Occidental Chemical Corporation
A subsidiary of Occidental Petroleum Corporation

OXYCHEM

CAUSTIC POTASH HANDBOOK

KOH

Occidental Chemical Corporation (OxyChem) is a leading North American manufacturer of polyvinyl chloride (PVC) resins, chlorine and caustic soda — key building blocks for a variety of indispensable products such as plastics, pharmaceuticals and water treatment chemicals. Other OxyChem products include caustic potash, chlorinated organics, sodium silicates, chlorinated isocyanurates and calcium chloride. OxyChem's market position is among the top three producers in the United States for the principal products it manufactures and markets. Based in Dallas, Texas, the company has manufacturing facilities in the U.S., Canada and Latin America.

OxyChem has been an active participant in the American Chemistry Council's Responsible Care® initiative since its inception in 1988. Demonstrating their commitment to attaining the highest levels of safety and environmental achievement, Responsible Care companies implement world-class management systems, measure performance based on industry-wide metrics, and are subject to review by independent auditors.

Foreword

This handbook outlines recommended methods for handling, storing, and using caustic potash. It also includes information on the manufacture, physical properties, safety considerations and analytical methods for testing caustic potash. Additional information and contacts can be found on the internet at www.oxychem.com.

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INTRODUCTION

Caustic potash (potassium hydroxide or KOH) is most commonly manufactured by the electrolysis of a potassium chloride (KCl) solution. OxyChem manufactures caustic potash using membrane electrolytic cells. OxyChem does not use mercury based electrolytic cells to produce caustic potash. The co-products formed from the electrolytic production of caustic potash are chlorine and hydrogen.

Liquid caustic potash is available as a 45% or 50% solution. OxyChem can also provide dilutions of various concentrations. Caustic potash is also available in dry form as flakes or crystals. Specification sheets for each grade can be found on our website at www.oxychem.com.

All OxyChem's caustic potash meets the test requirements specified in the Food Chemicals Codex (FCC). Not all OxyChem's caustic potash is produced following all cGMP (current Good Manufacturing Practices) requirements as defined by the FDA (Food and Drug Administration). OxyChem does produce a food grade caustic potash, which is produced following cGMP requirements as defined by the FDA. OxyChem does not represent or warrant general compliance of this product for food use. Each prospective use of a product in a food or food related application must be carefully assessed against appropriate regulations by the user and it cannot be assumed that products meeting FCC test requirements are satisfactory for all uses without such assessment.

OxyChem has played a leading role in providing caustic potash to meet the increasing demands of industry.

Caustic potash is shipped via tank trucks, railcars, barges and ships. The dry form of caustic potash ships in drums, supersacks and plastic bags. Terminals are used to maintain stocks of our caustic potash in many principal cities. Distributor stocks are also available in these and many other cities and form a network of supply for the end user's convenience.

The largest users of caustic potash are agricultural chemical, fertilizer, and potassium chemical industries. Minor uses for caustic potash are soaps and detergents, battery manufacturing and potassium phosphate chemicals.

PRINCIPAL USES AND CONSUMPTION OF CAUSTIC POTASH

Caustic potash is one of the very few chemicals utilized in a very broad range of applications. Some principal products or processes in which caustic potash is used are:

