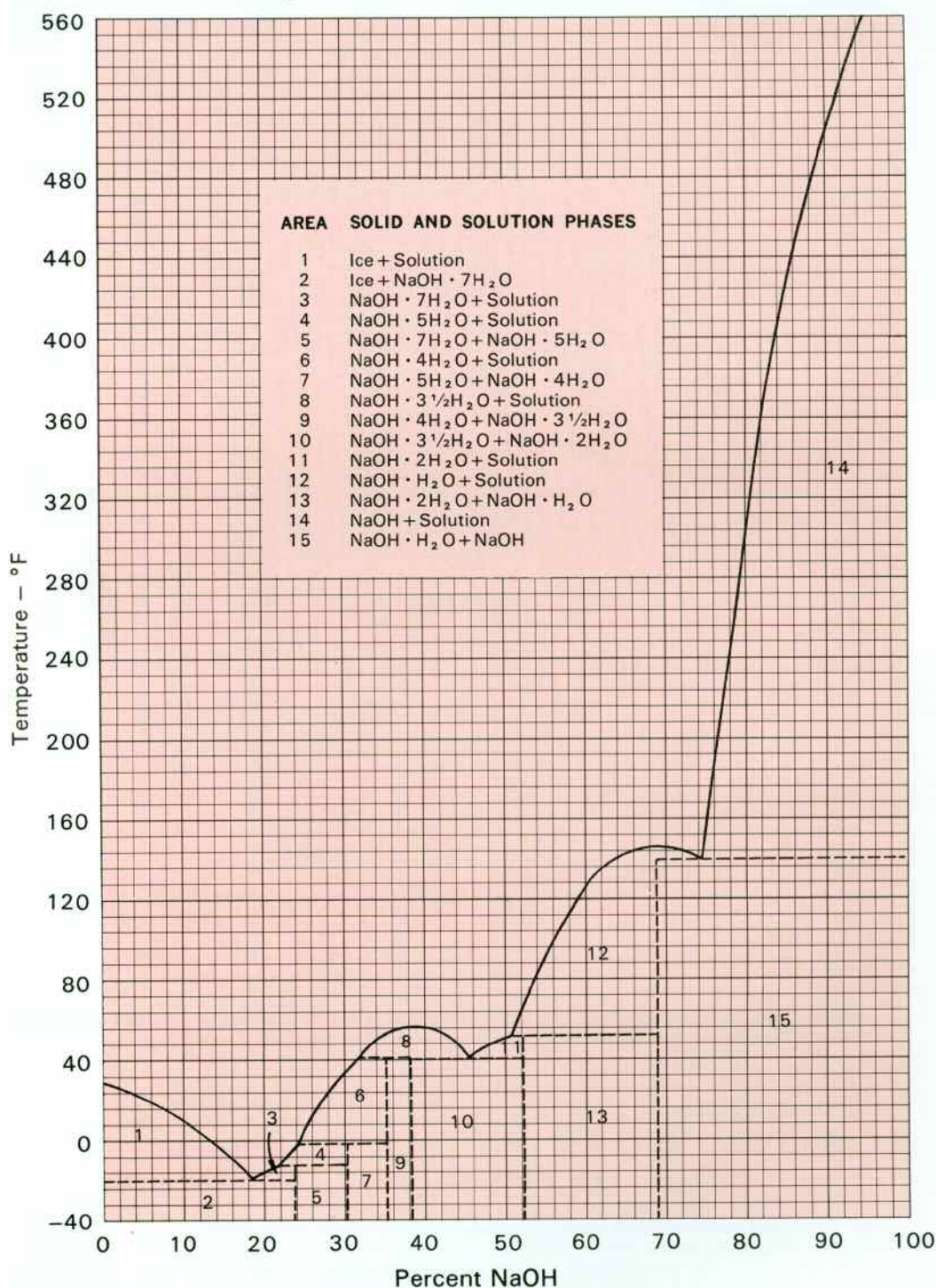
Molecular Weight	40.005
Melting Range	590 to 608°F (310 to 320°C)
Boiling Point	2534°F (1390°C)
Specific Gravity of Pure Solid	2.130
Refractive Index	1.3576
Heat of Fusion	72 Btu/lb 40.0 cal/g
Specific Heat at 20°C	0.48 Btu/(lb) (°F) 0.48 cal/(g) (°C)
Solubility in Water at 0°C	42 g/100 g water
At 100°C	347 g/100 g water

Caustic Soda Solutions	50 Percent Solution	73 Percent Solution
Melting Range (crystallization begins)	41 to 51° F (5 to 11° C)	140 to 143° F (60 to 62° C)
Solidification Point	41° F (5° C)	140° F (60° C)





Freezing Points of Caustic Soda Solutions



Derived from "International Critical Tables," Volume IV, page 235, and PPG Industries laboratories.

# Technical Data



**Specific Gravity of Caustic Soda Solutions\* At Various Temperatures**

Percent NaOH	32°F 0°C	50°F 10°C	68°F 20°C	86°F 30°C	104°F 40°C	122°F 50°C	140°F 60°C	176°F 80°C	194°F 90°C	212°F 100°C
2	1.024	1.023	1.021	1.018	1.014	1.010	1.004	0.993	0.987	0.980
4	1.048	1.046	1.043	1.039	1.035	1.031	1.025	1.014	1.008	1.001
6	1.071	1.068	1.065	1.061	1.056	1.052	1.046	1.035	1.028	1.022
8	1.094	1.091	1.087	1.083	1.078	1.073	1.068	1.056	1.050	1.043
10	1.117	1.113	1.109	1.014	1.100	1.094	1.089	1.077	1.071	1.064
12	1.140	1.136	1.131	1.126	1.121	1.116	1.110	1.098	1.092	1.086
14	1.162	1.158	1.153	1.148	1.143	1.137	1.132	1.120	1.113	1.107
16	1.185	1.180	1.175	1.170	1.165	1.159	1.153	1.141	1.134	1.128
18	1.207	1.202	1.197	1.192	1.186	1.181	1.175	1.162	1.156	1.149
20	1.230	1.224	1.219	1.214	1.208	1.202	1.196	1.183	1.177	1.170
22	1.252	1.247	1.241	1.235	1.230	1.224	1.217	1.205	1.198	1.191
24	1.274	1.269	1.263	1.257	1.251	1.245	1.239	1.226	1.219	1.212
26	1.295	1.291	1.285	1.279	1.273	1.267	1.260	1.247	1.241	1.234
28	1.318	1.312	1.306	1.300	1.294	1.288	1.281	1.268	1.262	1.255
30	.....	1.334	1.328	1.322	1.315	1.309	1.302	1.289	1.282	1.276
32	.....	1.355	1.349	1.343	1.336	1.330	1.323	1.310	1.303	1.296
34	.....	.....	1.370	1.363	1.357	1.350	1.343	1.330	1.323	1.316
36	.....	.....	1.390	1.384	1.377	1.370	1.363	1.350	1.343	1.336
38	.....	.....	1.410	1.404	1.397	1.390	1.383	1.370	1.363	1.356
40	.....	.....	1.430	1.423	1.416	1.410	1.403	1.389	1.382	1.375
42	.....	.....	1.449	1.443	1.436	1.429	1.422	1.408	1.401	1.394
44	.....	.....	1.468	1.462	1.455	1.448	1.441	1.427	1.420	1.413
46	.....	.....	1.487	1.481	1.473	1.466	1.459	1.445	1.438	1.432
48	.....	.....	1.506	1.500	1.492	1.485	1.478	1.464	1.457	1.450
50	.....	.....	1.525	1.518	1.511	1.504	1.497	1.483	1.476	1.470
52	.....	.....	.....	1.537	1.530	1.524	1.517	1.503	1.496	1.490
54	.....	.....	.....	.....	1.549	1.543	1.536	1.523	1.516	1.510
56	.....	.....	.....	.....	1.568	1.562	1.556	1.543	1.536	1.530
58	.....	.....	.....	.....	.....	1.581	1.575	1.563	1.556	1.550
60	.....	.....	.....	.....	.....	.....	1.597	1.583	1.576	1.570
62	.....	.....	.....	.....	.....	.....	.....	1.603	1.596	1.590
64	.....	.....	.....	.....	.....	.....	.....	1.623	1.616	1.610
66	.....	.....	.....	.....	.....	.....	.....	1.643	1.636	1.630
68	.....	.....	.....	.....	.....	.....	.....	1.663	1.656	1.650
70	.....	.....	.....	.....	.....	.....	.....	1.683	1.676	1.670
72	.....	.....	.....	.....	.....	.....	.....	1.703	1.696	1.690
74	.....	.....	.....	.....	.....	.....	.....	1.723	1.716	1.710

\*These values are for pure sodium hydroxide in distilled water. Data below 50 percent were derived from "International Critical Tables," Volume III, page 79. Data above 50 percent were obtained by extrapolation.





Specific Gravity of Caustic Soda Solutions\*



\*These values are for pure sodium hydroxide in distilled water. Data below 50 percent were derived from "International Critical Tables," Volume III, page 79. Data above 50 percent were obtained by extrapolation.

# Technical Data



## Specific Gravity of Mercury Cell and Rayon Grade Caustic Soda Solutions\* – In Range from 50.6 to 52.1%

Percent NaOH, 76% Na<sub>2</sub>O Basis (This is the billing basis described on page 8.)

°F	50.6	50.7	50.8	50.9	51.0	51.1	51.2	51.3	51.4	51.5	51.6	51.7	51.8	51.9	52.0	52.1
76	1.519	1.520	1.521	1.522	1.523	1.524	1.525	1.526	1.527	1.528	1.529	1.530	1.531	1.532	1.533	1.534
78	1.518	1.519	1.520	1.521	1.522	1.523	1.524	1.525	1.526	1.527	1.528	1.529	1.530	1.531	1.532	1.533
80	1.518	1.519	1.520	1.521	1.522	1.523	1.524	1.525	1.526	1.527	1.528	1.529	1.530	1.531	1.532	1.533
82	1.517	1.518	1.519	1.520	1.521	1.522	1.523	1.524	1.525	1.526	1.527	1.528	1.529	1.530	1.531	1.532
84	1.516	1.517	1.518	1.519	1.520	1.521	1.522	1.523	1.524	1.525	1.526	1.527	1.528	1.529	1.530	1.531
86	1.515	1.516	1.517	1.518	1.519	1.520	1.521	1.522	1.523	1.524	1.525	1.526	1.527	1.528	1.529	1.530
88	1.514	1.515	1.516	1.517	1.518	1.519	1.520	1.521	1.522	1.523	1.524	1.525	1.526	1.527	1.528	1.529
90	1.514	1.515	1.516	1.516	1.517	1.518	1.520	1.520	1.521	1.522	1.523	1.524	1.526	1.527	1.527	1.528
92	1.513	1.514	1.515	1.516	1.517	1.518	1.519	1.520	1.521	1.522	1.523	1.524	1.525	1.526	1.527	1.528
94	1.512	1.513	1.514	1.515	1.516	1.517	1.518	1.519	1.520	1.521	1.522	1.523	1.524	1.525	1.526	1.527
98	1.510	1.511	1.512	1.513	1.514	1.515	1.516	1.517	1.518	1.519	1.520	1.521	1.522	1.523	1.524	1.525
100	1.509	1.510	1.511	1.512	1.513	1.514	1.515	1.516	1.517	1.518	1.519	1.520	1.521	1.522	1.523	1.524
102	1.509	1.510	1.511	1.512	1.512	1.514	1.515	1.516	1.516	1.517	1.519	1.520	1.520	1.521	1.522	1.523
104	1.508	1.509	1.510	1.511	1.512	1.513	1.514	1.515	1.516	1.517	1.518	1.519	1.520	1.521	1.522	1.523
106	1.507	1.508	1.509	1.510	1.511	1.512	1.513	1.514	1.515	1.516	1.517	1.518	1.519	1.520	1.521	1.522
108	1.506	1.507	1.508	1.509	1.510	1.511	1.512	1.513	1.514	1.515	1.516	1.517	1.518	1.519	1.520	1.521
110	1.505	1.506	1.507	1.508	1.509	1.510	1.511	1.512	1.513	1.514	1.515	1.516	1.517	1.518	1.519	1.520
112	1.504	1.505	1.506	1.507	1.508	1.509	1.510	1.511	1.512	1.513	1.514	1.515	1.516	1.517	1.518	1.519
114	1.504	1.505	1.506	1.507	1.507	1.508	1.510	1.511	1.511	1.512	1.513	1.514	1.515	1.516	1.517	1.518
116	1.503	1.504	1.505	1.506	1.507	1.508	1.509	1.510	1.511	1.512	1.513	1.514	1.515	1.516	1.517	1.518
118	1.502	1.503	1.504	1.505	1.506	1.507	1.508	1.509	1.510	1.511	1.512	1.513	1.514	1.515	1.516	1.517
120	1.501	1.502	1.503	1.504	1.505	1.506	1.507	1.508	1.509	1.510	1.511	1.512	1.513	1.514	1.515	1.516
122	1.500	1.501	1.502	1.503	1.504	1.505	1.506	1.507	1.508	1.509	1.510	1.511	1.512	1.513	1.514	1.515
124	1.500	1.500	1.502	1.502	1.503	1.504	1.505	1.506	1.507	1.508	1.509	1.510	1.511	1.512	1.513	1.514
126	1.499	1.500	1.501	1.502	1.503	1.504	1.505	1.506	1.507	1.507	1.509	1.510	1.511	1.512	1.513	1.514
128	1.498	1.499	1.500	1.501	1.502	1.503	1.504	1.505	1.506	1.507	1.508	1.509	1.510	1.511	1.512	1.513
130	1.497	1.498	1.499	1.500	1.501	1.502	1.503	1.504	1.505	1.506	1.507	1.508	1.509	1.510	1.511	1.512
132	1.496	1.497	1.498	1.499	1.500	1.501	1.502	1.503	1.504	1.505	1.506	1.507	1.508	1.509	1.510	1.511
134	1.495	1.496	1.497	1.498	1.499	1.500	1.501	1.502	1.503	1.504	1.505	1.506	1.507	1.508	1.509	1.510
136	1.495	1.495	1.497	1.498	1.499	1.500	1.500	1.502	1.502	1.503	1.505	1.505	1.506	1.507	1.508	1.509
138	1.494	1.495	1.496	1.497	1.498	1.499	1.500	1.501	1.502	1.503	1.504	1.505	1.506	1.507	1.507	1.508
140	1.493	1.494	1.495	1.496	1.497	1.498	1.499	1.500	1.501	1.502	1.503	1.504	1.505	1.506	1.507	1.508

\*The specific gravity values in this table do not correlate with those on page 27, which are based on pure sodium hydroxide in distilled water. In contrast, the values on this page are on a 76 percent Na<sub>2</sub>O basis, which includes sodium carbonate, as explained in detail on page 8. Furthermore, the standard and low-iron grade caustic soda solutions contain over two percent sodium chloride on a 100% NaOH basis.

Note: Table data based on water at 39.2°F (4°C).



# Technical Data



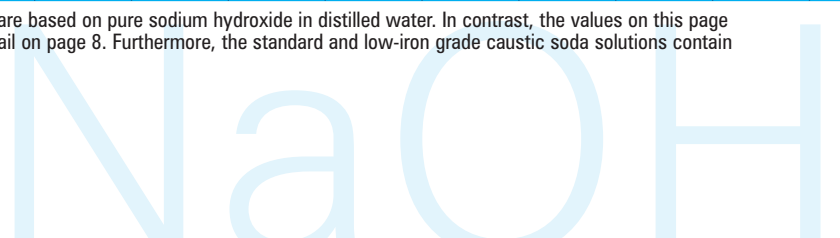
## Specific Gravity of Standard and Low-Iron Grade Caustic Soda Solutions\* – In Range from 50.6 to 52.1%

Percent NaOH, 76% Na<sub>2</sub>O Basis (This is the billing basis described on page 11.)