Dehydrating agent for drying gases

Lubricant in the extrusion pressing of high melting alloys

Scavenger in gasoline treating process (dual layer) for removing mercaptans

Methylating agent

Alkaline builder in detergent formulations

Refining petroleum fractions

Removing insulating coatings from wire

Purifying olefin feedstock

Stabilizing synthetic lubricants

Removing naphthenic acids from natural gas

Fertilizers

Descaling ferrous metals

Sweetening sour petroleum fractions

Fused alkaline salt mixture used for metal cleaning

Lye peeling

Electrolytic stripping baths

Chemical compounding

Molten bath for removing polyesters and polyurethanes from steel objects

Chemical desiccant

Agent for lowering the sulfur content of coal

Alkaline batteries

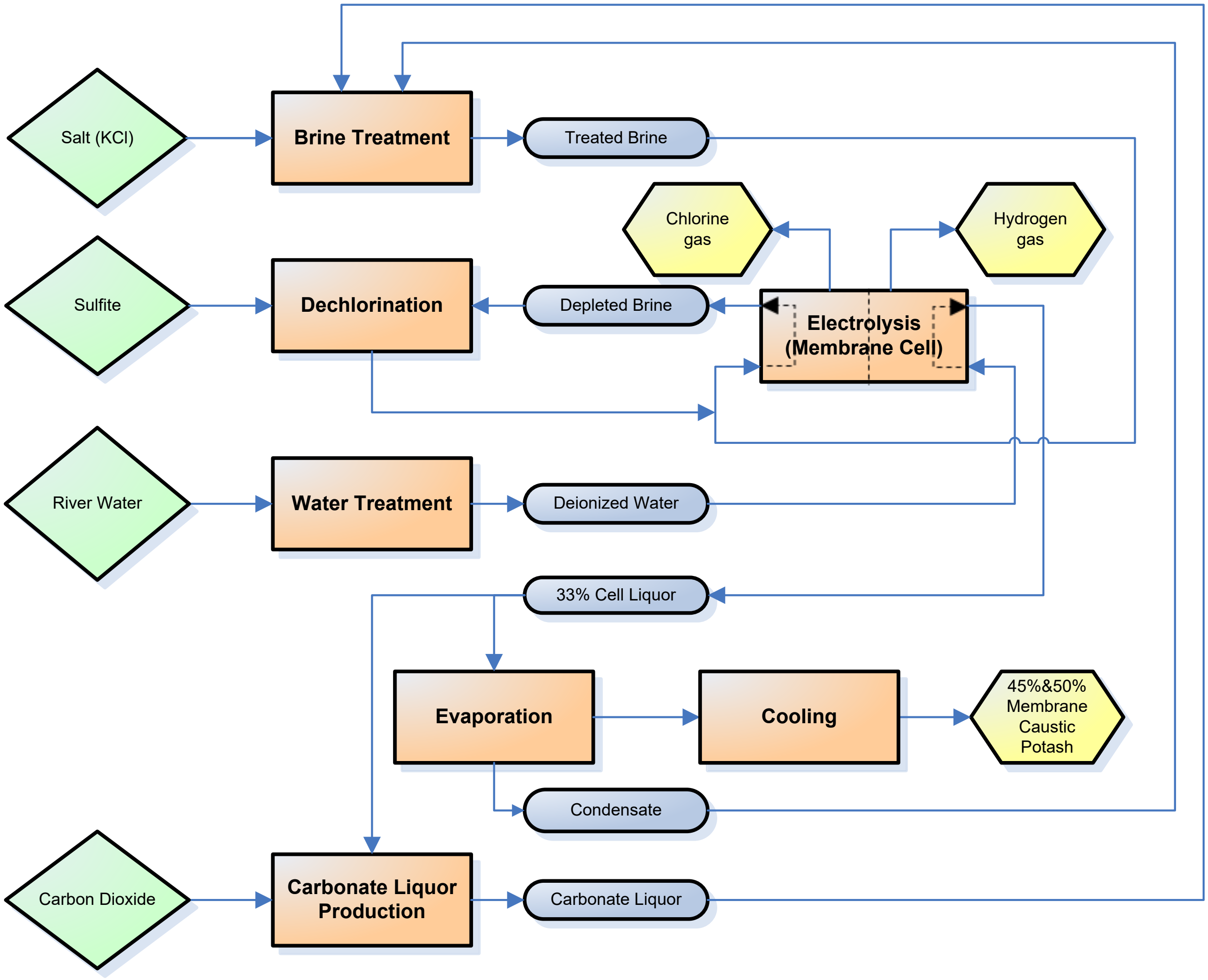
Catalyst for biodiesel production

MANUFACTURING PROCESS

Caustic potash is produced by OxyChem by an electrolytic process as shown in the following diagram. Brine, prepared from potassium chloride (KCl), is electrolyzed in a membrane cell. The production of caustic potash (KOH) also results in the co-products of chlorine and hydrogen.

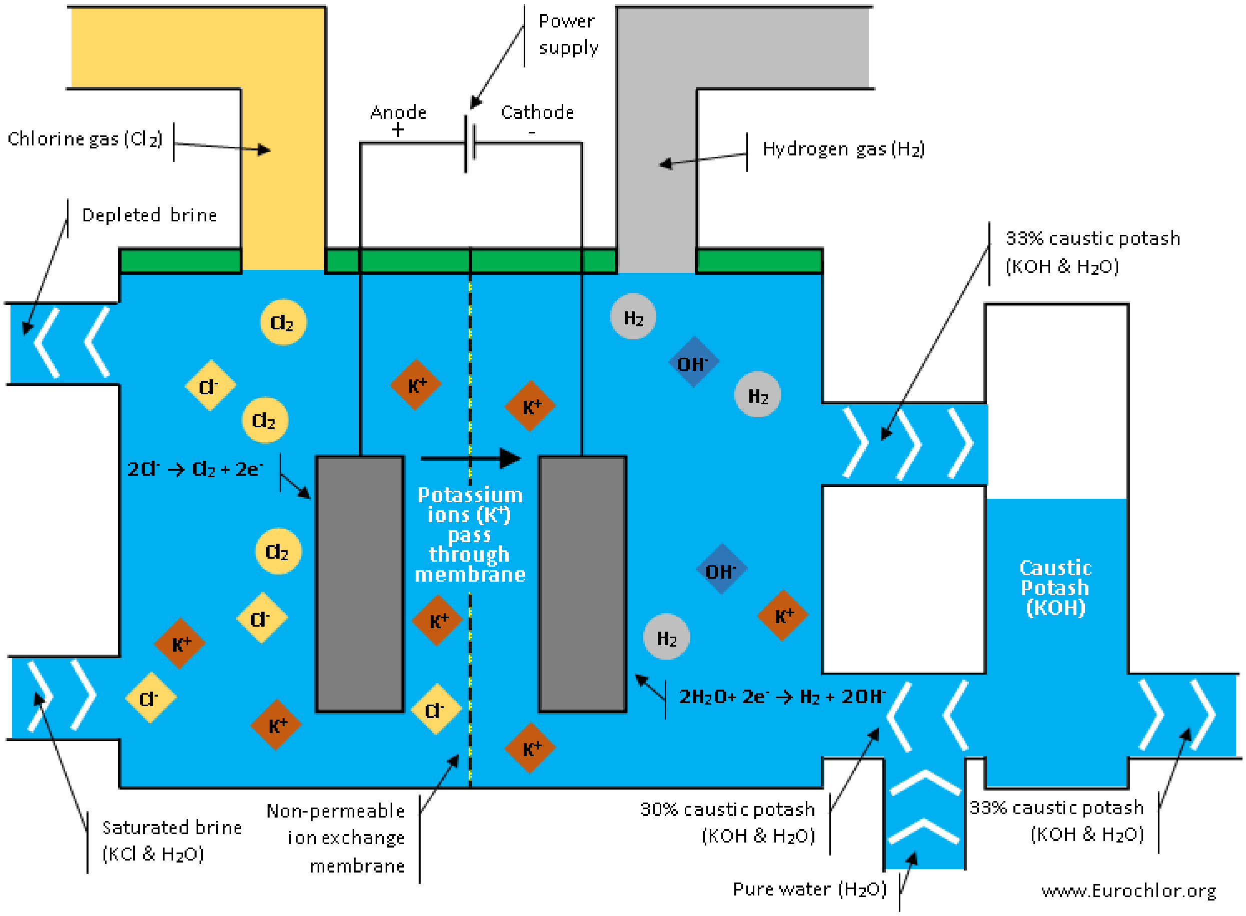
In the membrane process, a solution of approximately 33% strength is formed. The solution is then sent to evaporators, which concentrate it to a strength of 45% or 50% by removing the appropriate amount of water.

MEMBRANE CELL FLOW DIAGRAM



MANUFACTURING PROCESS

MEMBRANE CELL



SHIPPING METHODS

Liquid caustic potash is available from OxyChem's plant and terminals in tank truck, rail car, barge and ship quantities. Each form of transportation has its own advantages. The type of shipping method selected will depend upon such factors as size and location of storage, rate of consumption, plant location, freight rates, etc.

Anhydrous caustic potash is marketed in flake and crystal form. Drum packages include sizes of 100, 400 and 450 pounds. Both flake and crystal forms are also available in bulk 2000 pound supersacks. The flake form is available in 50 pound bags.

Caustic potash is regulated by the U.S. Department of Transportation (DOT) and is classified as a corrosive material. The DOT identification number is UN 1814 for liquid and UN 1813 for dry caustic potash.

SAFE HANDLING CAUSTIC POTASH

***Caustic potash* in any concentration must be respected by everyone who handles and uses it. Before starting to work with it, the user should be aware of its properties, know what safety precautions to follow, and know how to react in case of contact. Accidental exposure to caustic potash may occur under several conditions. Potentially hazardous situations include handling and packaging operations, equipment cleaning and repair, decontamination following spills and equipment failures. Employees who may be subject to such exposure must be provided with proper personal protective equipment and trained in its use. Some general guidelines follow:**

1. Read and understand the latest Safety Data Sheet.
2. Provide eyewash fountains and safety showers in all areas where caustic potash is used or handled. Any caustic potash burn may be serious. DO NOT use any kind of neutralizing solution, particularly in the eyes, without direction by a physician.
3. Move the patient to a hospital emergency room immediately after first aid measures are applied.

FIRST AID MEASURES

INHALATION: If inhalation of mists, vapors, or spray occurs and adverse effects result, remove to uncontaminated area. Evaluate ABC's (is Airway constricted, is Breathing occurring, and is blood Circulating) and treat symptomatically. GET MEDICAL ATTENTION IMMEDIATELY.

SKIN CONTACT: Immediately flush contaminated areas with water. Remove contaminated clothing, jewelry and shoes. Wash contaminated areas with large amounts of water. GET MEDICAL ATTENTION IMMEDIATELY. Thoroughly clean and dry contaminated clothing before reuse. Discard contaminated leather goods.

EYE CONTACT: Immediately flush contaminated eyes with a directed stream of water for as long as possible. Remove contact lenses, if present, then continue rinsing. GET MEDICAL ATTENTION IMMEDIATELY.

INGESTION: If swallowed, do not induce vomiting. For definite or probable ingestion, do not administer oral fluids. If vomiting occurs spontaneously, keep airway clear. Monitor airway. Volume resuscitation (IV fluids) and circulatory support (CPR) may be required. Never give anything by mouth to an unconscious or convulsive person. GET MEDICAL ATTENTION IMMEDIATELY.

SAFE HANDLING CAUSTIC POTASH

PERSONAL PROTECTIVE EQUIPMENT

OSHA requires employers to supply suitable protective equipment for employees. When handling caustic potash, the following protective equipment is recommended:

Wear suitable chemical splash goggles for eye protection during the handling of caustic potash in any concentration. The goggles should be close fitting and provide adequate ventilation to prevent fogging, without allowing entry of liquids. The use of a face shield may be appropriate when splashing can occur, including loading and unloading operations.

Wear rubber gloves or gloves coated with rubber, synthetic elastomers, PVC, or other plastics to protect the hands while handling caustic potash. Gloves should be long enough to come well above the wrist. Sleeves should be positioned over the glove.

Caustic potash causes leather to disintegrate quite rapidly. For this reason, wear rubber boots. Wear the bottoms of trouser legs outside the boots. DO NOT tuck trouser legs into boots.

Wear chemical resistant clothing for protection of the body. Impregnated vinyl or rubber suits are recommended.

Wear hard hats for protection of the head, face and neck.

If exposures are expected to exceed accepted regulatory limits or if respiratory discomfort is experienced use a NIOSH approved air purifying respirator with high efficiency dust and mist filters.

PROTECTIVE PRACTICES

Keep equipment clean by immediately washing off any spill or accumulation of caustic potash.

Weld pipelines where practical. Use flanged joints with gaskets made of caustic potash resistant material such as rubber, PTFE, or EPDM rubber. If a screwed fitting is used, apply Teflon® tape to the threads.

When disconnecting equipment for repairs, first verify there is no internal pressure on the equipment and the equipment has been drained and washed.

Provide storage tanks with suitable overflow pipes. Overflow pipes should be directed to a protected overflow area away from operations.

Shield the seal area of pumps to prevent spraying of caustic solutions in the event of a leak.

When releasing air pressure from a pressurized system, take every precaution to avoid spurts or sprays of caustic solution.

SAFE HANDLING CAUSTIC POTASH

In case of a spill or leak, stop the leak as soon as possible. After containment, collect the spilled material and transfer to a chemical waste area. Remove large liquid spills by vacuum truck. Neutralize residue with dilute acid. Flush spill area with water and follow with a liberal covering of sodium bicarbonate or other acceptable drying agent.

HANDLING LIQUID CAUSTIC POTASH

In handling caustic potash solutions, care must be taken to avoid solidification which will plug pipelines and equipment. Graph 1 on page 30 shows the freezing points for solutions of caustic potash at various concentrations.

Should a caustic potash solution become frozen in process equipment or piping, care must be taken when thawing the material. The use of atmospheric pressure steam is suggested. Accelerated corrosion can occur in areas where equipment is subjected to extremely high temperatures.

HANDLING ANHYDROUS CAUSTIC POTASH

Extreme care must be exercised when adding anhydrous caustic potash to water or to a solution. Its high heat of solution generates large amounts of heat which can cause local boiling or spurting. When making solutions always add the caustic potash slowly to the water surface with constant stirring. Never add the water to the caustic potash.

Anhydrous caustic potash will dissolve freely in a well agitated solution under proper conditions. Without agitation, the anhydrous caustic potash will fall to the bottom and form a layer of hydrate which dissolves quite slowly.

UNLOADING AND HANDLING CAUSTIC POTASH IN RAILCARS

GENERAL INFORMATION

Liquid caustic potash has a markedly corrosive action on all body tissue. Even dilute solutions may have a destructive effect on tissue after prolonged contact. Inhalation of mists can cause damage to the upper respiratory tract, while ingestion of liquid caustic potash can cause severe damage to the mucous membranes or other tissues where contact is made.

It is important that those who handle caustic potash are aware of its corrosive properties and know what precautions to take. In case of accidental exposure, immediately flush the exposed area with large amounts of water and seek medical attention. For more specific information refer to the Safe Handling section of this handbook and in the OxyChem SDS for liquid caustic potash.