°F	50.6	50.7	50.8	50.9	51.0	51.1	51.2	51.3	51.4	51.5	51.6	51.7	51.8	51.9	52.0	52.1
76	1.526	1.527	1.529	1.530	1.531	1.532	1.533	1.534	1.535	1.536	1.537	1.537	1.538	1.539	1.540	1.541
78	1.525	1.527	1.528	1.529	1.531	1.532	1.532	1.533	1.534	1.535	1.536	1.537	1.538	1.539	1.540	1.541
80	1.525	1.526	1.527	1.529	1.530	1.531	1.531	1.533	1.534	1.534	1.535	1.536	1.537	1.538	1.539	1.540
82	1.524	1.525	1.527	1.528	1.529	1.530	1.531	1.532	1.533	1.534	1.534	1.535	1.536	1.537	1.538	1.539
84	1.523	1.524	1.526	1.527	1.528	1.529	1.530	1.531	1.532	1.533	1.533	1.534	1.535	1.536	1.537	1.538
86	1.522	1.524	1.525	1.526	1.528	1.529	1.529	1.530	1.531	1.532	1.533	1.534	1.535	1.536	1.537	1.537
88	1.522	1.523	1.524	1.525	1.527	1.528	1.528	1.530	1.530	1.531	1.532	1.533	1.534	1.535	1.536	1.537
90	1.521	1.522	1.523	1.525	1.526	1.527	1.528	1.529	1.530	1.530	1.531	1.532	1.533	1.534	1.535	1.536
92	1.520	1.521	1.523	1.524	1.525	1.526	1.527	1.528	1.529	1.530	1.530	1.531	1.532	1.533	1.534	1.535
94	1.519	1.521	1.522	1.523	1.524	1.525	1.526	1.527	1.528	1.529	1.530	1.531	1.532	1.533	1.534	1.534
96	1.518	1.520	1.521	1.522	1.524	1.525	1.525	1.526	1.527	1.528	1.529	1.530	1.531	1.532	1.533	1.534
98	1.518	1.519	1.520	1.522	1.523	1.524	1.525	1.526	1.527	1.527	1.528	1.529	1.530	1.531	1.532	1.533
100	1.517	1.518	1.520	1.521	1.522	1.523	1.524	1.525	1.526	1.527	1.527	1.528	1.529	1.530	1.531	1.532
102	1.516	1.517	1.519	1.20	1.521	1.522	1.523	1.524	1.525	1.526	1.527	1.528	1.529	1.530	1.530	1.531
104	1.515	1.517	1.518	1.519	1.521	1.522	1.522	1.523	1.524	1.525	1.526	1.527	1.528	1.529	1.530	1.531
106	1.515	1.516	1.517	1.519	1.520	1.521	1.521	1.522	1.523	1.524	1.525	1.526	1.527	1.528	1.529	1.530
108	1.514	1.515	1.516	1.518	1.519	1.520	1.521	1.522	1.523	1.523	1.524	1.525	1.526	1.527	1.528	1.529
110	1.513	1.514	1.516	1.517	1.518	1.519	1.520	1.521	1.522	1.523	1.523	1.524	1.525	1.526	1.527	1.528
112	1.512	1.514	1.515	1.516	1.517	1.518	1.519	1.520	1.521	1.522	1.523	1.524	1.524	1.526	1.527	1.527
114	1.511	1.513	1.514	1.515	1.517	1.518	1.518	1.519	1.520	1.521	1.522	1.523	1.524	1.525	1.526	1.527
116	1.511	1.512	1.513	1.515	1.516	1.517	1.518	1.519	1.519	1.520	1.521	1.522	1.523	1.524	1.525	1.526
118	1.510	1.511	1.513	1.514	1.515	1.516	1.517	1.518	1.519	1.520	1.520	1.521	1.522	1.523	1.524	1.525
120	1.509	1.510	1.512	1.513	1.514	1.515	1.516	1.517	1.518	1.519	1.520	1.520	1.521	1.522	1.523	1.524
122	1.508	1.510	1.511	1.512	1.514	1.515	1.515	1.516	1.517	1.518	1.519	1.520	1.521	1.522	1.523	1.524
124	1.508	1.509	1.510	1.512	1.513	1.514	1.514	1.515	1.516	1.517	1.518	1.519	1.520	1.521	1.522	1.523
126	1.507	1.508	1.510	1.511	1.512	1.513	1.514	1.515	1.515	1.516	1.517	1.518	1.519	1.520	1.521	1.522
128	1.506	1.507	1.509	1.510	1.511	1.512	1.513	1.514	1.515	1.516	1.516	1.517	1.518	1.519	1.520	1.521
130	1.505	1.507	1.508	1.509	1.510	1.511	1.512	1.513	1.514	1.515	1.516	1.517	1.518	1.519	1.520	1.520
132	1.504	1.506	1.507	1.508	1.510	1.511	1.511	1.512	1.513	1.514	1.515	1.516	1.517	1.518	1.519	1.520
134	1.504	1.515	1.506	1.508	1.509	1.510	1.511	1.511	1.512	1.513	1.514	1.515	1.516	1.517	1.518	1.519
136	1.503	1.504	1.506	1.507	1.508	1.509	1.510	1.511	1.511	1.512	1.513	1.514	1.515	1.516	1.517	1.518
138	1.502	1.503	1.505	1.506	1.507	1.508	1.509	1.510	1.511	1.512	1.513	1.513	1.514	1.515	1.517	1.517
140	1.501	1.503	1.504	1.505	1.506	1.507	1.508	1.509	1.510	1.511	1.512	1.513	1.514	1.515	1.516	1.517

\*The specific gravity values in this table do not correlate with those on page 27, which are based on pure sodium hydroxide in distilled water. In contrast, the values on this page are on a 76 percent Na<sub>2</sub>O basis, which includes sodium carbonate, as explained in detail on page 8. Furthermore, the standard and low-iron grade caustic soda solutions contain over two percent sodium chloride on a 100% NaOH basis.

Note: Table data based on water at 39.2°F (4°C).



# Technical Data


**Density of Caustic Soda Solutions at 60 Degrees Fahrenheit**

Percent NaOH	Percent Na <sub>2</sub> O	Specific Gravity 60/60°F	American Standard Baumé Degrees	Degrees Twaddell	Grams NaOH Per Liter	Pounds NaOH		Pounds of Solution	
						Per Gallon	Per Cubic Foot	Per Gallon	Per Cubic Foot
2	1.55	1.023	3.26	4.6	21	1.17	1.28	8.53	63.80
4	3.10	1.045	6.24	9.0	42	0.35	2.60	8.71	65.18
6	4.65	1.067	9.10	13.4	64	0.53	3.99	8.90	66.55
8	6.20	1.090	11.97	18.0	87	0.73	5.44	9.09	67.98
10	7.75	1.112	14.60	22.4	111	0.93	6.94	9.27	69.36
12	9.30	1.134	18.13	26.8	136	1.13	8.49	9.45	70.74
14	10.85	1.156	19.57	31.2	162	1.35	10.09	9.63	72.10
16	12.40	1.178	21.91	35.6	188	1.57	11.76	9.82	73.47
18	13.95	1.201	23.27	40.2	215	1.80	13.44	10.01	74.91
20	15.50	1.223	26.44	44.6	244	2.04	15.26	10.20	76.28
22	17.05	1.245	28.53	49.0	274	2.28	17.08	10.38	77.65
24	18.60	1.267	30.66	53.4	304	2.53	18.96	10.56	79.02
26	20.15	1.289	32.51	57.8	335	2.79	20.90	10.75	80.39
28	21.70	1.310	34.31	62.0	366	3.06	22.88	10.92	81.70
30	23.25	1.332	36.14	66.4	399	3.33	24.92	11.11	83.08
32	24.80	1.353	37.83	70.6	433	3.61	27.00	11.28	84.39
34	26.35	1.374	39.47	74.8	467	3.89	29.14	11.46	85.71
36	27.90	1.394	40.98	78.8	501	4.18	31.30	11.62	86.95
38	29.45	1.415	42.53	83.0	537	4.48	33.53	11.80	88.25
40	31.00	1.434	43.88	86.8	573	4.78	35.78	11.96	89.45
42	32.55	1.454	45.28	90.8	610	5.09	38.09	12.12	90.70
44	34.10	1.473	46.66	94.6	648	5.40	40.42	12.28	91.87
46	35.65	1.492	47.82	98.4	686	5.72	42.81	12.44	93.06
48	37.20	1.511	49.04	102.2	725	6.04	45.24	12.60	94.24
50	38.75	1.530	50.23	106.0	754	6.38	47.72	12.76	95.43
52	40.30	1.549	51.39	109.8	805	6.71	50.24	12.92	96.62

NaOH



# Technical Data


**Density of Caustic Soda Solutions at 212 Degrees Fahrenheit**

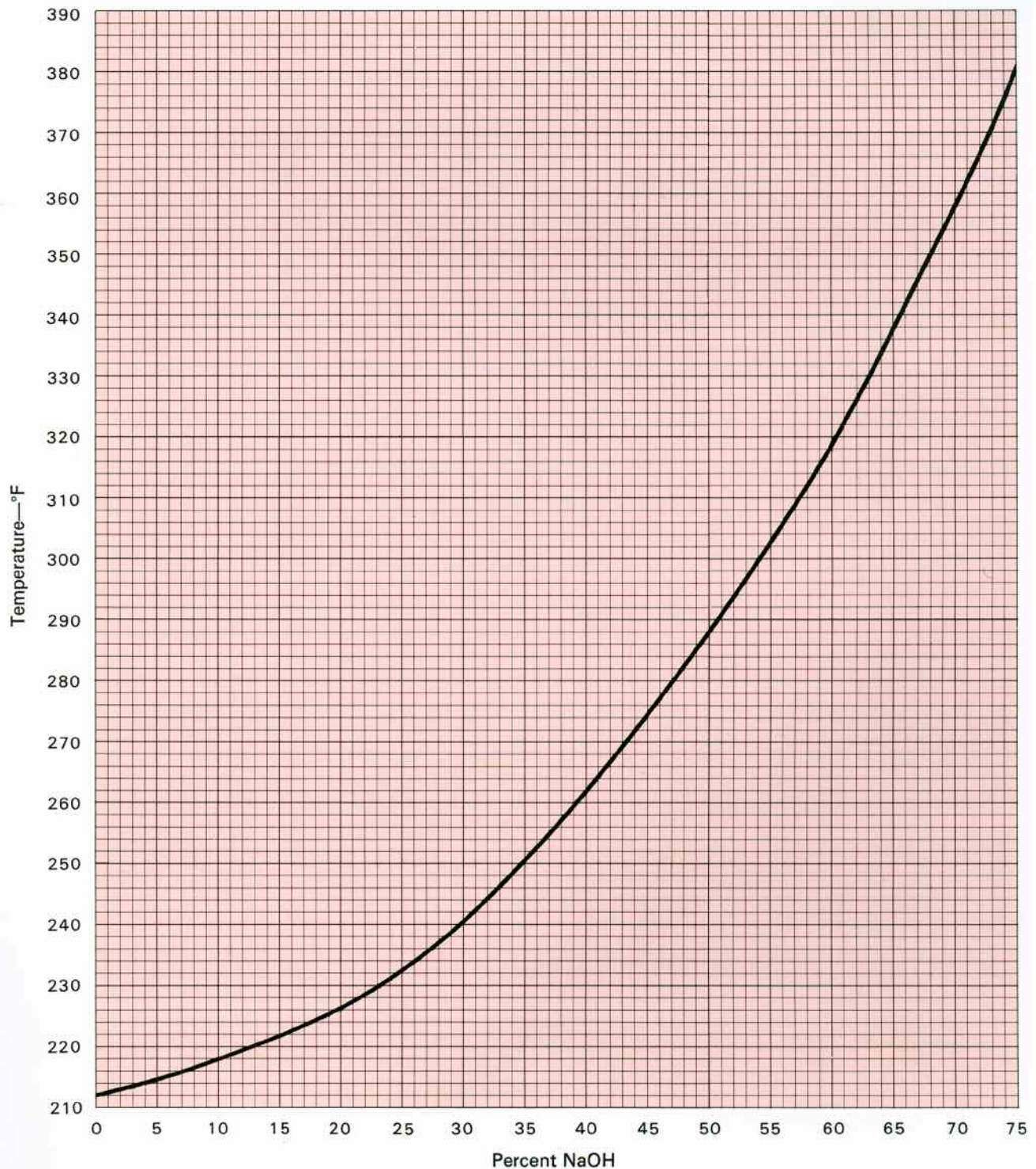
Percent NaOH	Percent Na <sub>2</sub> O	Specific Gravity 212/60°F	American Standard Baumé Degrees	Degrees Twaddell	Grams NaOH Per Liter	Pounds NaOH		Pounds of Solution	
						Per Gallon	Per Cubic Foot	Per Gallon	Per Cubic Foot
52	40.30	1.49	47.68	98	774	6.46	48.32	12.42	92.93
54	41.85	1.51	48.97	102	815	6.80	50.86	12.59	94.18
56	43.40	1.53	50.23	106	856	7.15	53.44	12.76	95.43
58	44.95	1.55	51.45	110	898	7.49	56.07	12.92	96.67
60	46.50	1.57	52.64	114	941	7.85	58.75	13.09	97.92
62	48.05	1.59	53.81	118	985	8.22	61.49	13.26	99.17
64	49.60	1.61	54.94	122	1030	8.59	64.27	13.42	100.42
66	51.15	1.63	56.04	126	1075	8.97	67.10	13.59	101.66
68	52.70	1.65	57.12	130	1121	9.36	69.98	13.76	102.91
70	54.25	1.67	58.17	134	1168	9.74	72.91	13.92	104.16
72	55.80	1.69	59.20	138	1216	10.14	75.90	14.09	105.41
74	57.35	1.71	60.20	142	1264	10.55	78.92	14.26	106.65

NaOH



# Technical Data

Boiling Points of Caustic Soda Solutions At Standard Atmospheric Pressure



Derived from "International Critical Tables," Volume III, page 326, and PPG Industries laboratories.



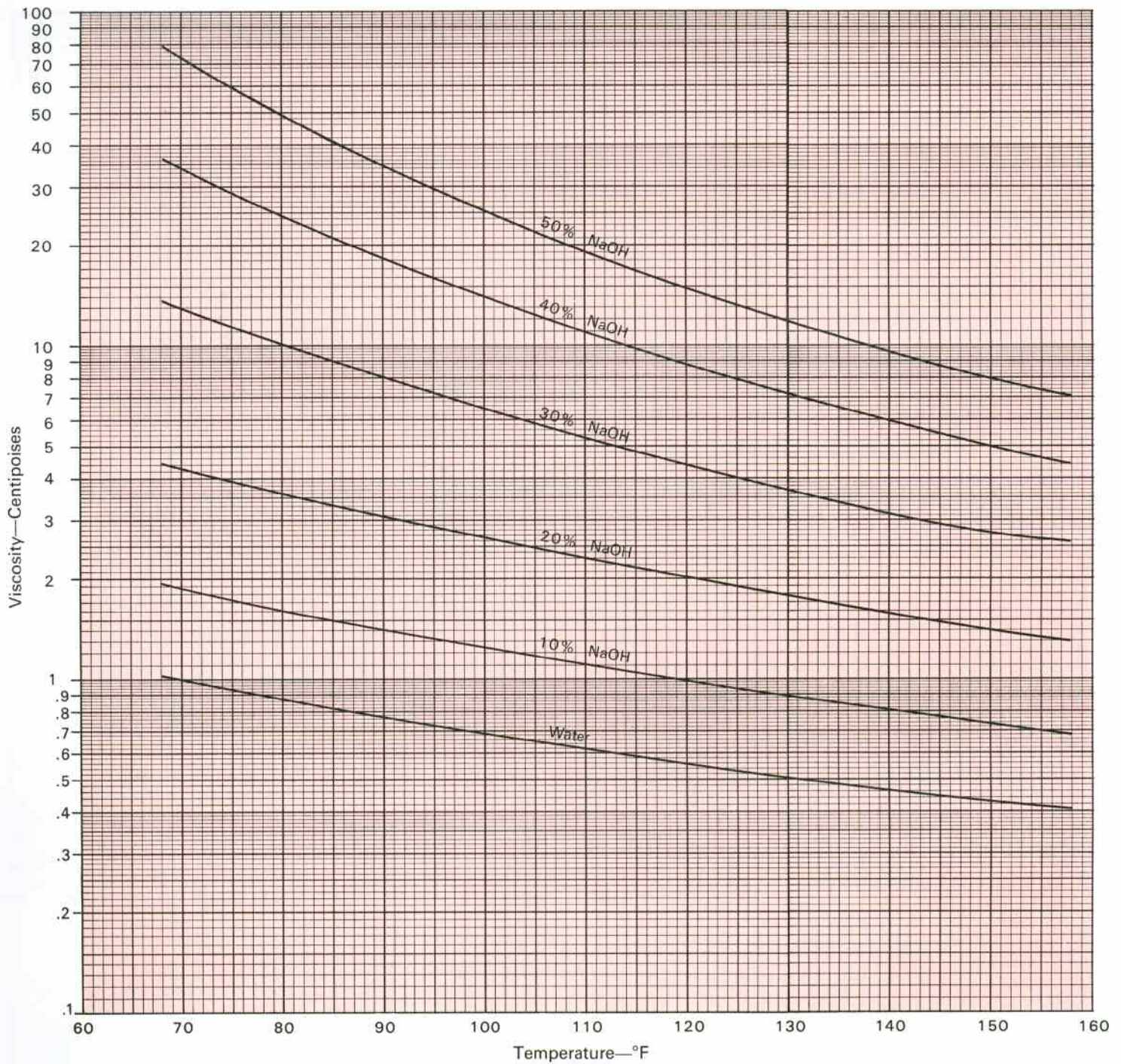
Viscosity of Caustic Soda Solutions						
Percent NaOH	Viscosity, centipoises					
	68°F 20°C	86°F 30°C	104°F 40°C	122°F 50°C	140°F 60°C	158°F 70°C
2	1.10	0.90	0.72	0.62	0.52	0.44
4	1.28	1.02	0.82	0.69	0.59	0.49
6	1.48	1.18	0.94	0.77	0.66	0.55
8	1.69	1.32	1.07	0.88	0.74	0.62
10	1.92	1.50	1.21	0.99	0.83	0.70
12	2.2	1.7	1.4	1.1	0.9	0.8
14	2.7	2.0	1.6	1.3	1.0	0.9
16	3.2	2.3	1.8	1.5	1.2	1.0
18	3.8	2.7	2.1	1.7	1.4	1.2
20	4.4	3.3	2.3	2.0	1.6	1.3
22	5.6	3.9	2.8	2.3	1.9	1.5
24	7.0	4.6	3.3	2.7	2.1	1.8
26	8.8	5.6	4.0	3.2	2.5	2.0
28	11.0	6.8	4.9	3.7	2.8	2.3
30	13.4	8.6	5.9	4.4	3.2	2.6
32	17.0	10.3	7.0	5.0	3.7	2.9
34	20.6	12.6	8.2	5.8	4.2	3.3
36	25.5	14.5	9.8	6.7	4.7	3.7
38	31.0	17.3	11.2	7.5	5.4	4.1
40	36.0	20.3	12.6	8.4	6.0	4.5
42	44.0	23.5	14.7	9.8	6.6	5.0
44	52.0	27.0	16.5	10.6	7.4	5.5
46	60.0	31.0	18.5	11.8	8.2	6.0
48	70.0	35.0	20.2	13.0	9.0	6.5
50	78.3	39.5	22.1	14.0	9.7	7.1