PLACEMENT OF THE RAILCAR FOR UNLOADING

1. After the car is at the loading spot, DOT regulations require the hand brake be set and the wheels blocked (chocks).
2. DOT regulations require caution sign(s) be placed on or next to the rail track to warn persons approaching the car from the access track end(s). Caution signs must be left up until the railcar is completely unloaded and disconnected from the customer’s connections. Signs must be made of metal or other suitable material, at least 12 x15 inches in size, and bear the words, “STOP-TANK CAR CONNECTED”, or “STOP-MEN AT WORK.”
3. It is recommended that a *locked* switch or derail device be placed at the access track end(s), a minimum of one car length away from the railcar to be unloaded.
4. A railcar of caustic potash should only be unloaded when adequate lighting is available.
5. A suitable ramp should be used to access the railcar top operating or safety platform. The access ramp chosen should accommodate a variety of platform widths because of variations in rail car manufacturers, and railcar heights since the railcar will rise while unloading.

UNLOADING PRECAUTIONS

- Before making any connections, verify the content of the railcar is caustic potash.
- Test the nearest eyewash and safety shower per your facility procedure.
- Only responsible and properly trained personnel should unload liquid caustic potash. Unloading operations must be monitored while the car is connected.

UNLOADING AND HANDLING CAUSTIC POTASH IN RAILCARS

- Since serious burns can result from contact of caustic potash with the eyes and skin, the following personal protective equipment (PPE) listed below is recommended when handling equipment for caustic potash. Based on a job task analysis or job hazard assessment more PPE may be appropriate.

  Hard hat
  Mono-goggles
  Face shield
  Rubber, steel-toed boots
  Chemical gloves
  Chemical resistant suit
  Respiratory protection

- Make certain the storage tank is vented and has sufficient room available for the contents of the entire railcar.
- Consider restricting access to the area around the railcar and transfer line to the storage during the actual transfer operation.
- If a railcar needs to be moved when partially unloaded, DOT regulations require that all unloading lines must be disconnected and railcar closures must be replaced.
- A railcar may be sampled either from the top manway, or from the top eductor line using a suitable procedure. Sampling from the bottom unload piping is not recommended. If a railcar has partially frozen and has been thawed, special sampling techniques may be required due to stratified concentrations that may be present.
- OxyChem's liquid caustic potash is shipped in insulated and lined railcars. Typical linings have an upper temperature limit of 225°F. Therefore, recommended 'trapped steam' pressure used in the coils to heat up a railcar is limited to 15 psig max. Most customers use atmospheric steam in the coils.
- Unloading lines should be insulated and heated when used to transfer liquid caustic potash to storage in cold climates. The preferred method of heating is to provide electric or steam heat tracing around the unloading line, under the insulation. An alternate method is to provide tees in the unloading line so that steam (or hot water) can be run through the unloading line just prior to its use. Use caution using this method so as to NOT put water or steam directly into the railcar of caustic potash.

  NOTE: ***Running steam through unlined steel piping systems can result in increased corrosion and iron pickup in the product.***

- A properly designed and equipped padding system must be used if the railcar will be pad pressure unloaded. Compressed air is the most common padding gas. Nitrogen can also be used but it is more costly and it has the additional hazard of being an asphyxiant gas, thus extra precautions are required. All fittings used for padding a railcar should be inspected for defects before each use.
- The air supply system should be oil free, have a pressure relief valve set at 25 psig and a pressure regulator set at 18 to 20 psig. The removable connection piping used on the railcar vent valve should also have a pressure gauge, a release (vent) valve and a shut-off valve.

UNLOADING AND HANDLING CAUSTIC POTASH IN RAILCARS

General purpose caustic railcars are constructed to a 100 psig design with a burst rating of approximately 500 psig. Per 49 CFR 179.15, caustic railcars can have a pressure relief device installed that is set for 75 to 165 psig. The lower rated relief devices are sometimes hydraulically activated during rough transport handling and can release some liquid onto the jacket area.

Even though they are designed and equipped as stated, these are NOT pressure railcars and should not be padded above the **OxyChem and the Chlorine Institute recommended range of 20 to 25 psig max.**

At OxyChem after a railcar is loaded with product, it is padded up to approximately 30 psig and leak checked to meet the DOT shipping requirement. That test pad pressure is released prior to shipping.

Because of inherent shaking and jarring experienced by the railcar and contents during the shipping process, gaskets and joints can loosen up and customers should be cautious of that possibility. If pressure unloading, particular areas to watch are the manway gasket, the bottom flange gasket (when bottom unloading), and the top operator stuffing box gland area for the bottom outlet valve (BOV) if so equipped.

Additionally, the top operated bottom outlet valve reach rods can, in rare instances, come detached during transit. There have been reports that these detached rods can be *pushed up* from a railcar if unloading pressures greater than 30 psig are used .

HANDLING IN COLD WEATHER

Since OxyChem tank cars are well insulated and liquid caustic potash is loaded hot (~100°F), product usually arrives at its destination in a liquid state. However, since 50% liquid caustic potash begins to crystallize at 40°F, in cases of unusual delays in transit, partial freezing may take place in cold weather.

A temperature measurement of the caustic potash is the best indication if steaming is required. If the railcar product is 70°F or cooler, heating is recommended prior to unloading. Even in a liquid phase, cold material may flow poorly due to its higher viscosity.

If a railcar temperature is over 65°F but product will not flow from the BOV (bottom unloading), try applying a steam lance for a few minutes to the BOV and auxiliary valve. This uninsulated piping area is prone to freezing. If freezing has occurred, the following procedure should be used:

1. Vent the railcar – OxyChem recommends opening the railcar manway cover.
2. Connect a steam line to the bottom jacket connection pipe, and/or at the BOV connection. Connect a condensate return line at the steam condensate outlet pipe. If a steam trap is used on the exit of a railcar, OxyChem recommends regulating the steam pressure into the coils to a maximum of 15 psig. This is to protect the lining from excessive spot heating, which will damage the lining. If a condensate return line is not used, the condensate must be disposed of or otherwise utilized in a manner compliant with all environmental regulations.