NaOH





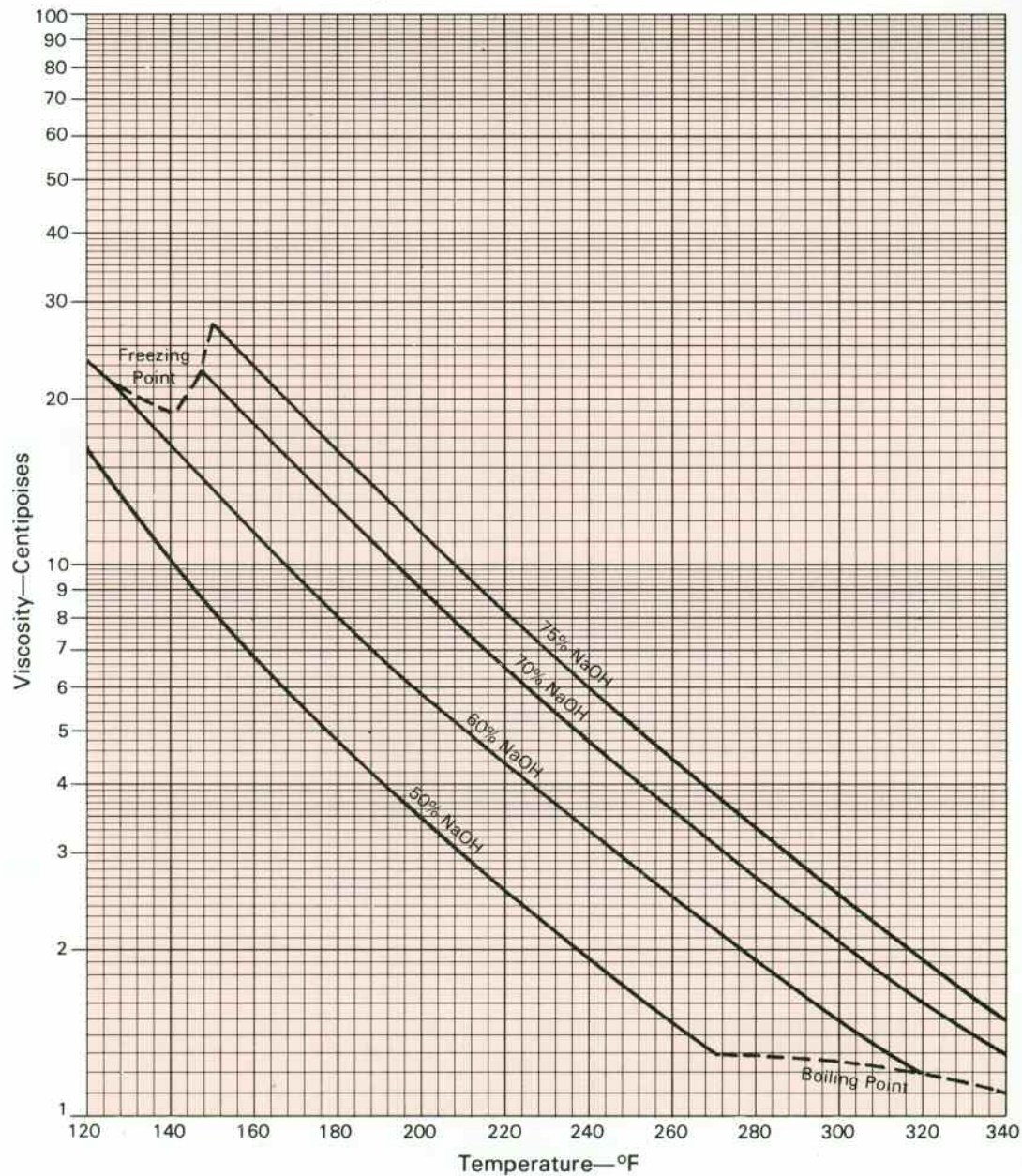
Viscosity of Caustic Soda Solutions







Viscosity of Caustic Soda Solutions



Viscosity of Caustic Soda Solutions

Percent NaOH	Viscosity, centipoises					
	140°F 60°C	180°F 82°C	220°F 104°C	260°F 127°C	300°F 149°C	340°F 171°C
50	10.0	4.8	2.6	1.5	Vapor	Vapor
60	16.7	7.9	4.3	2.5	2.0	1.3
70	Solid	13.0	6.5	3.6	2.5	1.5
75	Solid	16.7	8.0	4.4	2.5	1.5

# Technical Data



## Specific Heat of Caustic Soda Solutions

Percent NaOH	Specific Heat, Btu per Pound per Degree Fahrenheit															
	32°F	40°F	50°F	60°F	80°F	100°F	120°F	140°F	160°F	180°F	200°F	220°F	240°F	260°F	280°F	300°F
0	1.004	1.003	1.001	0.999	0.998	0.997	0.998	0.999	1.000	1.002	1.004	.....	.....	.....	.....	.....
2	0.965	0.967	0.968	0.969	0.972	0.974	0.977	0.978	0.980	0.983	0.986	.....	.....	.....	.....	.....
4	0.936	0.940	0.943	0.946	0.951	0.954	0.957	0.960	0.962	0.965	0.966	.....	.....	.....	.....	.....
6	0.914	0.920	0.924	0.928	0.933	0.938	0.941	0.944	0.946	0.948	0.950	.....	.....	.....	.....	.....
8	0.897	0.902	0.907	0.911	0.918	0.923	0.927	0.930	0.932	0.934	0.936	.....	.....	.....	.....	.....
10	0.882	0.888	0.893	0.897	0.905	0.911	0.916	0.918	0.920	0.922	0.923	.....	.....	.....	.....	.....
12	0.870	0.877	0.883	0.887	0.894	0.901	0.906	0.909	0.911	0.912	0.913	.....	.....	.....	.....	.....
14	0.861	0.868	0.874	0.879	0.886	0.892	0.897	0.901	0.903	0.903	0.904	.....	.....	.....	.....	.....
16	0.853	0.860	0.866	0.871	0.880	0.886	0.891	0.894	0.896	0.897	0.897	.....	.....	.....	.....	.....
18	0.847	0.854	0.860	0.865	0.873	0.880	0.885	0.888	0.890	0.891	0.891	.....	.....	.....	.....	.....
20	0.842	0.848	0.854	0.859	0.868	0.875	0.880	0.884	0.886	0.886	0.887	.....	.....	.....	.....	.....
22	0.837	0.844	0.849	0.854	0.863	0.870	0.876	0.880	0.882	0.882	0.883	.....	.....	.....	.....	.....
24	.....	0.839	0.844	0.849	0.858	0.866	0.873	0.877	0.879	0.879	0.880	.....	.....	.....	.....	.....
26	.....	0.835	0.840	0.845	0.854	0.863	0.869	0.874	0.875	0.876	0.876	.....	.....	.....	.....	.....
28	.....	0.830	0.836	0.841	0.850	0.859	0.866	0.870	0.872	0.872	0.873	.....	.....	.....	.....	.....
30	.....	0.826	0.832	0.837	0.846	0.855	0.862	0.866	0.868	0.869	0.869	.....	.....	.....	.....	.....
32	.....	0.822	0.828	0.833	0.842	0.850	0.857	0.862	0.863	0.864	0.864	.....	.....	.....	.....	.....
34	.....	.....	0.823	0.828	0.837	0.845	0.852	0.856	0.857	0.858	0.858	.....	.....	.....	.....	.....
36	.....	.....	0.819	0.824	0.832	0.840	0.845	0.849	0.850	0.851	0.851	.....	.....	.....	.....	.....
38	.....	.....	0.816	0.820	0.827	0.833	0.837	0.841	0.842	0.842	0.843	.....	.....	.....	.....	.....
40	.....	.....	0.812	0.815	0.821	0.826	0.829	0.831	0.832	0.832	0.832	.....	.....	.....	.....	.....
42	.....	.....	0.807	0.809	0.813	0.816	0.819	0.819	0.820	0.820	0.820	.....	.....	.....	.....	.....
44	.....	.....	.....	0.802	0.804	0.806	0.807	0.807	0.807	0.806	0.804	.....	.....	.....	.....	.....
46	.....	.....	.....	0.793	0.794	0.795	0.794	0.794	0.793	0.791	0.789	.....	.....	.....	.....	.....
48	.....	.....	.....	.....	0.783	0.782	0.781	0.780	0.779	0.777	0.776	.....	.....	.....	.....	.....
50	.....	.....	.....	.....	0.771	0.769	0.768	0.767	0.765	0.765	0.764	0.763	0.762	0.762	0.761	0.761
52	.....	.....	.....	.....	0.759	0.757	0.756	0.754	0.753	0.752	0.751	0.749	0.748	0.747	0.746	0.745
54	.....	.....	.....	.....	0.746	0.744	0.741	0.739	0.739	0.738	0.737	0.735	0.733	0.731	0.730	0.728
56	.....	.....	.....	.....	0.733	0.730	0.728	0.726	0.724	0.723	0.722	0.721	0.719	0.717	0.715	0.713
58	.....	.....	.....	.....	.....	0.719	0.717	0.715	0.713	0.711	0.709	0.707	0.705	0.703	0.702	0.700
60	.....	.....	.....	.....	.....	0.706	0.705	0.703	0.701	0.699	0.697	0.695	0.693	0.691	0.690	0.688
62	.....	.....	.....	.....	.....	.....	0.694	0.692	0.690	0.688	0.687	0.685	0.683	0.681	0.679	0.677
64	.....	.....	.....	.....	.....	.....	0.684	0.682	0.681	0.679	0.677	0.675	0.673	0.671	0.670	0.668
66	.....	.....	.....	.....	.....	.....	0.675	0.673	0.671	0.669	0.668	0.666	0.664	0.662	0.660	0.658
68	.....	.....	.....	.....	.....	.....	.....	0.663	0.662	0.660	0.658	0.656	0.655	0.653	0.651	0.649
70	.....	.....	.....	.....	.....	.....	.....	0.655	0.653	0.651	0.649	0.647	0.646	0.644	0.642	0.640
72	.....	.....	.....	.....	.....	.....	.....	.....	0.645	0.643	0.641	0.639	0.637	0.635	0.634	0.632
74	.....	.....	.....	.....	.....	.....	.....	.....	.....	0.635	0.633	0.631	0.629	0.628	0.626	0.624
76	.....	.....	.....	.....	.....	.....	.....	.....	.....	0.628	0.627	0.625	0.623	0.621	0.619	0.617
78	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	0.620	0.618	0.616	0.615	0.613	0.611

# Technical Data



## HOW TO USE HEAT CONTENT (ENTHALPY) CHART

Use of the chart on the facing page simplifies the calculation of many problems frequently encountered in diluting, cooling or heating caustic soda solutions such as:

1. How to determine the temperature of the final solution resulting from the dilution of a caustic soda solution, assuming no heat loss.
2. How to determine the amount of heat to remove from or add to a caustic soda solution to change its temperature a desired number of degrees.

### Examples

#### PROBLEM 1:

What will be the temperature of a 50 percent caustic soda solution prepared by diluting a 73 percent solution at 200°F with water at 80°F?

#### SOLUTION:

Draw a straight line between these two points on the chart: (1) the intersection of the 73 percent caustic soda line and the 200°F curve, and (2) the intersection of the 0 percent caustic soda line and the 80°F curve. The line drawn crosses the 50 percent caustic soda line at 263°F, which is the answer.

#### PROBLEM 2:

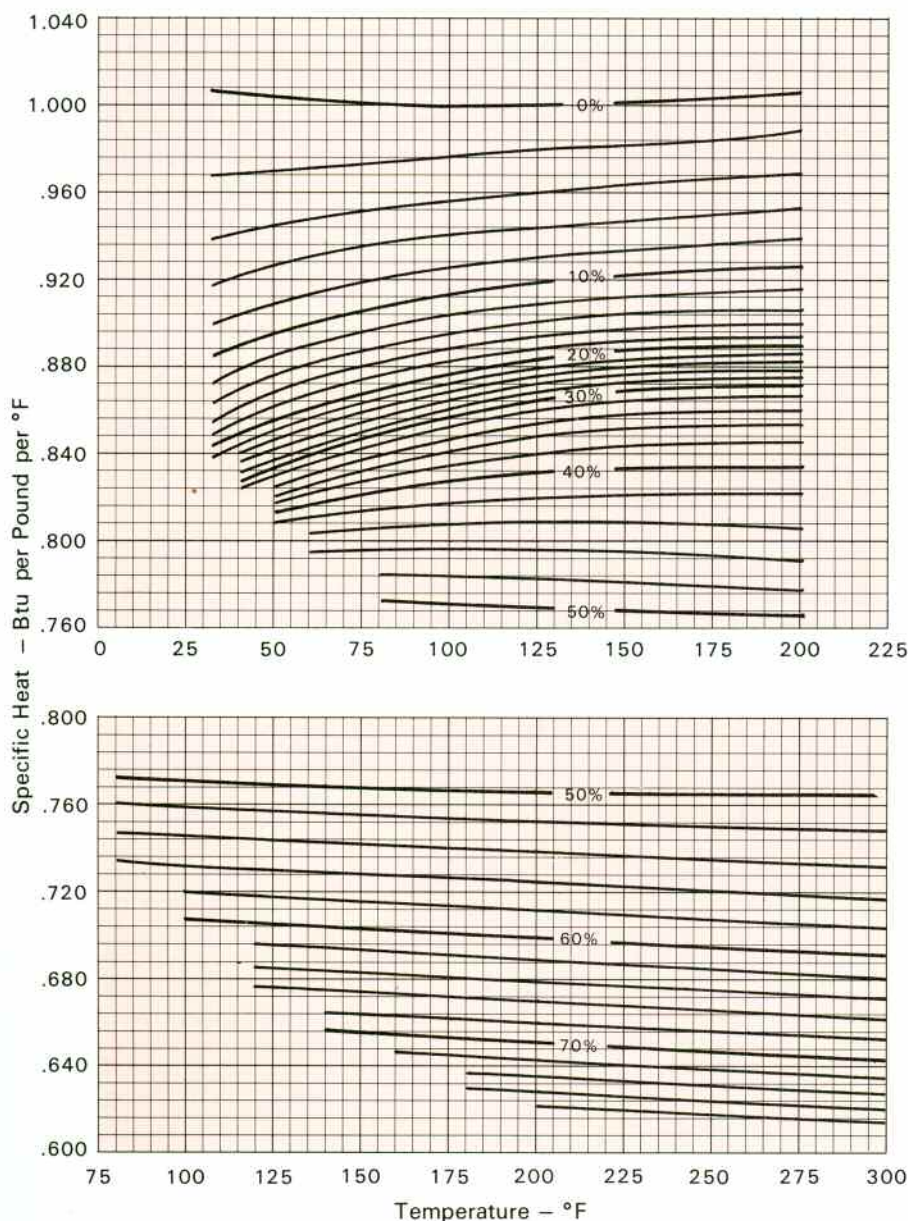
How many Btu per pound of solution must be removed to cool a 50 percent caustic soda solution from 263°F to 120°F?

#### SOLUTION:

The relative heat contents of a 50 percent caustic soda solution at 263°F and at 120°F appear on the chart at the 272 and 162 heat content lines

respectively. The difference between these heat content values, 100 Btu per pound of solution, is the answer.