UNLOADING AND HANDLING CAUSTIC POTASH IN RAILCARS

The amount of material that is frozen in a caustic potash rail car is dependent upon the outside temperature, wind chill, and the time elapsed between when the railcar was loaded and is unloaded. In very cold winter conditions, it is possible for a caustic potash railcar to be totally frozen. For this case, it may be necessary to steam the rail car for as long as three days (72+ hours) to fully liquefy all of the contents.

A temperature measurement is the best indication as to when the contents have liquefied and the caustic potash is ready for unloading. ***The recommended minimum unloading temperature of 50% caustic potash is 70°F. At 70°F through 90°F steaming may be desirable to reduce viscosity.***

If the above measures do not liquefy the contents on the railcar, contact your OxyChem technical representative.

ADDITIONAL NOTES FOR HEATING RAILCARS:

Do not exceed a steam pressure of 15 psig on the jacket. Certain railcar linings can be damaged using steam pressure above15 psig.

Do not keep steam on while emptying the railcar. Exposing the steam coils without fluid present to dissipate the heat can damage the lining due to local heating.

Do not heat the product over 120°F to minimize corrosion of unlined steel piping systems and equipment.

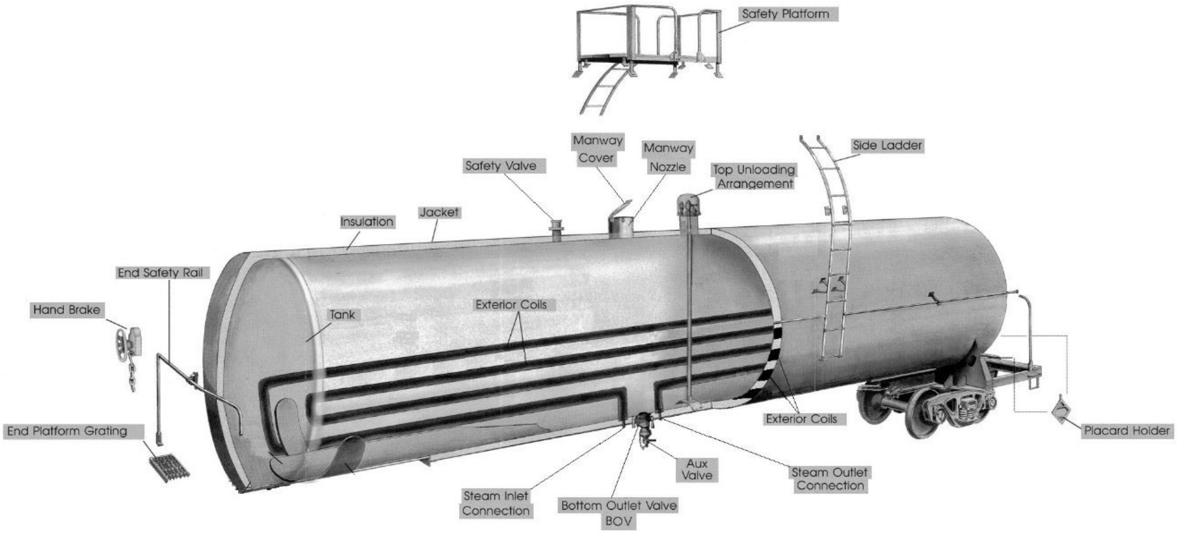


Figure 1 - Common Equipment on a Caustic Railcar

Figure 2 - Unloading Arrangements on Caustic Railcars

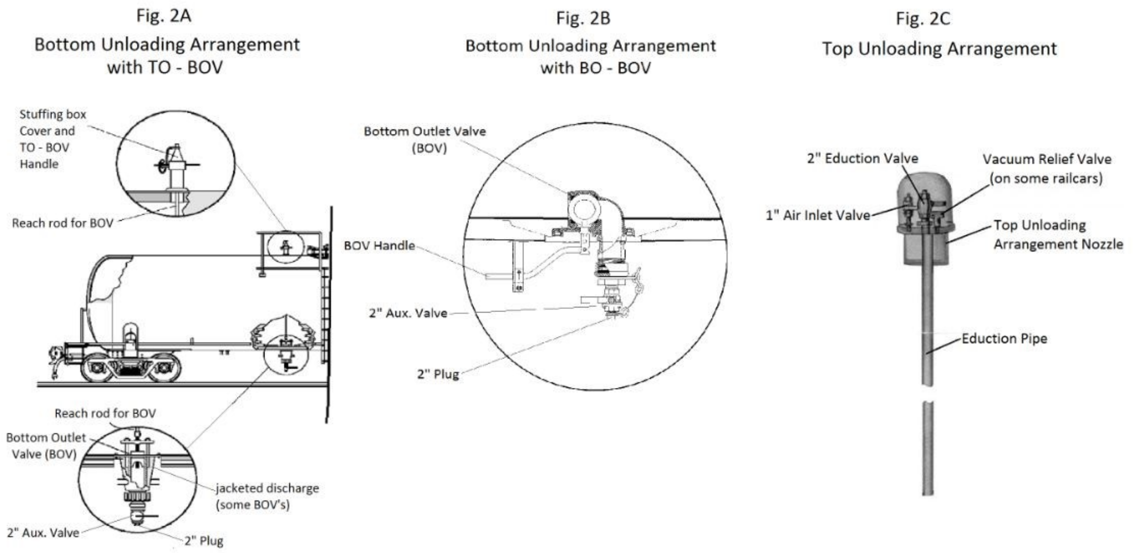


Fig. 2A
Bottom Unloading Arrangement with TO - BOV

Fig. 2B
Bottom Unloading Arrangement with BO - BOV

Fig. 2C
Top Unloading Arrangement

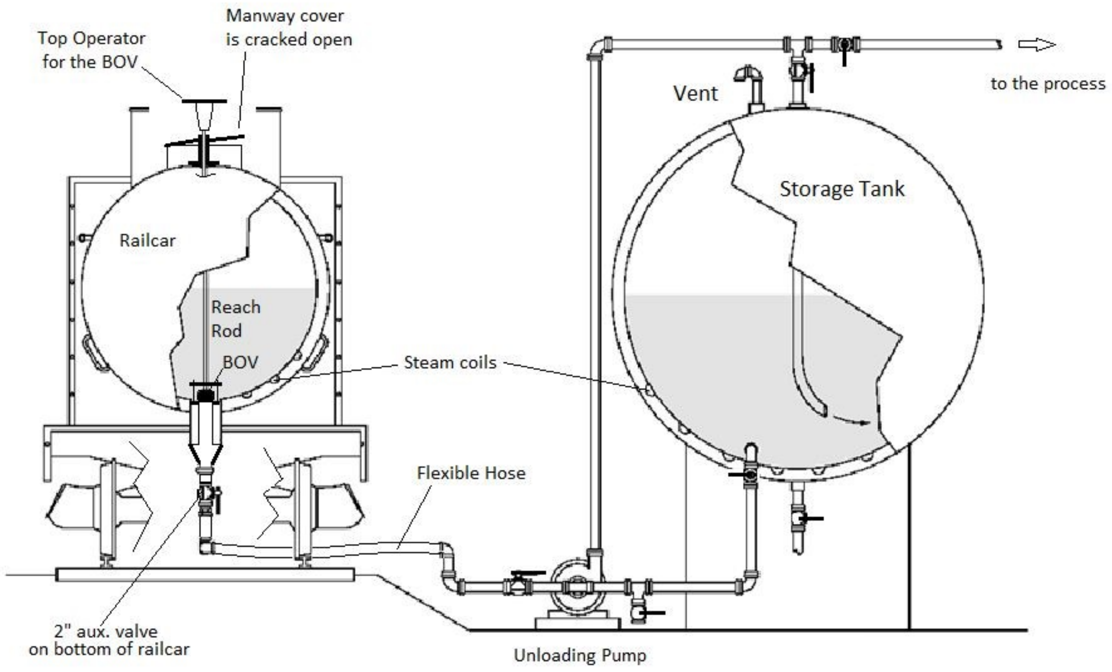


Figure 3 - Bottom Unloading to Storage

UNLOADING AND HANDLING CAUSTIC POTASH IN RAILCARS

UNLOADING RAILCARS

This section provides comments when unloading caustic potash from railcars in three ways: 1) via the bottom outlet valve (BOV) using gravity; 2) via the BOV using pad pressure; or 3) top unloaded through the well line using pad pressure. Refer to Figure 2 for the various unloading arrangements present on a caustic railcar.

BOTTOM UNLOADING - GRAVITY

1. Verify the contents are liquid and at the desired unloading temperature. If not, see “Handling in Cold Weather.”
2. Vent the railcar – OxyChem recommends opening the railcar manway cover.
3. **Refer to Figure 2A or 2B**. Ensure the internal bottom outlet valve (BOV) is closed tightly. If a top operated BOV, the valve rod which operates the bottom outlet valve has a handle on it which is located at the top of the railcar. The handle can be reversed and serves as a cap in transit.
4. Remove the plug from the auxiliary. valve, then attach an appropriate fitting and unloading line. A flexible connection hose is recommended since a railcar will rise as it is unloading.
5. Check the downstream unloading line to see that all valves are in the proper position for unloading to the storage tank.
6. Open the bottom auxiliary valve, then the internal BOV either by rotating the handle 90° if it is a bottom operated BOV, or by turning the top operator to allow contents to begin flowing by gravity to the pump or tank. If the BOV does not open upon application of light pressure, frozen caustic potash is probably present in the bottom of the car. Application of steam to the BOV area via a steam lance, or hookup to the heat coils may be necessary. See “Handling in Cold Weather.”
7. When the tank car is empty and the discharge pipe has completely drained, close the internal BOV and the auxiliary valve.
8. Disconnect the unloading fittings and hose and install the plug tool tight in the auxiliary valve.
9. Close the manway cover and secure all bolts tool tight. Close the vent valve if open and install the plug tool tight if removed.
10. Prepare the railcar for return.

    Note: See **Figure 3** for an example setup for Bottom Unloading using gravity feed to an unloading pump and then to a storage tank.

UNLOADING AND HANDLING CAUSTIC POTASH IN RAILCARS

BOTTOM UNLOADING – PAD PRESSURE

Compressed air can be used to increase the flow rate of caustic potash to storage or to transfer product without the use of a pump. If compressed air is to be used, the prior section instructions for bottom unloading should be modified as follows:

1. Close and secure the manway cover tool tight.
2. Remove the plug from the vent valve and connect the air supply piping and flexible hose to it.
3. Open the vent valve, and apply air pressure slowly to the railcar until there is a normal flow of liquid to the storage tank. The pressure should be maintained until the railcar is completely empty. OxyChem recommends use of 20-25 psig max. A drop in air pressure or the sound of air rushing through the unloading line indicates the railcar is empty.
4. When the railcar is empty, shut off the air supply to the railcar and allow the residual air pad to vent from the railcar either through the unloading line or through the vent valve on the air system piping - venting the pad from the railcar to a safe location.
5. Close the vent valve on the railcar.
6. Remove the air supply line and reinstall the plug tool tight.

TOP UNLOADING THROUGH THE EDUCTION (WELL) LINE – PAD PRESSURE

1. Verify the contents are liquid and at the desired unloading temperature. If not, see “Handling in Cold Weather.”
2. Refer to **Figure 2C.** Open the housing cover protecting the top unloading valves.
3. Remove the plug from the eduction valve, and using appropriate fittings, connect a flexible unloading hose. This is necessary since the railcar will rise while being unloaded.
4. Remove the plug from the vent valve, and connect the air supply piping and flexible hose to it.
5. Open the eduction valve and any other valves necessary to the storage tank.
6. Open the vent valve, and apply air pressure slowly to the railcar until there is a normal flow of liquid to the storage tank. The pressure should be maintained until the railcar is completely empty. OxyChem recommends use of 20-25 psig max. A drop in air pressure or the sound of air rushing through the unloading line indicates that the tank car is empty.
7. When the railcar is empty, shut off the air supply to the railcar and allow the residual air pad to vent from the railcar either through the unloading line, or through the vent valve on the air system piping - venting the pad from the railcar to a safe location.
8. When the railcar is at atmospheric pressure, close the eduction valve and disconnect the unloading line from the railcar.
9. Close the vent valve and disconnect the air supply from the railcar.
10. If desired, open the manway cover to verify the railcar is empty. Do not enter the car to make an inspection.
11. Replace both plugs in their respective valves tool tight, and secure the protective housing cover.