Specific Heat of Caustic Soda Solutions

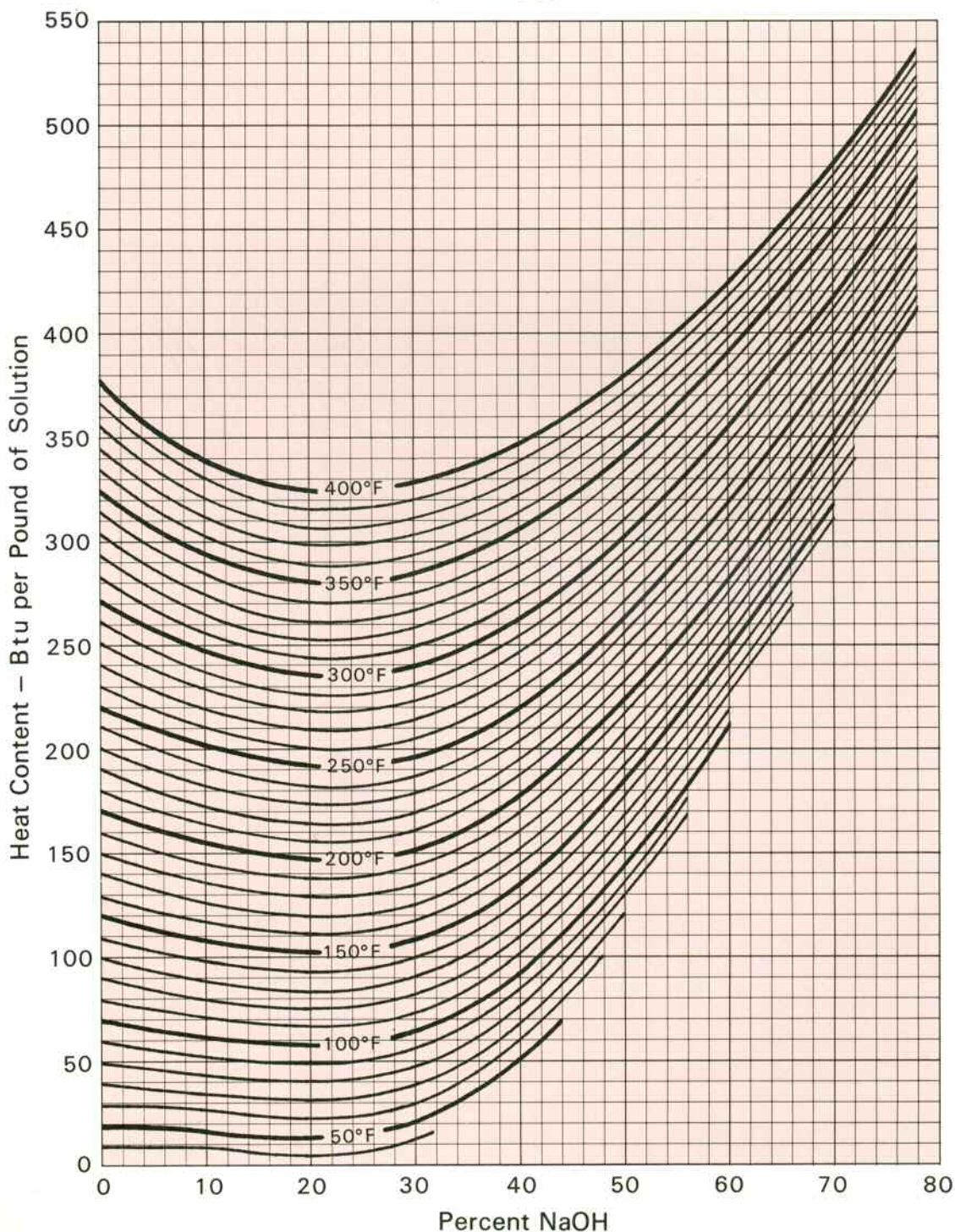


Derived from data of Bertetti and McCabe, "Industrial Engineering Chemistry," Volume 28, page 378, and McCabe and Wilson, "Industrial Engineering Chemistry," Volume 34, page 558.





Relative Heat Content (Enthalpy) of Caustic Soda Solutions

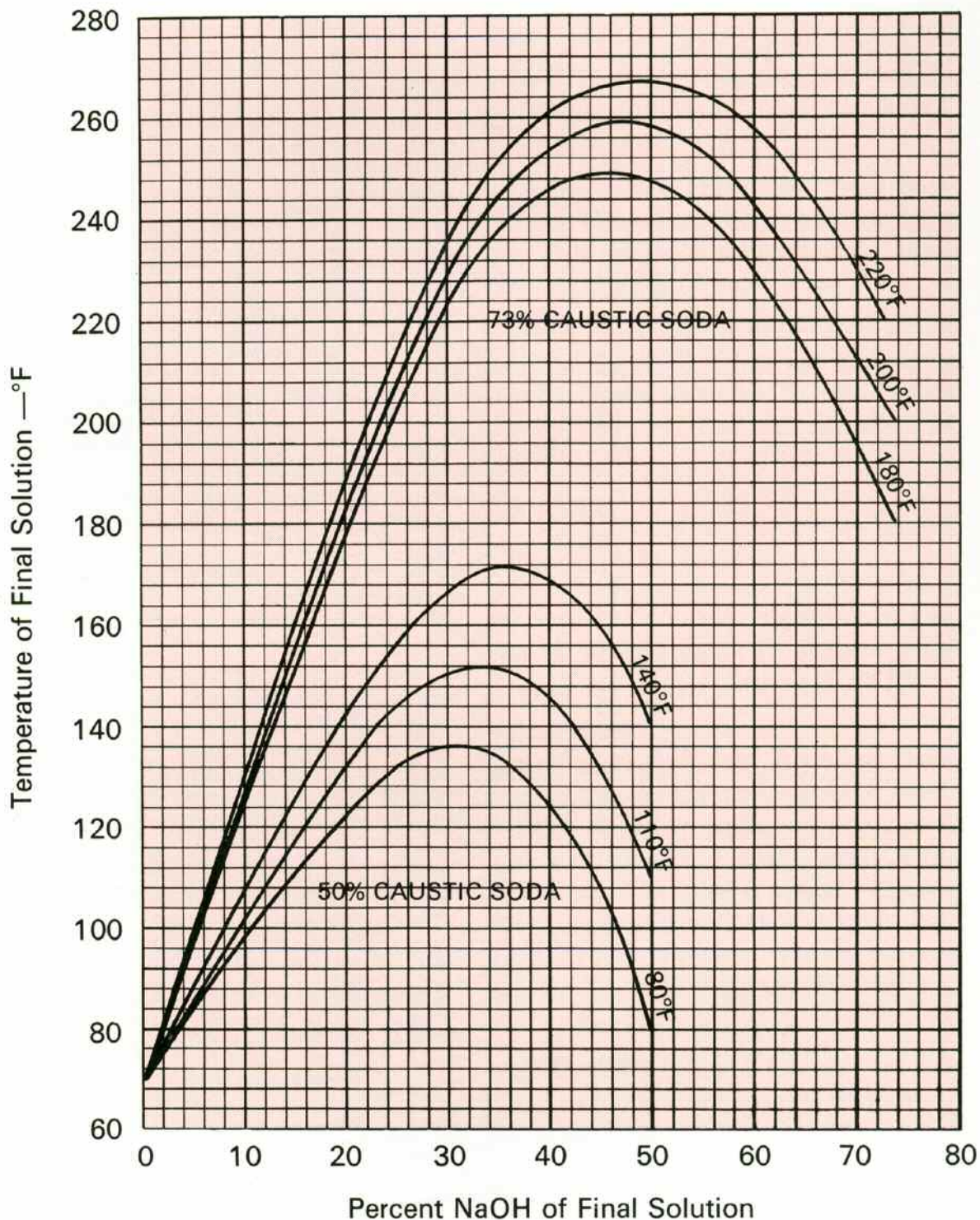


Derived from data of Bertetti and McCabe, "Industrial Engineering Chemistry," Volume 28, page 378, and McCabe and Wilson, "Industrial Engineering Chemistry," Volume 34, page 558.





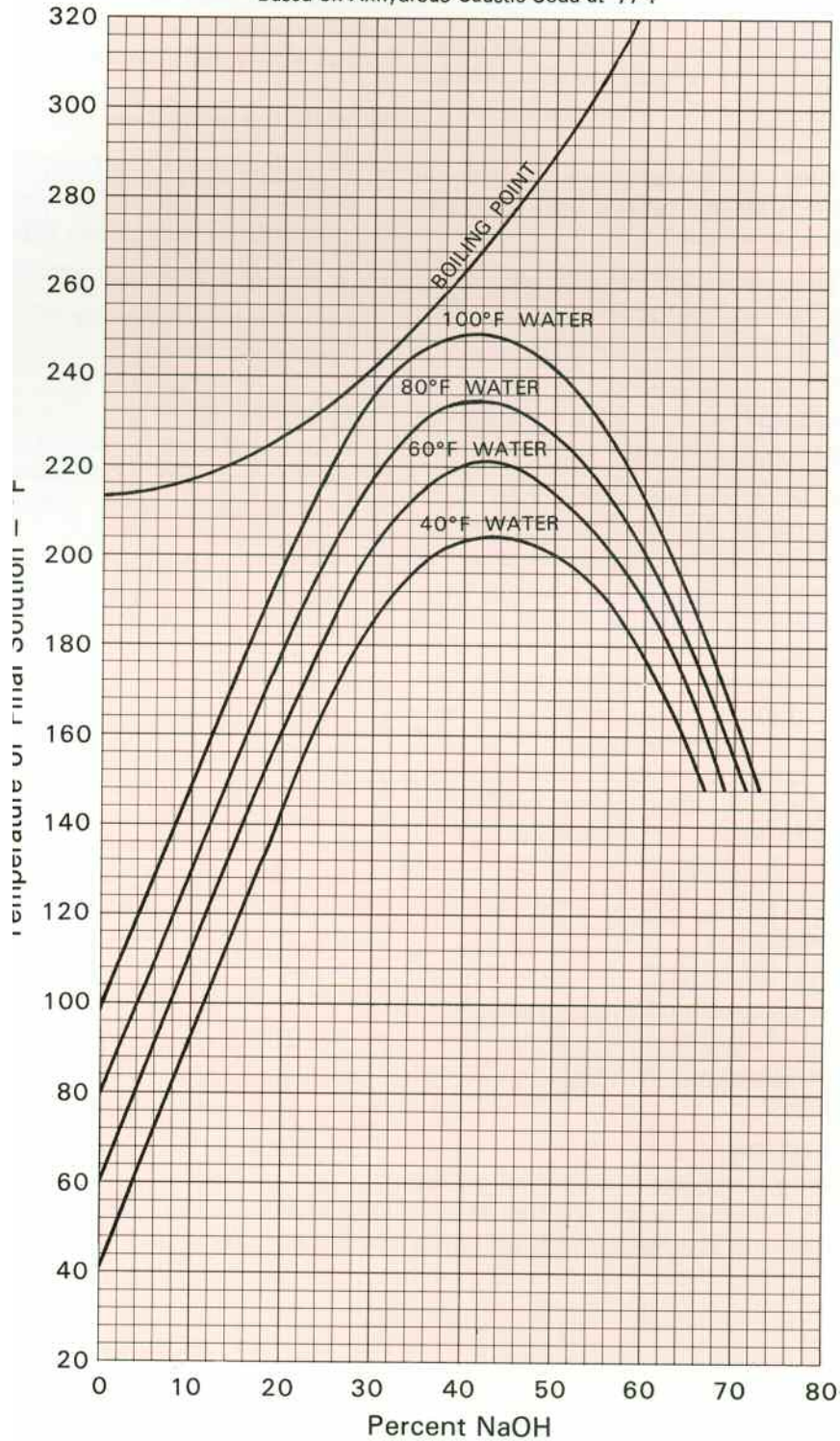
Dilution Temperatures of Caustic Soda Solutions  
Based on Diluting Water Temperature of 70°F





Dilution Temperatures of Anhydrous Caustic Soda

Based on Anhydrous Caustic Soda at 77°F





# Technical Data



## HOW TO USE CHART FOR DILUTING AND MIXING CAUSTIC SODA SOLUTIONS

Use of the chart on the facing page simplifies the calculation of problems such as:

1. How much water is needed to dilute a strong caustic soda solution to a weaker one?
2. How many gallons of strong solution are needed to bring a weak solution to a higher concentration?
3. What is the strength of a solution formed by mixing equal volumes of water and a caustic soda solution?

The left-hand line of the chart, zero percent, is of course water. The decreasing heights from left to right of the vertical lines for percentage concentration reflect the greater weight per gallon of stronger solutions. The chart does not take into consideration differences in initial temperatures of solutions and water as well as volumetric changes caused by heat of dilution since these conditions usually have relatively small effects on final volume.

### Examples

#### PROBLEM 1 (a):

How many gallons of water should be added to dilute 16,000 gallons of 73 percent caustic soda solution to 50 percent?

#### SOLUTION:

Draw a line from zero percent on the weak solution scale at top to 73 percent on the strong solution scale at bottom. From the point where the drawn line intersects the 50 percent vertical line, read 44 gallons of weak solution needed at right and 56 gallons of strong solution needed at left.

Multiply the percentage ratio,

$$\frac{44 \text{ gallons weak solution}}{56 \text{ gallons strong solution}}$$

$$\times 16,000 \text{ gallons strong solution} = 12,571 \text{ gallons weak solution}$$

The answer is the number of gallons of water to be added.

#### PROBLEM 1 (b):

How many gallons of water should be added to dilute 1,000 gallons of 50 percent caustic soda solution to 19 percent? (The latter concentration has the lowest freezing point and can withstand storage temperatures down to approximately  $-18^{\circ}\text{F}$ .)

#### SOLUTION:

Draw a line from zero percent on the weak solution scale at top to 50 percent on the strong solution scale at bottom. From the point where the

drawn line intersects the 19 percent vertical line, read 70 gallons of weak solution needed at right and 30 gallons of strong solution needed at left.

Multiply the percentage ratio,

$$\frac{70 \text{ gallons weak solution}}{30 \text{ gallons strong solution}}$$

$$\times 1,000 \text{ gallons strong solution} = 2,333 \text{ gallons weak solution}$$

The answer is the number of gallons of water to be added.

#### PROBLEM 2:

How many gallons of 50 percent caustic soda solution should be added to 300 gallons of 10 percent solution to obtain a final solution of 18 percent concentration?

#### SOLUTION:

Draw a line from 10 percent on the weak solution scale at top to 50 percent on the strong solution scale at bottom. From the point where the drawn line intersects the 18 percent vertical line, read 84 gallons of weak solution needed at right and 16 gallons of strong solution needed at left.

Multiply the percentage ratio,

$$\frac{16 \text{ gallons weak solution}}{84 \text{ gallons strong solution}}$$

$$\times 300 \text{ gallons weak solution} = 57 \text{ gallons strong solution}$$

The answer is the number of gallons of 50 percent caustic soda solution to be added.

# Technical Data



**PROBLEM 3:**

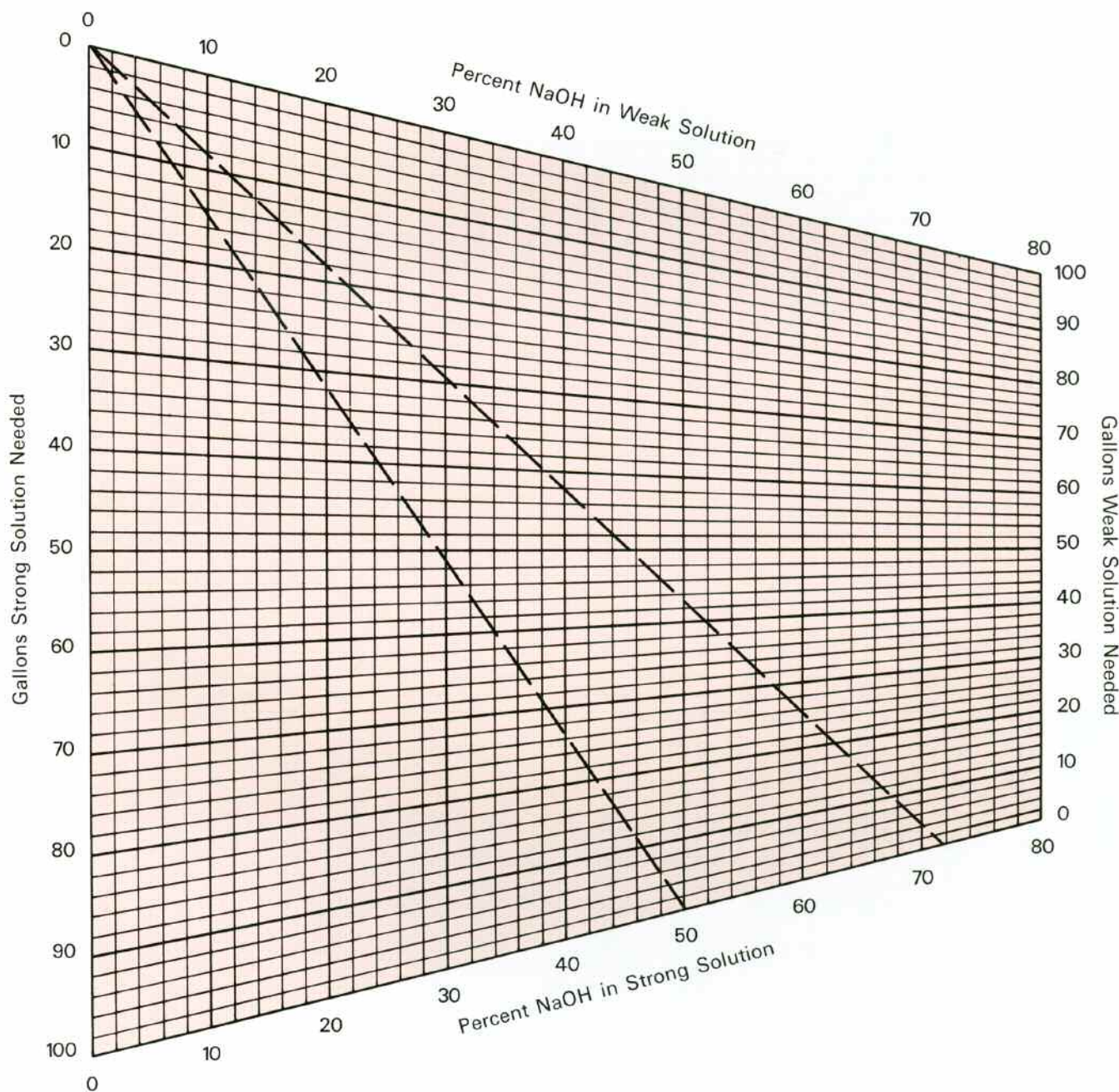
What is the strength of the solution formed by mixing 10,000 gallons of 40 percent caustic soda solution with 10,000 gallons of water?

**SOLUTION:**

Draw a line from zero percent on the weak solution scale at top to 40 percent on the strong solution scale at bottom. The horizontal line connecting 50 on both left and right scales represents the 50:50 percentage ratio of the 10,000-gallon volumes stated in the problem. The drawn line intersects the horizontal ratio line at the 23 percent vertical line, which is the concentration of the final solution.

sents the 50:50 percentage ratio of the 10,000-gallon volumes stated in the problem. The drawn line intersects the horizontal ratio line at the 23 percent vertical line, which is the concentration of the final solution.