See **Figure 4** for an example setup for Top Unloading to a storage tank. Care should be taken not to spill caustic potash on the railcar, since it will cause damage to the paint and may endanger workers handling the empty railcar on its return. Wash off any spilled or dripped caustic.

PREPARING AN EMPTY RAILCAR FOR RETURN

1. Ensure both top valves are closed and plugs are installed tool tight. Secure the cover over the valves.
2. Close the manway cover taking care to ensure the gasket does not fall into the railcar, shift or fold. Ensure all manway cover bolts are tool tight.
3. Disconnect any steam lines used to heat the railcar. Do not place any caps or closures on the railcar steam pipes.
4. Make sure the bottom outlet valve (BOV) and auxiliary valve are closed, and the plug is installed tool tight. Return the empty railcar promptly in accordance with the shipper's instructions. The shipper's routing directions must be followed in all instances.

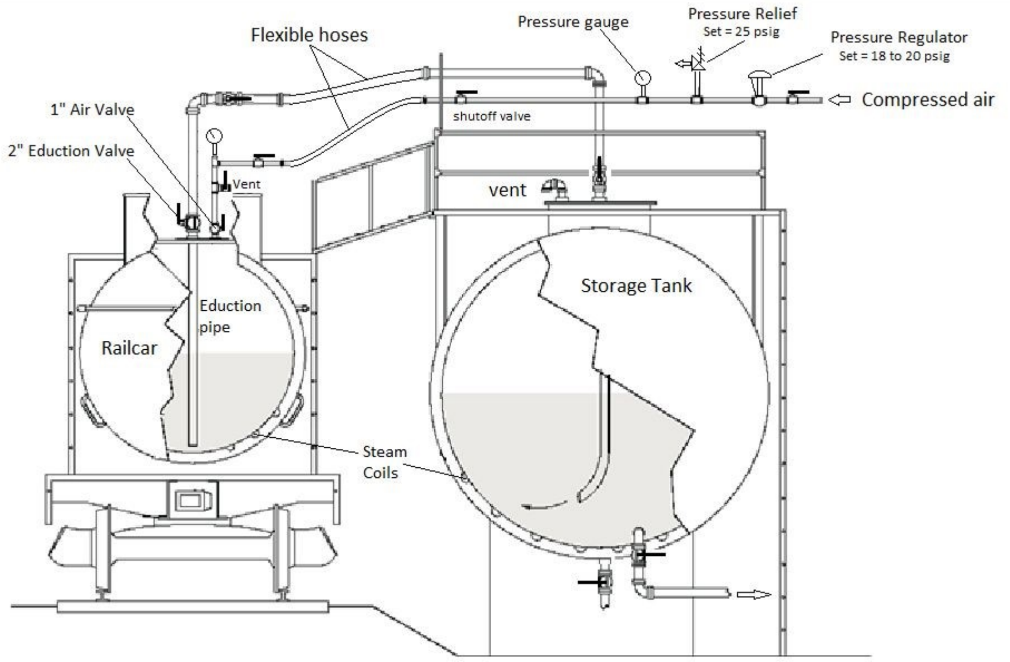


Figure 4 - Top Unloading to Storage

UNLOADING CAUSTIC POTASH TANK TRUCKS

CARRIER RESPONSIBILITIES

OxyChem tank truck drivers have received instructions regarding equipment and delivery procedures. If an OxyChem arranged carrier, delivering caustic potash to your plant, fails to adhere to the following guidelines, please contact OxyChem so that corrective action can be taken.

Equipment

Equipment must meet Department of Transportation regulations, Code of Federal Regulations (CFR), Title 49.

Tank Truck Specification

Tank trucks should meet the established DOT requirements for hauling liquid caustic potash.

Four(4) DOT “CORROSIVE” 1814 placards must be affixed to the cargo tank. One on each side.

Unloading Equipment

If unloading is by gravity to storage or customer’s unloading pump, no special equipment is needed.

If unloading is by truck-mounted pump, use only an all iron or stainless steel unit. The pump can be driven by a tractor powered take-off or an auxiliary gasoline engine. Use at least a 2-inch pump line.

If unloading is by customer compressed air, the line used to supply air to the tank truck is required to be equipped with a pressure reducing valve, a pressure relief valve, a pressure gauge and a block valve. The relief valve should be set at a maximum pressure of 20 PSIG and the pressure reducing valve should be set at 2 to 3 pounds lower.

A 40 foot length of air hose is required if the customer’s air supply is used. When compressed air is not available from the customer’s plant, trucks equipped with pumps or air compressors can be provided at the customer’s request.

Unloading Lines

Unloading hoses must be constructed of material resistant to caustic potash. Hoses should be at least 2 inches in diameter and 15 to 30 feet in length.

Whether the unloading hose is fitted with a union, pipe flange, or a quick type coupler, the truck driver should have available matching fittings and tools to facilitate a connection to a 2-inch or 3-inch threaded pipe.

UNLOADING CAUSTIC POTASH TANK TRUCKS

TRUCK DRIVER RESPONSIBILITIES

Truck drivers must obtain permission to unload from the proper site personnel and observe any special instructions from the customer.

Truck drivers must wear the protective equipment required by OxyChem as listed under Protective Equipment, (pg. 11) or by the customer, whichever is more inclusive, and at all times follow safe handling practices. Customers must not allow truck drivers who do not meet these requirements to unload.

The following unloading procedures are recommended:

Check the operation of the safety shower and eyewash fountain. Purge water through each to remove rust that may have accumulated and ensure that clean water flows out.

Connect the unloading hose to the discharge outlet on the tank truck.

Connect the other end of the unloading hose to the customer's storage tank fill line.

During cold weather one may use steam to preheat the fill line, the unloading hose, and, if needed, the truck outlet.

Customer should verify all valves to the storage are properly set and then open their unloading valve.

Start the pump or start pressurizing the tank, depending on the type of equipment used.

Open the valves on the truck discharge line.

Stand by until the truck cargo is completely unloaded.

If compressed air is used, allow the air to flush out the lines to the storage tank and then close and disconnect the air supply.

Close the truck discharge valves and customer unloading valve.