**Diluting and Mixing Caustic Soda Solutions**





# Technical Data



## Alkali Conversion Tables

Equivalence of Sodium Oxide (Na<sub>2</sub>O), Caustic Soda (NaOH) and Soda Ash (Na<sub>2</sub>CO<sub>3</sub>)

Na <sub>2</sub> O	NaOH	Na <sub>2</sub> CO <sub>3</sub>	Na <sub>2</sub> O	NaOH	Na <sub>2</sub> CO <sub>3</sub>	Na <sub>2</sub> O	NaOH	Na <sub>2</sub> CO <sub>3</sub>	Na <sub>2</sub> O	NaOH	Na <sub>2</sub> CO <sub>3</sub>
1	1.2906	1.7099	21	27.10	35.91	41	52.91	70.11	61	78.73	104.30
2	2.58	3.42	22	28.39	37.62	42	54.20	71.82	63	81.31	106.01
3	3.87	5.13	23	29.68	39.33	43	55.50	73.53	63	81.31	107.72
4	5.16	6.84	24	30.97	41.04	44	56.79	75.24	64	82.60	109.43
5	6.45	8.55	25	32.26	42.75	45	58.08	76.94	65	83.89	111.14
6	7.74	10.26	26	33.56	44.46	46	59.37	78.65	55	84.18	112.85
7	9.03	11.97	27	34.85	46.17	47	60.66	80.36	67	86.47	114.56
8	10.32	13.68	28	36.14	47.88	48	61.95	82.07	68	87.76	116.27
9	11.61	15.39	29	37.43	49.59	49	63.24	83.78	59	89.05	117.98
10	12.90	17.10	30	38.72	51.30	50	64.53	85.49	70	90.34	119.69
11	14.20	18.81	31	40.01	53.01	51	65.82	87.20	71	91.63	121.40
12	15.49	20.52	32	41.30	54.72	52	67.11	88.91	72	92.92	123.11
13	16.78	22.23	33	42.59	56.43	53	68.40	90.62	73	94.21	124.82
14	18.07	23.94	34	42.88	58.14	54	69.69	92.33	74	95.50	126.53
15	19.36	25.65	35	45.17	59.85	55	70.98	94.04	75	96.79	128.24
16	20.65	27.36	36	46.46	61.56	56	72.27	95.75	76	98.08	129.95
17	21.94	29.07	37	47.75	63.27	57	73.56	97.46	77	99.38	131.66
18	23.23	30.78	38	49.04	64.98	58	74.85	99.17	77.48	100	132.49
19	24.52	32.49	39	50.33	66.69	59	76.14	100.88			
20	25.81	34.20	40	51.62	68.40	60	77.44	102.59			
NaOH	Na <sub>2</sub> O	Na <sub>2</sub> CO <sub>3</sub>	NaOH	Na <sub>2</sub> O	Na <sub>2</sub> CO <sub>3</sub>	NaOH	Na <sub>2</sub> O	Na <sub>2</sub> CO <sub>3</sub>	NaOH	Na <sub>2</sub> O	Na <sub>2</sub> CO <sub>3</sub>
1	.7748	1.3249	26	20.15	34.45	51	39.52	67.57	76	58.88	100.69
2	1.55	2.65	27	20.92	35.77	52	40.29	68.89	77	59.66	102.02
3	2.32	3.97	28	21.69	37.10	53	41.07	70.22	78	60.44	103.34
4	3.10	5.30	29	22.47	38.42	54	41.84	71.54	79	61.21	104.67
5	3.87	6.62	30	23.24	39.75	55	42.62	72.87	80	61.98	105.99
6	4.65	7.95	31	24.02	41.07	56	43.39	74.19	81	62.76	107.32
7	5.42	9.27	32	24.79	42.40	57	44.16	75.52	82	63.53	108.64
8	6.20	10.60	33	25.57	43.72	58	44.94	76.84	83	64.31	109.97
9	6.97	11.92	34	26.34	45.05	59	45.71	78.17	84	65.08	111.29
10	7.75	13.25	35	27.12	46.37	60	46.49	79.49	85	65.86	112.62
11	8.52	14.57	36	27.89	47.70	61	47.26	80.82	86	66.63	113.94
12	9.30	15.90	37	28.67	49.02	62	48.04	82.14	87	67.41	115.27
13	10.07	17.22	38	29.44	50.35	63	48.81	83.47	88	68.18	116.59
14	10.85	18.55	39	30.22	51.67	64	49.59	84.79	89	68.95	117.92
15	11.62	19.87	40	30.99	53.00	65	50.36	86.12	90	69.73	119.24
16	12.40	21.20	41	31.77	54.32	66	51.14	87.44	91	70.51	120.57
17	13.17	22.52	42	32.54	55.65	67	51.91	88.77	92	71.28	121.89
18	13.95	23.85	43	33.32	56.97	68	52.69	90.09	93	72.06	123.22
19	14.72	25.17	44	34.09	58.29	69	53.46	91.42	94	72.83	124.54
20	15.50	26.50	45	34.87	59.62	70	54.24	92.74	95	73.61	125.87
21	16.27	27.82	46	35.64	60.94	71	55.01	94.07	96	74.38	127.19
22	17.05	29.14	47	36.42	62.27	72	55.79	95.39	97	75.16	128.52
23	17.82	30.47	48	37.19	63.59	73	56.56	96.72	98	75.93	129.84
24	18.60	31.80	49	37.97	64.92	74	57.34	98.04	99	76.71	131.17
25	19.37	33.12	50	38.74	66.24	75	58.11	99.37	100	77.48	132.49



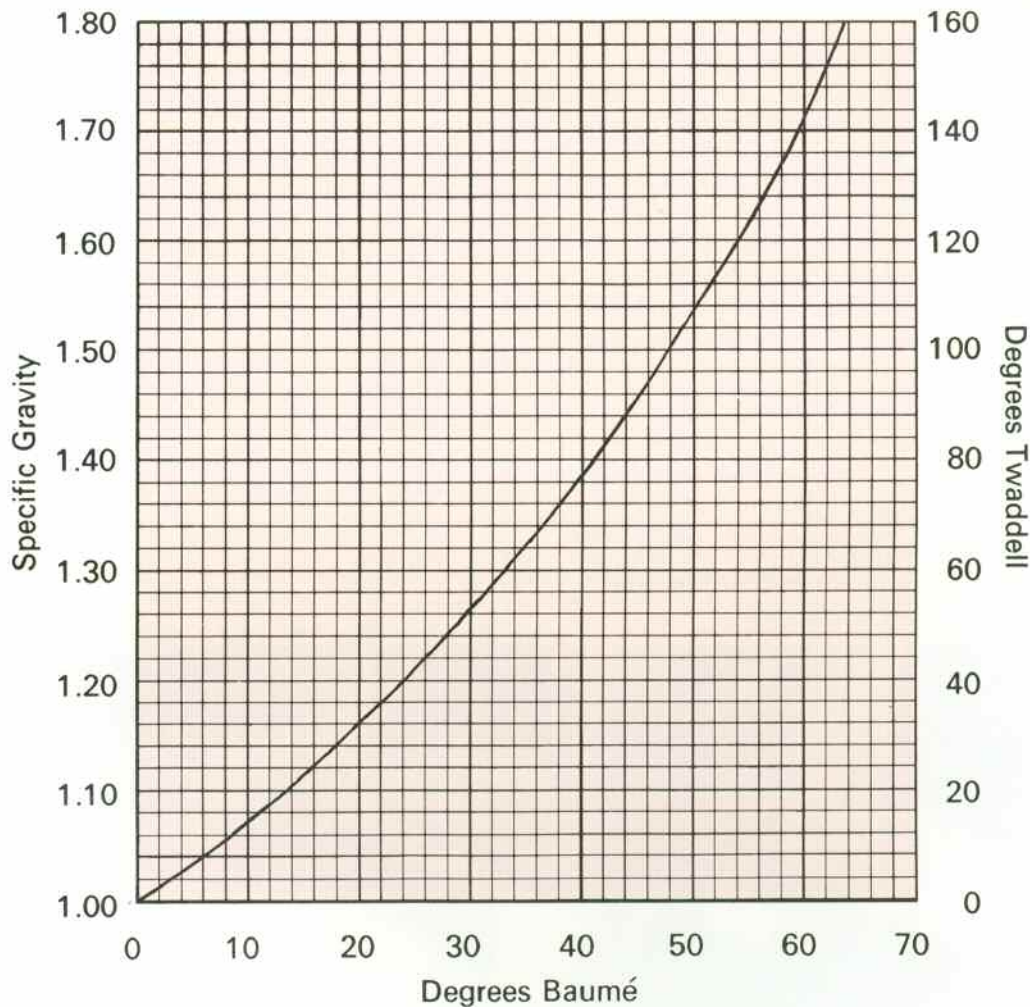
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Equivalence of Sodium Oxide ( Na<sub>2</sub>O), Caustic Soda (NaOH) and Soda Ash ( Na<sub>2</sub>CO<sub>3</sub>)

Na <sub>2</sub> CO <sub>3</sub>	Na <sub>2</sub> O	NaOH	Na <sub>2</sub> CO <sub>3</sub>	Na <sub>2</sub> O	NaOH	Na <sub>2</sub> CO <sub>3</sub>	Na <sub>2</sub> O	NaOH	Na <sub>2</sub> CO <sub>3</sub>	Na <sub>2</sub> O	NaOH
1	.5848	.7548	26	15.20	19.62	51	29.82	38.49	76	44.44	57.36
2	1.17	1.51	27	15.79	20.38	52	30.41	39.25	77	45.03	58.12
3	1.75	2.26	28	16.37	21.13	53	30.99	40.00	78	45.61	58.87
4	2.34	3.02	29	16.96	21.89	54	31.58	40.76	79	46.20	59.63
5	2.92	3.77	30	17.54	22.64	55	32.16	41.51	80	46.78	60.38
6	3.51	4.53	31	18.13	23.40	56	32.75	42.27	81	47.37	61.14
7	4.09	5.28	32	18.71	24.15	57	33.33	43.02	82	47.95	61.89
8	4.68	6.04	33	19.30	24.91	58	33.91	43.78	83	48.54	62.65
9	5.26	6.79	34	19.88	25.66	59	34.50	44.53	84	49.12	63.40
10	5.85	7.55	35	20.47	26.42	60	35.09	45.29	85	49.71	64.16
11	6.43	8.30	36	21.05	27.17	61	35.67	46.04	86	50.29	64.91
12	7.02	9.06	37	21.64	27.93	62	36.26	46.80	87	50.88	65.67
13	7.60	9.81	38	22.22	28.68	63	36.84	47.55	88	51.46	66.42
14	8.19	10.57	39	22.81	29.44	64	37.43	48.31	89	52.05	67.18
15	8.77	11.32	40	23.39	30.19	64	38.01	49.06	90	52.63	67.93
16	9.36	12.08	41	23.98	30.95	66	38.60	49.82	91	53.22	68.69
17	9.94	12.83	42	24.56	31.70	67	39.18	50.57	92	53.80	69.44
18	10.53	13.59	43	25.15	32.56	68	39.77	51.33	93	54.39	70.20
19	11.11	14.34	44	25.73	33.21	69	40.35	52.08	94	54.97	70.95
20	11.70	15.10	45	26.32	33.97	70	40.94	52.84	95	55.56	71.71
21	12.28	15.85	46	26.90	34.72	71	41.52	53.59	96	56.14	72.46
22	12.87	16.60	47	27.49	35.48	72	42.11	54.35	97	56.73	73.22
23	13.45	17.36	48	28.07	36.23	73	42.69	55.10	98	57.31	73.97
24	14.04	18.12	49	28.66	36.99	74	43.28	55.86	99	57.90	74.73
25	14.62	18.87	50	29.24	37.74	75	43.86	56.61	100	58.48	75.48



Conversion Chart for Hydrometer Readings



**Equations for Converting Hydrometer Readings Of Liquids Heavier Than Water**

**AMERICAN STANDARD BAUMÉ:**

$$\text{Specific Gravity} = \frac{145}{145 - \text{°Bé}}$$

$$\text{°Bé} = \frac{145(\text{sp gr} - 1)}{\text{sp gr}}$$

**TWADDELL:**

$$\text{Specific Gravity} = \frac{(0.5 \times \text{°Tw}) + 100}{100}$$

$$\text{°Tw} = (\text{sp gr} - 1)200$$

# Methods of Analysis



**C**autistic soda is a hazardous, reactive chemical. Before persons work with caustic soda, they should be instructed in safe handling practices and first aid, and should wear the recommended protective clothing and equipment (see chapter 3).

The analytical methods presented here were selected on the basis of their ability to provide the most accurate results consistent with simplicity of equipment and acceptable analytical practices. Many of the techniques were developed in PPG Industries' laboratories as improvements on classical methods. The procedures apply to both solution and anhydrous forms of caustic soda. The applicability to various PPG grades is listed under each method.

The laboratories of PPG Industries customarily determine trace elements in caustic soda by spectrometric procedures. On request, PPG personnel will share information on analytical procedures and techniques for determining certain trace substances not listed here.

Analytical determinations for the following substances appear in this chapter:

	Page
Sodium hydroxid total alkalinity . . . . .	54
Sodium carbonate (low concentrations) . .	56
Sodium chloride . . . . .	58
Sodium chloride (low concentrations) . .	59
Sodium sulfate . . . . .	60
Sodium sulfate (low concentrations) . .	60
Sodium chlorate . . . . .	61
Sodium chlorate (low concentrations) . .	62
Iron . . . . .	64

PPG practice is to report % NaOH and % Na<sub>2</sub>O on an "as is" basis, but all other compounds and metals on an "A.B." or anhydrous caustic soda basis so that a ready comparison can be made between 50 percent and 73 percent solutions.

## DETERMINATION OF SODIUM HYDROXIDE (NaOH), SODIUM CARBONATE (Na<sub>2</sub>CO<sub>3</sub>) AND SODIUM OXIDE (Na<sub>2</sub>O)—TOTAL ALKALINITY

### Double End Point Titration Method

#### Abstract

This method involves titration of the caustic soda sample by a dual end point titration technique, first to the phenolphthalein end point and then to the methyl orange-xylene cyanole end point. The alkali components are calculated from the titration data.

#### Application

NaOH, Na<sub>2</sub>CO<sub>3</sub> and Na<sub>2</sub>O in standard, low-iron and rayon grade caustic soda.

Na<sub>2</sub>O grade. Na<sub>2</sub>CO<sub>3</sub> in mercury cell grade is determined separately by the CO<sub>2</sub> evolution-conductometric method on page 56.

#### Precision

The precision of the method is ±0.10% NaOH, Na<sub>2</sub>CO<sub>3</sub> and Na<sub>2</sub>O by weight.

### Reagents

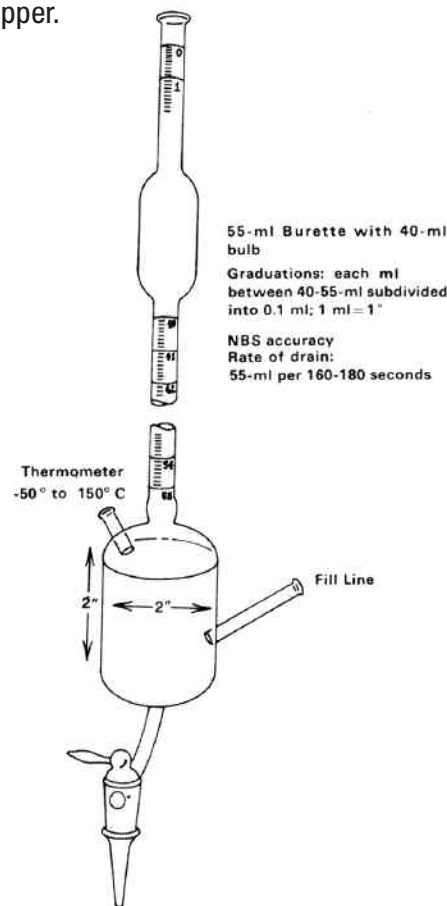
Standard 1.0N hydrochloric acid solution

Phenolphthalein indicator solution

Methyl orange-xylene cyanole indicator solution

### Procedure

Weigh a sample equivalent to approximately 2.0 g of anhydrous sodium hydroxide to 0.1 mg into a tared 10 ml Teflon or glass weighing bottle and stopper.



Volumetric burette for total alkalinity determination.



# Methods of Analysis



Measure approximately 40 ml of 1.0N hydrochloric acid solution (slightly less than the neutralization requirement of the sample) with a burette into a 250 ml beaker. Carefully remove the stopper from the weighing bottle and cautiously submerge both the stopper and weighing bottle in the acid solution. With the aid of a stirring rod, carefully manipulate the weighing bottle in such a manner that it is inverted with no air bubbles existing in the bottle until all the caustic is dissolved. Permit the bottle and stopper to remain in the beaker during the titration.

Add 2 drops of phenolphthalein indicator solution and continue titration with standard 1.0N hydrochloric acid solution from the burette until the pink color begins to fade.

Swirl the weighing bottle in the solution and carefully continue the titration to a colorless end point. Record the total volume of acid used in this titration (phenolphthalein end point) to the nearest 0.01 ml as value "A."

Add 2 drops of methyl orange-xylene cyanole indicator solution and continue titration to the steel-gray end point. Record the volume of acid used in the total titration to the nearest 0.01 ml as value "B."

Record the temperature of the acid used for titration and correct the volumes (values "A" and "B") to 20°C (see Temperature Correction Table, page 65).

## Calculation

$$\begin{aligned} & \% \text{ NaOH by weight} \\ &= \frac{[A - (B-A)] \times N \times 0.040 \times 100}{\text{Weight of Sample (as received)}} \end{aligned}$$

$$\begin{aligned} & \% \text{ Na}_2\text{O by weight} \\ &= \frac{B \times N \times 0.031 \times 100}{\text{Weight of Sample (as received)}} \end{aligned}$$

$$\begin{aligned} & \% \text{ Na}_2\text{CO}_3 \text{ by weight} \\ &= \frac{2(B-A) \times N \times 0.053 \times 100}{\text{Weight of Sample (as received)}} \end{aligned}$$

$$\begin{aligned} & \% \text{ Na}_2\text{CO}_3, \text{ A.B.*} \\ &= \frac{\% \text{ Na}_2\text{CO}_3 \text{ by weight} \times 100}{\% \text{ NaOH}} \end{aligned}$$

\*Anhydrous NaOH basis.

An alternate procedure for the NaOH and Na<sub>2</sub>CO<sub>3</sub> analyses is available which utilizes an automated titrator.

## Preparation of Reagents and Solutions

### 1. HYDROCHLORIC ACID 1.0N:

Dilute 83 ml of reagent grade concentrated hydrochloric acid, specific gravity 1.19, with distilled water to 1 liter in a volumetric flask and mix thoroughly.

**Standardization:** Weigh  $2 \pm 0.1$  g of anhydrous sodium carbonate (primary standard) to 0.1 mg into a 250 ml wide mouth Erlenmeyer flask, dissolve in 15 to 20 ml of distilled water, and add 2 drops of methyl orange-xylene

cyanole indicator solution. Titrate with approximately 1.0N hydrochloric acid to the end point.

$$\begin{aligned} & \text{Normality (N) of the HCl} \\ &= \frac{\text{g Na}_2\text{CO}_3 \times 1000}{53.00 \times \text{ml HCl used}} \end{aligned}$$

## 2. METHYL ORANGE-XYLENE CYANOLE INDICATOR SOLUTION

Dissolve 1.33 g of methyl orange and 3.00 g of xylene cyanole separately in distilled water. Mix them together when they are dissolved and dilute to 1 liter with distilled water.

**Note:** With this mixed indicator, alkaline solutions are green, acid solutions are purple-red, and neutral solutions are steel-gray.

## Special Equipment

A drawing of the 55 ml burette is shown on the previous page. The burette contains a 40 ml bulb. Each ml graduation between 40-55 ml is subdivided into 0.1 ml; 1 ml = 1 inch, NBS accuracy. Graduations are blue stripes on white background. The rate of drain is 55 ml per 160-180 seconds.

This burette is made under Sketch No. PPG 40476-G by SGA Scientific, Inc., 735 Broad Street, Bloomfield, NJ 07003, (201) 748-6600.



# Methods of Analysis



## DETERMINATION OF SODIUM CARBONATE ( $\text{Na}_2\text{CO}_3$ )

### $\text{CO}_2$ Evolution -

#### Conductometric Method

##### Abstract

The method involves the determination of sodium carbonate in caustic soda by conductometric measurement. The method entails the evolution of carbon dioxide from the sample by an acidification and purging process. The liberated carbon dioxide is absorbed in an excess of barium hydroxide solution. The change in electrical conductivity caused by the absorption of carbon dioxide to form barium carbonate is the means by which sodium carbonate is measured.

##### Application

Mercury cell grade caustic soda.

##### Precision

The precision of the method is  $\pm 0.003\%$   $\text{Na}_2\text{CO}_3$  by weight.

##### Special Apparatus and Operation

A conductance bridge is the principle item of special apparatus required for the method. The bridge should be capable of measuring differential resistances of 1 ohm in the desired concentration range.

Other items of apparatus required are common in most laboratories. The thermostated water bath (33) for regulating and controlling the temperature

of the conductivity cell (24) and barium hydroxide reservoir (30) should be of sufficient size and have sufficient capacity to function as a thermal regulator for these two items of apparatus. The temperature of this environment must be controlled closely. Operation at  $33 \pm 0.2^\circ\text{C}$  is recommended in order that precise and accurate results may be obtained by the procedure.

The general procedure first involves purging the system with nitrogen or air (free of carbon dioxide) for about 15 minutes prior to introducing a fresh barium hydroxide sample into the conductivity cell. The proper size sample of caustic soda is then pipetted into the dropping funnel (7) located above the reaction flask (10). The purge stream is attached to the dropping funnel and the sample forced into the reaction flask to provide minimum contamination due to carbon dioxide in the air during the transfer of the sample. The dropping funnel is washed well with distilled water with the aid of the purge stream, and the system is purged until a constant ohm reading is obtained from the conductivity cell. The acid is then introduced into the reaction flask in a similar manner as the sample, and the system is purged continuously for 15 minutes. The reaction flask is heated to  $60^\circ\text{C}$  during this period to aid in reducing the solubility of carbon dioxide in the acidified sample.

##### Analytical Procedure

In this procedure actual conductance or actual resistance of the solutions is measured. Before making any meas-

urements, check the temperature of the water bath and adjust it to  $33^\circ\text{C}$ . Clean the conductivity cell (24) with approximately 0.1N hydrochloric acid and rinse thoroughly with distilled water. Position the empty cell equipped with the electrodes and inlet and outlet tubes in the water bath and make the connections shown in the diagram. Purge the system for 15 minutes with a slow stream of nitrogen or air freed of carbon dioxide. Purge gas enters at (1) and leaves at (18).

Rinse the conductivity cell with the barium hydroxide solution by manipulation of the stopcocks and application of suction through tube (19).

Fill the conductivity cell with 0.014N barium hydroxide solution to some predetermined mark on the cell. This mark should be inscribed on the cell body so that fillings of the cell can be duplicated. The mark should be located so that the solution fills the cell a little more than halfway.

Add the solution of the weighed sample (equivalent to 6.0 g of anhydrous caustic soda) to the reservoir (7) and force it into the sample flask (10) by connecting the purge stream to the top of funnel (7) and opening stopcock (8). Wash the reservoir with three 15 ml portions of distilled water using the purge for forcing the washings into the reaction chamber. After the last washing, continue to purge the system at a rate of 0.1 standard liters per minute for 3 minutes and record initial ohm reading. Finally introduce 10 ml of dilute (1:1) sulfuric acid into the sam-

# Methods of Analysis



ple flask by way of the reservoir and purge stream. The sample must be acidified to a pH < 3.0. A 6.0-g sample of caustic soda containing 0.05% sodium carbonate (A.B.\*) should provide a differential resistance span of approximately 21 ohms in a conductivity cell with a 0.12 cell constant.

Adjust the purge gas flow to 0.1 standard liters per minute. This operation facilitates the transfer of any liberated carbon dioxide from the sample into the absorber solution through the fritted glass dispersion tube (17).

Continue this sweeping operation for 15 minutes. Then check the rate and measure the resistance of the absorber solution with the conductivity bridge.

Record the value and compute the change in resistance of this solution resulting from the formation of barium carbonate.

For results of greatest accuracy it is desirable to run a blank test with distilled water in place of the sample and make an appropriate blank correction.

When the determination is completed, remove the acidified sample solution by draining through stopcock (12) at the bottom of the flask. Rinse the flask thoroughly with distilled water in preparation for future tests. Remove "spent" barium hydroxide solution from the conductivity cell by suction through connection (19).

Consult the calibration chart (see below) to obtain the number of grams of sodium carbonate in the sample.

## Calculation

$$\begin{aligned} & \% \text{Na}_2\text{CO}_3 \text{ by weight} \\ & = \frac{\text{grams Na}_2\text{CO}_3 \text{ from chart} \times 100}{\text{Weight of Sample (as received)}} \end{aligned}$$

$$\begin{aligned} & \% \text{Na}_2\text{CO}_3, \text{ A.B.}^* \\ & = \frac{\text{Weight \% of Na}_2\text{CO}_3 \times 100}{\text{Weight \% NaOH in Sample Tested}} \end{aligned}$$

## Calibration of Conductivity Apparatus

Measure suitable increments ranging from 1 ml to 10 ml of the standard solution of sodium carbonate (see below) into the sample flask (10) and evolve the carbon dioxide into the absorber solution as described in the procedure above. The same purge rate (0.1 standard liters per minute) should be used in the calibration and in the sample procedure. Prepare a calibration chart for the apparatus from these data, relating the number of grams of sodium carbonate and the resistance span of the absorber solution.

## Special Reagents and Solutions

### 1. STANDARD SOLUTION OF SODIUM CARBONATE:

Dissolve 0.1000 g of reagent grade, anhydrous sodium carbonate in freshly boiled and cooled distilled water and dilute to 100 ml in a volumetric flask. One ml of this solution contains 0.001 g of sodium carbonate.

### 2. STANDARD BARIUM HYDROXIDE ABSORBER SOLUTION:

Dissolve 18.7 g of reagent grade barium hydroxide in 700 ml of boiling distilled water. After saturation, remove any undissolved solids by filtration and catch the filtrate in a suitably sized reagent bottle containing 6.9 l of distilled water which has previously been purged with a slow stream of nitrogen to expel carbon dioxide. Add a pinch of gelatin, stopper the reagent bottle and mix the solution thoroughly. The concentration of this solution should be about 0.014N and should be checked by titrating an aliquot of it with standardized 0.03N hydrochloric acid.

## DETERMINATION OF SODIUM CHLORIDE (NaCl)

### Volhard Method

#### Abstract

The chloride in caustic soda is determined according to the Volhard titration method. The method entails the acidification of the sample with nitric acid and the subsequent titration of the chloride with standard silver nitrate and standard potassium thiocyanate solutions.

#### Application

Standard, low-iron and rayon grade caustic soda (not applicable to mercury cell grade caustic soda).



# Methods of Analysis



## Precision

The precision of the method is  $\pm 0.10\%$  NaCl by weight.

## Reagents

Standard 0.1N silver nitrate solution (corrected to 20°C)

Standard 0.1N potassium thiocyanate solution (corrected to 20°C)

Concentrated nitric acid (C.P.), sp gr 1.42

Ferric nitrate indicator solution

## Procedure

Weigh a sample equivalent to approximately 10 g of anhydrous sodium hydroxide to 0.1 g into a 250 ml beaker, and add 35 ml of distilled water. Add 2 ml of ferric nitrate indicator solution and neutralize with concentrated nitric acid till iron precipitate dissolves. Then add 1 to 2 ml excess.

Add 0.2 ml of standard 0.1N potassium thiocyanate from a burette and titrate slowly with standard 0.1N silver nitrate solution until the red color disappears. Then add an excess of at least 2 ml. Stir vigorously until the silver chloride coagulates. Quantitatively filter the sample through a medium-porosity sintered-glass filter to remove the silver chloride precipitate. Wash the precipitate with 5% nitric acid until it is free of silver nitrate. Back-titrate the excess silver nitrate in the filtrate with standard 0.1N potassium thiocyanate solution until a faint rust-red

color persists. Compute the net volume in ml of standard silver nitrate solution consumed.

## Calculation

$$\begin{aligned} & \% \text{ NaCl by weight} \\ &= \frac{(\text{ml AgNO}_3 \times N) - (\text{ml KCNS} \times N)}{\text{Weight of Sample (as received)} \times 0.0585 \times 100} \end{aligned}$$

$$\begin{aligned} & \% \text{ NaCl, A.B.*} \\ &= \frac{\% \text{ NaCl by Weight} \times 100}{\% \text{ NaOH by Weight}} \end{aligned}$$

## Preparation of Special Reagents and Solutions

### 1. PREPARATION AND STANDARDIZATION OF SILVER NITRATE AND POTASSIUM THIOCYANATE:

#### Preparation of 0.1N Silver Nitrate:

Dissolve 17 g  $\pm 0.1$  g of silver nitrate in distilled water. Add 5.5 ml of concentrated nitric acid and dilute to 1 liter.

Preparation of 0.1N Potassium Thiocyanate: Dissolve 9.78 g  $\pm 0.1$  g potassium thiocyanate (A.R.) in distilled water and dilute to one liter.

If it is desirable to adjust the silver nitrate and potassium thiocyanate to exactly the same normality, titrate a quantity of the potassium thiocyanate with silver nitrate according to the procedure and determine the ratio of standard solution.

$$C = \frac{\text{ml AgNO}_3}{\text{ml KCNS}}$$

To dilute a solution with water:

$$\frac{A - D}{D} \times X = Y$$

To strengthen a weak solution with a stronger solution:

$$\frac{D - A}{C - D} \times Y = X$$

where A = Actual concentration of the solution that is to be corrected

D = desired concentration

C = concentration of strengthening solution

X = amount of stronger solution to be added, taken, or prepared

Y = amount of weaker solution or water to be added or taken

Standardization: Dry a small quantity of potassium chloride (A.R.) in the drying oven for 2 hours. Weigh 0.07 grams  $\pm 0.1$  mg of dry, cool potassium chloride into a weighing bottle. Dissolve the potassium chloride in water and wash it into a 250 ml beaker. Titrate according to the general procedure.

AgNO<sub>3</sub> Normality

$$= \frac{\text{Grams KC1}}{0.07456 [\text{ml AgNO}_3 - (\text{ml KCNS}) \times C]}$$

KCNS Normality = AgNO<sub>3</sub> Normality  $\times$  C

# Methods of Analysis



## 2. FERRIC NITRATE INDICATOR SOLUTION

Dissolve 100 g of ferric nitrate (A.R) in distilled water and add 100 ml nitric acid (C.P.) sp gr 1.42. Dilute to one liter.

**Note:** For assay work the solutions should be standardized on the day used and titrant volumes corrected to 20°C per table on page 65. An alternate procedure for the Vilhard salt method is available which utilizes an automated titrator.

## DETERMINATION OF LOW CONCENTRATIONS OF SODIUM CHLORIDE (NaCl)

### Turbidimetric Method

#### Abstract

The method involves the measurement of low concentrations of sodium chloride by a turbidimetric procedure. The method entails reaction of the acidified sample with an excess of silver nitrate. The resulting turbidity due to silver chloride is determined spectrophotometrically.

#### Application

Mercury cell grade caustic soda.

#### Precision

The precision of the method is  $\pm 0.002\%$  NaCl by weight.

#### Reagents

Phenolphthalein indicator solution

Dilute nitric acid (1:1)

Standard silver nitrate solution (0.1N)

#### Procedure

Weigh a sample equivalent to approximately 5 g of anhydrous sodium hydroxide to 0.1 g into a 250 ml beaker. Dilute the sample with 20 ml of distilled water, chloride-free. Add 1 drop of phenolphthalein indicator solution and neutralize the sample solution cautiously by the addition of dilute (1:1) nitric acid. Render the solution slightly acidic by adding 1 drop of acid in excess.

Transfer the solution to a 100 ml volumetric flask, cool to room temperature, and dilute to the mark with distilled water. Prepare another 100 ml volumetric flask filled to the mark with distilled water containing the same amount of phenolphthalein indicator and excess acid as used in the sample above. This solution serves as a blank.

Add 1 ml of diluted (1:1) nitric acid solution to both the sample and blank solutions in the flasks. Mix thoroughly. Add 1 ml of 0.1N silver nitrate solution to each flask and mix by inverting once only, then set aside in the dark for 15 minutes. The total volume of the sample and standard solutions in the 100 ml flasks is 102.0 ml. It is not necessary to standardize the silver nitrate solution volumetrically for this test.

At the end of the digestion period transfer a sufficient amount of the blank solution to the 5 cm comparison cell of the spectrophotometer and measure the light transmittance of the blank at 425 ml. Record the transmittance value.

Repeat this measurement with the prepared sample solution, and record the transmittance value. Compute the grams of chloride in the sample (blank-corrected) from a previously prepared calibration for the spectrophotometer (see below).

#### Calculation

$$\begin{aligned} \% \text{ NaCl by weight} \\ &= \frac{\text{Grams of Cl}^- \text{ from Chart} \times 1.6483 \times 100}{\text{Weight of Sample (as received)}} \end{aligned}$$

$$\begin{aligned} \% \text{ NaCl, A.B.*} \\ &= \frac{\% \text{ NaCl by weight} \times 100}{\% \text{ NaOH by weight}} \end{aligned}$$

## Calibration of Spectrophotometer

### Standard Chloride Solution:

Weigh 2.1143 grams of oven-dried, reagent-grade potassium chloride and transfer to a 1000 ml volumetric flask. Dissolve the salt in distilled water and dilute to the mark. Mix thoroughly.

Measure a 10 ml aliquot portion of this solution into another 1000 ml volumetric flask and dilute to the mark with distilled water. Mix thoroughly. One ml of the resulting solution contains 0.00001 g of chloride.

\*Anhydrous NaOH basis.

# Methods of Analysis



## Standardization Procedure

Prepare a series of standards by measuring 1.0, 3.0, 5.0 and 10.0 ml of the standard chloride solution respectively into a series of 100 ml volumetric flasks. Dilute each standard to 100 ml with distilled water and mix thoroughly. Proceed with the turbidity development and measurement according to the directions given above for the samples. Conduct the turbidimetric measurements versus distilled water in the reference cells. From these data prepare a calibration curve.

## DETERMINATION OF IRON (Fe)

### *o*-Phenanthroline Colorimetric Method

#### Abstract

The method involves the determination of iron in caustic soda by the spectrophotometric measurement of the iron ortho-phenanthroline complex. The method entails acidification of the sample with hydrochloric acid, treatment with a solution of hydroxylamine hydrochloride to reduce the iron to Fe(II), adjustment of the pH value of the solution, and the addition of the complexing agent orthophenanthroline solution. The color of the solution is measured spectrophotometrically at a wavelength of 510 m $\mu$ . The procedure is standardized and performed in a sodium chloride matrix.

## Application

All grades of caustic soda.

## Precision

The precision of the method is  $\pm 0.00005\%$  Fe by weight.

## Reagents

Concentrated hydrochloric acid (C.P.), sp gr 1.19

Hydroxylamine hydrochloride solution, 10% W/V

Acetate buffer solution

*o*-Phenanthroline solution, 1% W/V

2,4-Dinitrophenol indicator solution, 0.1% W/V

Sodium chloride crystals (purified grade)

## Procedure

Weigh a sample equivalent to 5.0 g of anhydrous sodium hydroxide to 0.1 g into a 125 ml beaker and dilute to 50 ml with distilled water. Add 0.5 ml of dinitrophenol indicator solution and neutralize the sample with concentrated hydrochloric acid. Cool the solution to room temperature and then render slightly acidic by the addition of two drops of concentrated hydrochloric acid. Transfer the slightly acidic solution to a 100 ml volumetric flask and reserve.

Since the spectrophotometric measurements are made against a reagent blank containing 7.3 g of sodium chloride, its preparation should be initiated at this point in the analysis.

Weigh 7.3 g of purified sodium chloride (iron-free) into a 100 ml volumetric flask and dissolve in approximately 50 ml of distilled water. Add 0.5 ml of dinitrophenol indicator solution and neutralize by dropwise additions of concentrated hydrochloric acid or ammonium hydroxide, then render the solution slightly acidic by adding two drops of concentrated hydrochloric acid.

To both the sample and blank, add 1 ml of 10 percent hydroxylamine hydrochloride solution and mix thoroughly. Allow the solutions to digest 10 minutes to effect reduction of the iron to the ferrous state. After reaction add 10 ml of the acetate buffer solution and mix thoroughly. The pH of this solution should be between 4 and 5. Add 5 ml of ortho-phenanthroline solution, dilute to 100 ml with distilled water and mix the solutions thoroughly.

Allow the solutions to stand for 15 minutes, then transfer an appropriate volume of the prepared solutions to a 5 cm comparison cell of the spectrophotometer. Measure the transmittance of the sample at 510 m $\mu$  as compared with the blank.

Consult the calibration curve for the spectrophotometer to obtain the number of grams of iron in the sample.

\*Anhydrous NaOH basis.



# Methods of Analysis



## Calculation

% Fe by weight

$$= \frac{\text{g Fe from Calibration Curve} \times 100}{\text{Weight of Sample (as received)}}$$

Fe, A.B.\*

$$= \frac{\% \text{ Fe by weight} \times 100}{\% \text{ NaOH by weight}}$$

## Calibration of Spectrophotometer

Place 7.3 g of pure sodium chloride (iron-free) into a series of 100 ml volumetric flasks and dissolve the salt in approximately 30 ml of distilled water. Measure 0.0, 0.5, 1.0, 2.0, 4.0, 7.0, and 10.0 ml respectively of the standard iron solution into the flasks.

Add 0.5 ml of dinitrophenol indicator solution and neutralize by the dropwise addition of ammonium hydroxide, then render the solution slightly acidic by the addition of 2 drops of concentrated hydrochloric acid. Proceed with the preparation of the standards as described in the procedure above. Construct a calibration curve from the calibration data.

## Special Reagents and Solutions

### 1. STANDARD IRON SOLUTION:

Dissolve 0.1000 g of iron (A.R.) in a mixture of 20 ml of hydrochloric acid and 50 ml of distilled water. Heat the mixture gently to expedite solution of the iron metal. When the solution is dissolved, dilute it to 1 liter with distilled water and mix. Dilute a 100-ml

aliquot of this solution to 1000 ml and mix. One ml of the resulting solution contains 0.00001 g of iron.

### 2. ACETATE BUFFER SOLUTION:

Dissolve 272 grams of sodium acetate trihydrate in 500 ml of distilled water. Add 250 ml of glacial acetic acid, cool and dilute to one liter with distilled water.

### 3. o-PHENANTHROLINE SOLUTION, 1% (W/V):

Dissolve 5.0 g of 1,10-ortho-phenanthroline in methyl alcohol and dilute to 500 ml with alcohol. Store this solution in a glass-stoppered amber bottle. Prepare fresh solution each 3 to 4 weeks.

### 4. 2,4-DINITROPHENOL INDICATOR SOLUTION 0.1% (W/V):

Dissolve 0.1 g of 2,4-dinitrophenol in distilled water and dilute to 100 ml.

### 5. HYDROXYLAMINE HYDROCHLORIDE SOLUTION, 10% (W/V):

Dissolve 50 g of hydroxylamine hydrochloride in distilled water and dilute to 500 ml. An alternative method is available utilizing optical emission spectrography.

## Volumetric Temperature Correction Table

Water and 0.1N		
Temperature, °C	Solutions, ml/l <sub>1</sub>	1 N HCl <sub>2</sub>
15	+0.77	+0.97
16	+0.64	+0.79
17	+0.50	+0.61
18	+0.34	+0.41
19	+0.18	+0.21
20	0	0
21	-0.18	-0.22
22	-0.38	-0.44
23	-0.59	-0.67
24	-0.81	-0.91
25	-1.03	-1.17
26	-1.27	-1.43
27	-1.52	-1.70
28	-1.77	-1.98
29	-2.04	-2.26
30	-2.31	-2.55

<sup>1</sup> "Handbook of Chemistry and Physics," 48th ed, The Chemical Rubber company, Cleveland, Ohio, 1967-1968, p F2.

<sup>2</sup> "Chemical Annual," 7th ed, D.Van Nostrand Company, Inc., Princeton, New Jersey, 1934, p 71. This table gives the correction to be added per liter to the observed volume of water, or standard 0.1N solution, to give the volume at the standard temperature 20°C. Conversely, by applying the corrections to the volume desired at 20°C, the volume that must be measured out at the designated temperature in order to give the desired volume at 20°C will be obtained. The volumes are measured in glass apparatus having a coefficient of cubical expansion of 0.000025 per degree C.

A formula for calculating volumetric temperature corrections appears in "ASTM Standards," Part 22, 1968, Designation E-200-67, page 457.

\*Anhydrous NaOH basis.

# Uses of Caustic Soda



**C**austic soda is a hazardous, reactive chemical. Before persons work with caustic soda, they should be instructed in safe handling practices and first aid, and should wear the recommended protective clothing and equipment (see chapter 3).

Caustic soda has a host of different uses. Its major markets are the chemical and pulp and paper industries, but considerable quantities of caustic soda are used to produce rayon, aluminum, soap, textiles and petroleum. Other high-volume uses include detergents and cleaners, boiler compounds, paint-and varnish-removers, water treatment, food processing and leather manufacturing.

Because of inherent processing advantages, caustic soda is replacing soda ash (sodium carbonate) as a source of sodium oxide in several uses. These include the manufacture of glass, paper pulp, phosphates and silicates, and water-treatment.



*Caustic soda is important in the manufacture of aluminum.*



*Chemical processing is the major user of caustic soda.*



*Caustic soda is a major processing chemical for pulp and paper.*

# NaOH

# Methods of Manufacture

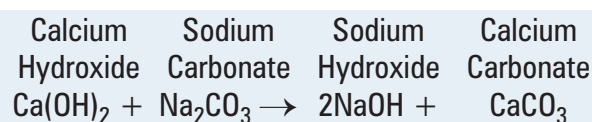


**C**autistic soda is a hazardous, reactive chemical. Before persons work with caustic soda, they should be instructed in safe handling practices and first aid, and should wear the recommended protective clothing and equipment (see chapter 3).

## LIME-SODA PROCESS

The lime-soda process was the only commercial method of manufacturing caustic soda until 1890, when processes for simultaneous production of caustic soda and chlorine by the electrolysis of brine were introduced. Lime-soda process techniques improved over the centuries from the time soap was made in ancient days, but every process still depended on the reaction of slaked lime (calcium hydroxide) with soda ash (sodium carbonate) to produce caustic soda (sodium hydroxide) and calcium carbonate.

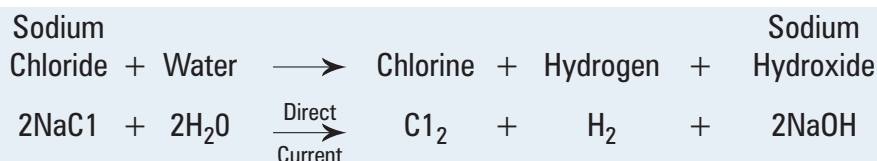
In chemical notation:



## DIAPHRAGM CELL PROCESS

After 1890, the number of electrolytic chlor-alkali plants increased until, by 1940, most caustic soda was produced by the electrolysis of brine.

The diaphragm cell method is a one-step process. The over-all reaction is:



The earlier diaphragm cells consisted of a shell having a concrete bottom, a steel mid-section, and a concrete top. The electrodes consisted of a graphite anode and a steel wire mesh cathode. With the conversion to the dimensionally stable anodes (DSA) in the early 1970s and improved materials of construction, several changes in diaphragm cell design occurred. Instead of using concrete tops and bottoms, the modern diaphragm cells are constructed with steel bottoms and plastic tops. The DSA anodes are titanium with an electrolytic coating. The cathode is still steel but in selected cases an electrolytic coating is used. The conversion to DSA anodes provided a significant reduction in the cell power consumption. The diagram on the facing page shows a design of a modern monopolar diaphragm cell.

The anode and cathode are separated by a "diaphragm" made from a deposited layer of asbestos fiber that coats each cathode. The diaphragm serves to keep the caustic soda and hydrogen separated from the anolyte, but it also has an additional function by controlling the flow of electrolyte to the cathode. In this manner, the optimum efficiency in

formation of caustic and chlorine can be maintained.

The electrolyte in the cell is a solution of sodium chloride. Natural salt, however, contains varying quantities of calcium, magnesium and other impurities. The brine is treated to remove these contaminants by precipitation. The brine is then filtered to remove the precipitates and any other undissolved solids that may be present. At this stage the brine has too low a concentration of sodium chloride for efficient cell operation. It is brought to the desired strength either by addition of purified salt or by evaporation of some of the water.

For convenience, cells are arranged in series electrically, each circuit consisting of several rows of cells. Two rows share a brine distribution line and collecting systems for chlorine and hydrogen. On the aisle side of each row is a pipe line for collecting caustic. Brine flow to each cell is individually controlled.

When direct current electricity flows between anodes and cathodes and through the brine, chlorine collects at the anode plates and hydrogen collects inside the cathode screen. Sodium combines with the hydroxyl

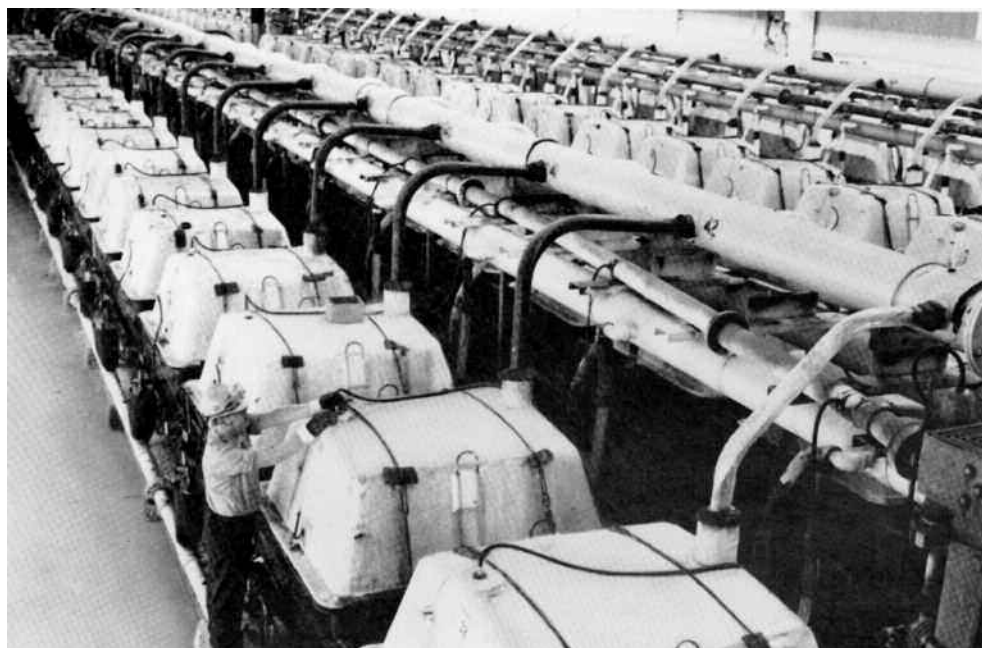


# Methods of Manufacture

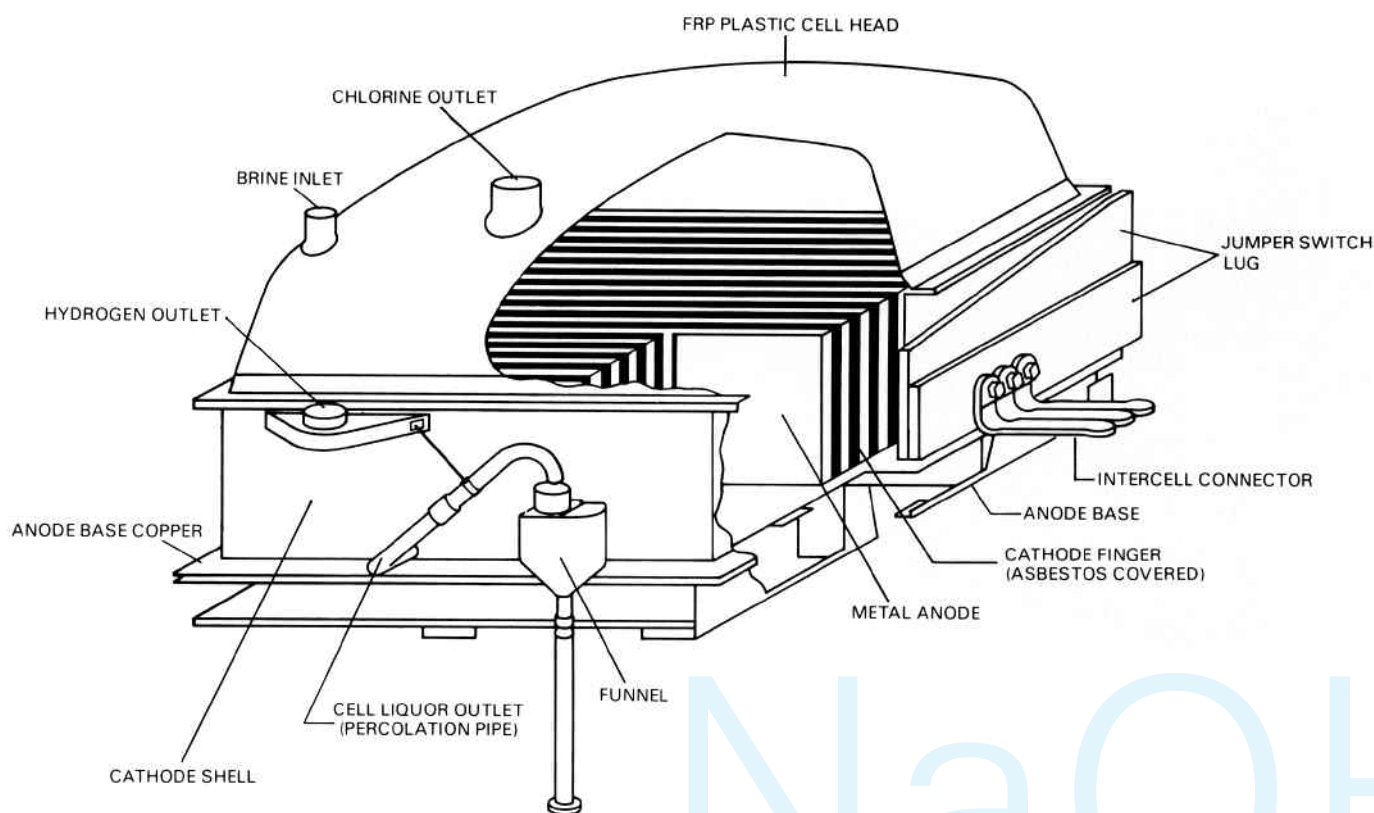


ion of water to form caustic soda (sodium hydroxide) at the cathodes. The chlorine bubbles up through the brine and is carried away by the chlorine-collecting system. Similarly, hydrogen is collected from the cathode section. The caustic solution is drawn off from the cell's lower side.

The caustic solution as it comes from the cell is approximately 12 percent sodium hydroxide. The solution is evaporated to 50 percent or 73 percent sodium hydroxide. The latter can be further concentrated to the anhydrous state and sold in the form of *PELS* caustic soda beads.



Above, circuit of diaphragm cells with dimensionally stable anodes. Below, Diaphragm Cell.



NaOH

# Methods of Manufacture



## BIPOLAR DIAPHRAGM ELECTROLYZERS

The diaphragm cells just described are classed as monopolar; each cell in a series circuit is connected by bus bars. Bipolar electrolyzers consist of a number of bipolar diaphragm cells, usually eleven, in a series circuit of up to 20 electrolyzers. The bipolar design permits current to flow internally within an electrolyzer from one cell to another, instead of through external bus bars, which are needed only between electrolyzers.

The bipolar electrolyzer design is the result of a joint development effort begun by PPG and de Nora in 1969 after many years of working separately on

bipolar cell design. These bipolar electrolyzers were commercialized in 1973.

The bipolar electrolyzers have dimensionally stable metal anodes made of titanium coated with mixed metal oxides. A constant small gap is maintained between anode and cathode, minimizing resistance loss through the electrode. Because the open design of the dimensionally stable anodes lets the chlorine escape from behind, the resistive path in front of the anodes is also reduced.

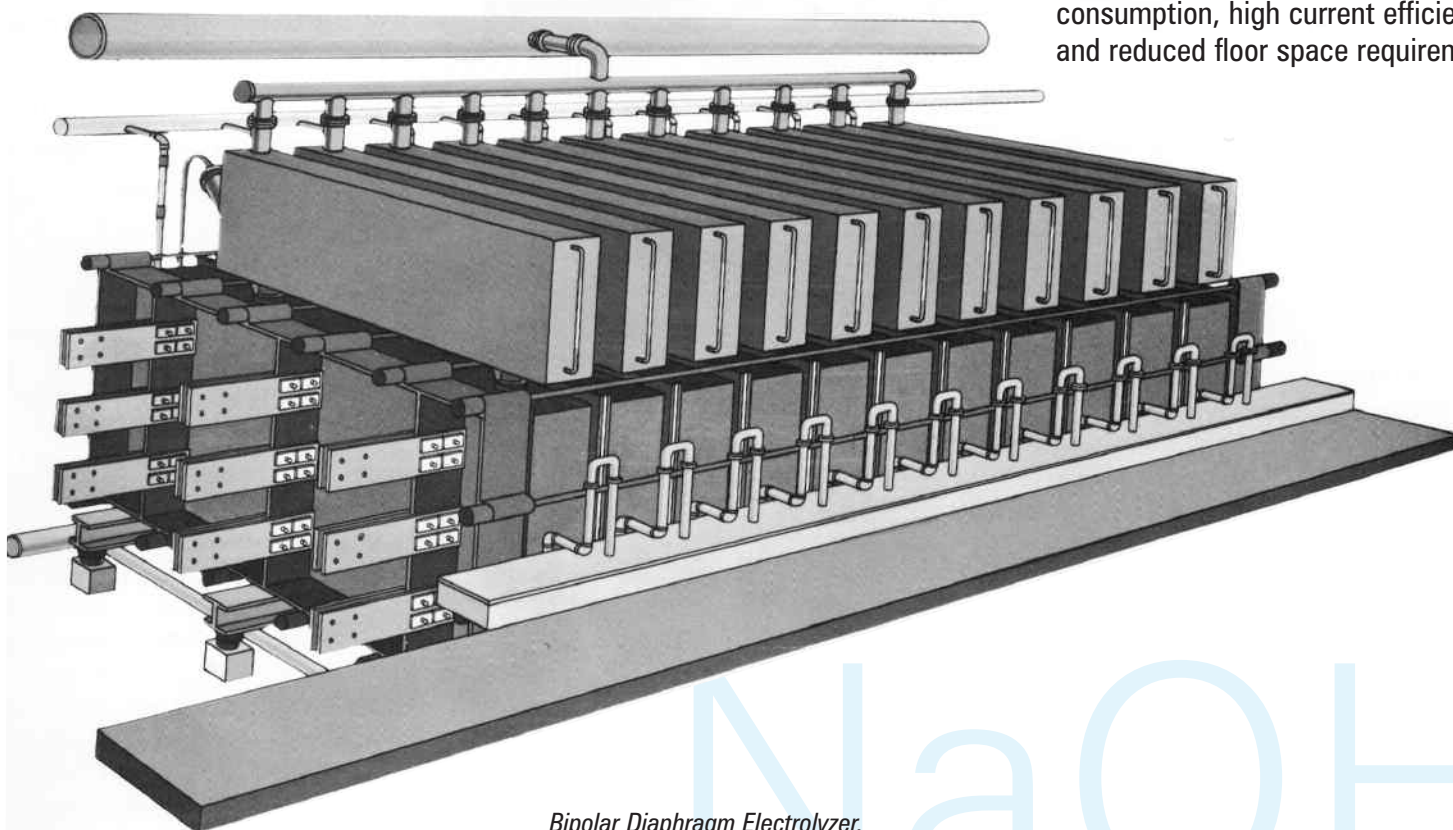
The bipolar design also lowers the resistance through the electrolyte and in the fastenings and connections between cells of an electrolyzer. As a result, the cell voltage of bipolar elec-

trolyzers is substantially less than that of monopolar diaphragm cells.

Each cell of a bipolar electrolyzer has its own brine reservoir made of fiberglass-reinforced polyester. The reservoir provides an emergency stand-by supply of brine in case brine flow to the cell should be temporarily stopped.

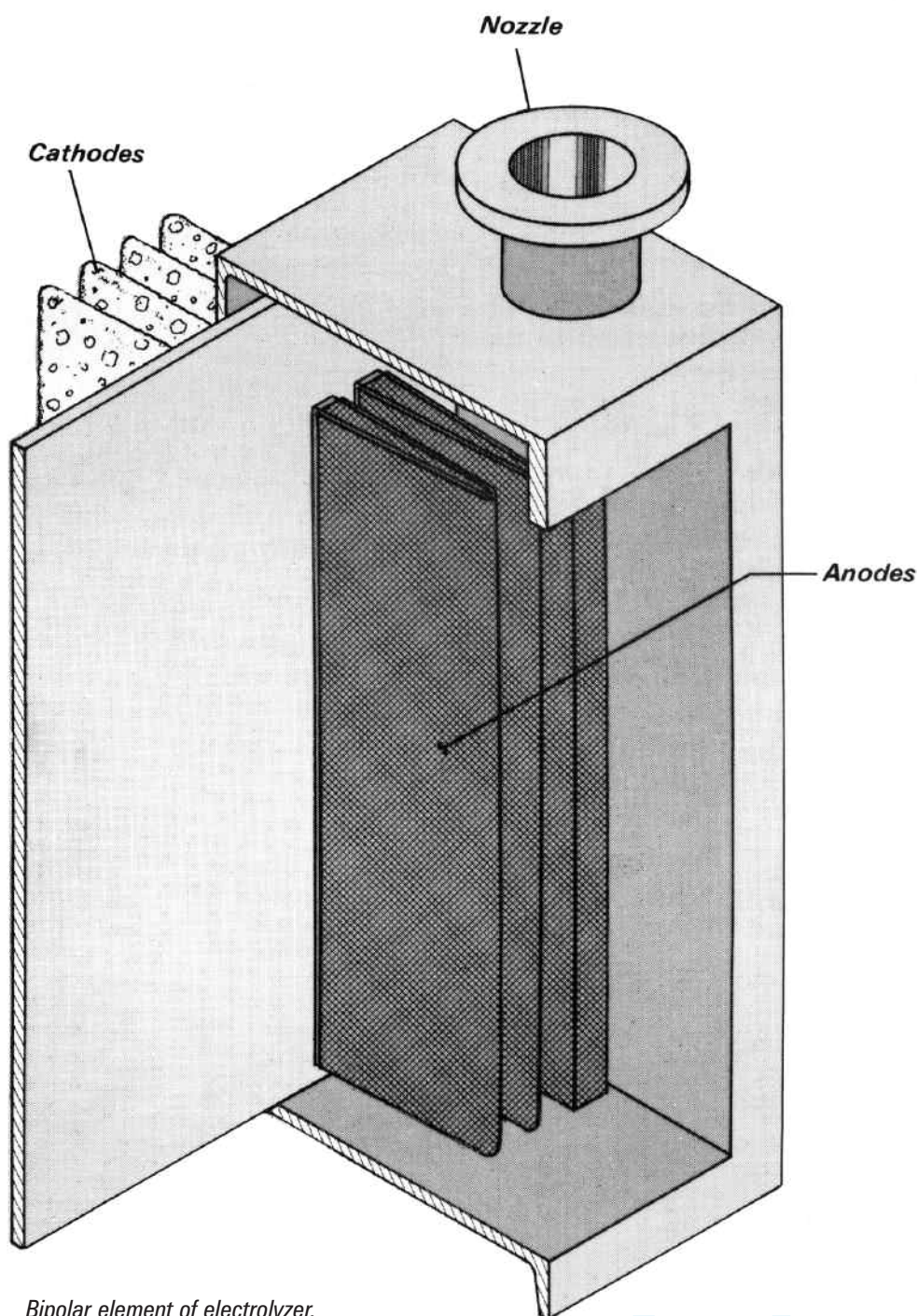
Bipolar electrolyzer cell rooms have portable cutout switches which permit single electrolyzers to be taken out of service without interrupting the operation of an entire circuit. The electrolyzer can be removed from the cell room to a cell renewal facility by a transporter system.

Major benefits of the bipolar electrolyzer include lower power consumption, high current efficiency, and reduced floor space requirement.



*Bipolar Diaphragm Electrolyzer.*





Bipolar element of electrolyzer.

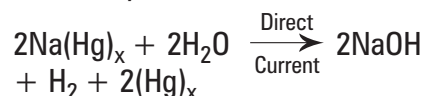
### MERCURY CELL PROCESS

The mercury cell is really two cells. In the primary unit, chlorine and sodium are released by electrolysis of brine; the chlorine is drawn off, and the sodium dissolves in the mercury cathode, forming an amalgam. In the secondary unit, the sodium in this amalgam reacts with water, forming sodium hydroxide and releasing hydrogen. The reaction for this method is shown.

The mercury cell process is in two steps. The overall reaction in the primary cell is:



The sodium produced at the cathode dissolves in the mercury to form an amalgam. The overall reaction in the secondary cell is:



There are several different types of mercury cells, some vertical, some horizontal. PPG Industries Chemicals Group uses two types of horizontal cells, the German-designed Uhde cells and the Italian de Nora cells. The primary cell is a broad, shallow steel trough, quite long, although shorter than an average battery of diaphragm cells. The top is a tightly fitting flat steel plate, or rubber cover.

The sides of the cell and the underside of the cover, which come into contact with chlorine, are protected by a rubber lining.



# Methods of Manufacture



The cathode is a slowly flowing stream of mercury which spreads across the floor of the cell. The mercury inlet end of the cell is slightly higher than the outlet end, so that the desired rate of flow is maintained by gravity. The metal anodes are dimensionally stable and mounted with the broad face parallel to the mercury bed. Vertical position of anodes is adjustable from outside the cell, permitting maintenance of optimum space between anodes and cathode (see diagram shown below).

The secondary cell, at one side of the primary, is of the same length but is much smaller in cross-section and is unlined. This cell is operated so that the mercury becomes the anode;

graphite grids form the cathode. Anode and cathode are in contact. The cell functions, in effect, like an internally shorted battery.

Raw brine for the mercury cell process is purified and concentrated in much the same way as for the diaphragm cell process. The effluent of brine from the mercury cell is depleted by partial removal of chlorine and sodium. This brine is recovered, resaturated and recirculated.

The amalgam from the primary cell flows to the secondary cell. Pure water flows over this amalgam; its decomposition is aided by the electric current between the amalgam and the graphite grids; hydrogen is liberated and caustic soda solution is formed.

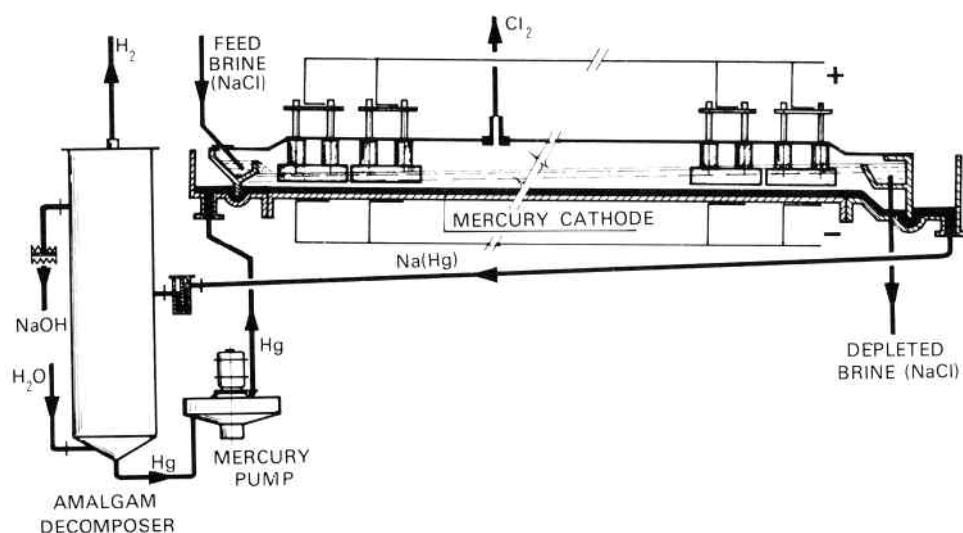
## MEMBRANE CELL PROCESS

The membrane cell in many respects is similar to a diaphragm cell except that a high strength, high purity caustic is produced at the cell. Both monopolar and bipolar cells are available. The use of an ion exchange membrane in lieu of an asbestos diaphragm and nickel instead of steel construction for the cathode compartment constitute the major design differences. A schematic of a membrane cell is shown on the following page.

Brine treatment for the membrane cell process is the same as for the diaphragm cell except that the purified brine is passed through an ion exchange resin. Also, since the brine does not pass through the membrane, the depleted brine from the cell is normally recirculated in a closed loop system. This requires dechlorinating and resaturating the brine. The brine in selected situations is used on a "once through basis" by resaturating the depleted brine and feeding it to a diaphragm cell circuit.

In the electrolysis of brine, chlorine is produced at the anode. The sodium ion that is generated is selectively allowed to pass through the membrane. In the cathode compartment, hydrogen is produced at the cathode with the sodium ion reacting with the hydroxyl ion to the caustic soda. The caustic solution is recirculated and deionized water added to maintain the sodium hydroxide concentration at normally between 32 to 35% depending on the membrane used. For certain

De Nora Mercury Cell

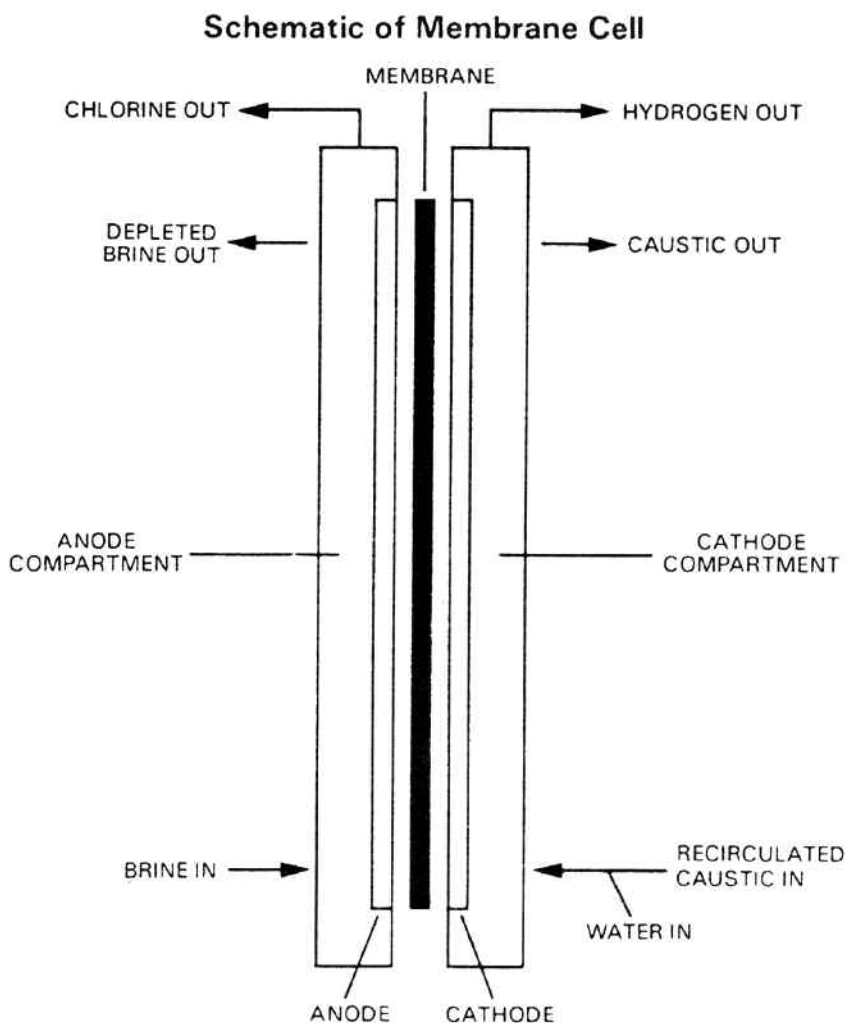




membranes, a lower caustic concentration must be maintained.

Because of the higher caustic strength, the cathode compartment is generally constructed with nickel. Since the nickel cathode is not as good an electrode material as steel, an electrolytic cathode coating is generally used to minimize the cell power consumption.

The caustic solution from the cells can be used "as is" for most captive requirements or evaporated to 50 percent employing a relatively simplistic evaporator system. This high purity caustic can also be concentrated to 73 percent or to anhydrous caustic soda.



NaOH



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