Drain the hose to an appropriate container and disconnect from truck and customer unloading connection points.

Reapply any flange cover or cap on the customer line.

Cap the truck discharge line and secure both hoses in the carrier tubes or tray.

EQUIPMENT FOR HANDLING CAUSTIC POTASH

GENERAL CONSIDERATIONS

Caustic potash is a corrosive chemical which is normally handled in either steel, nickel, nickel alloys or certain types of plastic equipment. The specific material will depend on the conditions under which the material is being used. Temperature, solution concentration, location and safety considerations are all important factors in equipment selection.

MATERIALS OF CONSTRUCTION

Carbon steel is the most commonly used material of construction for caustic potash at low to moderate temperatures. The ideal storage temperature for caustic potash in carbon steel is 80 to 100°F. Temperatures above 120°F will cause accelerated corrosion of the carbon steel and subsequent iron contamination of the caustic potash (above 120°F, cracking can occur if concentrated caustic is processed in steel equipment that has not been stress relieved). Where iron contamination or corrosion is unacceptable, epoxy lined carbon steel, 316L and 304L stainless steels are recommended. 316L and 304L stainless is acceptable to 200°F. At temperatures above 200°F, nickel is typically used but Monel®, Inconel®, or Hastelloy® can also be used. Consult with the supplier about the working temperature range of a particular lining.

Plastics, such as polyethylene, polypropylene, PVC, and CPVC, can be used. They do not contribute to iron contamination. They are chemically compatible with caustic potash so long as their maximum temperature limitation is not exceeded. When using PVC or CPVC as the material of construction, use a glue that does not contain silica as a filler. PVC and CPVC glues contain a filler (typically silica). Caustic potash will attach the silica filler causing leaks to develop at the glue joints. The manufacturer of the tank, drum, piping or equipment in question should be contacted to determine the exact limitations of the specific plastic.

DANGER: Aluminum, copper, zinc, lead and their alloys (e.g., brass and bronze) are NOT suitable for handling or storing caustic potash. Caustic potash readily attacks these materials.

STORAGE TANKS

Tanks can be either vertical or horizontal. They are usually fabricated from at least 1/4-inch steel plate. A 1/8-inch corrosion allowance should be included in the design. If iron contamination is a problem, tanks can be fabricated from 304L or 316L stainless steel. If the tanks are large, it's usually more economical to fabricate a steel tank and line it with an epoxy coating. Plastic tanks are usually fabricated from polypropylene or fiberglass reinforced plastics (FRP). Since caustic potash can attacks glass reinforcement fibers of improperly constructed FRP tanks, care must be taken to ensure the FRP tanks are built with the proper reinforcing materials, resins, catalysts, curing procedures and corrosion barriers.

Storage tanks should have a level indicating device for measuring liquid level.

Where heating is required, an external heat exchanger with a circulating pump or internal steam heating coils are most commonly employed. The preferred materials for the coils are nickel, Monel®, or Inconel®. Despite this, stainless steel is commonly used because of cost considerations. At high temperatures, stainless steel may crack. If it is necessary to insulate the storage tank, a two-inch layer of polyurethane foam or cellular glass should be adequate.

Proper design of a storage system will include adequate containment in case of tank failure. State and local regulatory authorities should always be consulted during the design phase of construction.

TANK CLEANING AND PASSIVATION

Tank cleaning is dependent on the previously stored product. A tank that previously contained caustic potash requires wall thickness testing, scale removal, floor cleaning, rinsing, passivation, and immediate filling. A tank previously containing another product requires cleaning with an appropriate solvent or cleaning agent, as well as the other steps mentioned above.

Scale removal is accomplished by blasting the walls with an abrasive such as sand or pecan shells. Abrasives containing high percentages of metals are not recommended.

The wall thickness of the tank should be measured to ensure the tank has structural integrity for the density of the product and the height of product in the tank.

Passivation requires permeation of the steel tank walls with caustic potash. This is usually accomplished by spraying the cleaned walls with a hot solution of caustic potash. Temperatures of 100-140°F and solutions of 5-20% are recommended. A standard recommendation would be spraying the walls for 2-4 hours with 10% solution at 140°F. The larger the tank the longer it should be sprayed to complete the passivation. Utilizing a hotter and stronger solutions will require less time for passivation. One way to achieve the solution heat necessary is to dilute 50% caustic potash to 20%. The heat of dilution will cause the caustic potash temperature to rise. Additional heat may be necessary to achieve optimal solution temperatures. The coating of the tank walls is best accomplished with an elliptical sprayer.

After passivation, the tank bottom must be cleaned out as well as possible. The quality of the initial product stored in the tank will depend greatly upon the extent to which the tank bottom is cleaned. After spraying, a squeegee will need to be used to clean the tank bottom.

After cleaning, the tank should be filled with caustic potash as soon as possible. This will prevent the tank walls from losing their passivation. If the tank cleaning is not completely successful, it may be necessary to filter the initial product from the tank to remove any remaining particulate matter. This would require a 5-10 micron filter media housed in a unit that would be compatible with the temperature, pressure, and chemical.

EQUIPMENT FOR HANDLING CAUSTIC POTASH

PIPING AND VALVES

Pipelines are usually at least two inches in diameter and constructed of Schedule 40 black iron or mild steel with welded or flanged joints. Where disconnects are necessary, flanged joints are preferred to facilitate maintenance. A safety flange guard of wrap-around polypropylene is recommended for all flanged joints. This will protect against spraying in case a gasket leaks.

Proper pipeline design includes an adequate pitch to permit complete draining. Avoid any loops or pockets. Lines should also include water or air connections for purging after use.

Where slight iron contamination is unacceptable, CPVC, polypropylene, polypropylene-lined steel, and Teflon® lined steel pipe are suitable materials. Pay special attention to suitable operating temperatures and pressures with these materials.

Ductile iron, cast steel, stainless steel, Alloy 20, and Teflon®-lined quarter-turn plug or ball valves are recommended for caustic potash service.

PUMPS

Centrifugal pumps of stainless steel or Alloy 20 construction, with either double mechanical seals or a packing gland, is recommended. Packing material should be Teflon® impregnated, caustic resistant fibers, or equivalent. To avoid seals altogether, magnetically coupled pumps could be used.

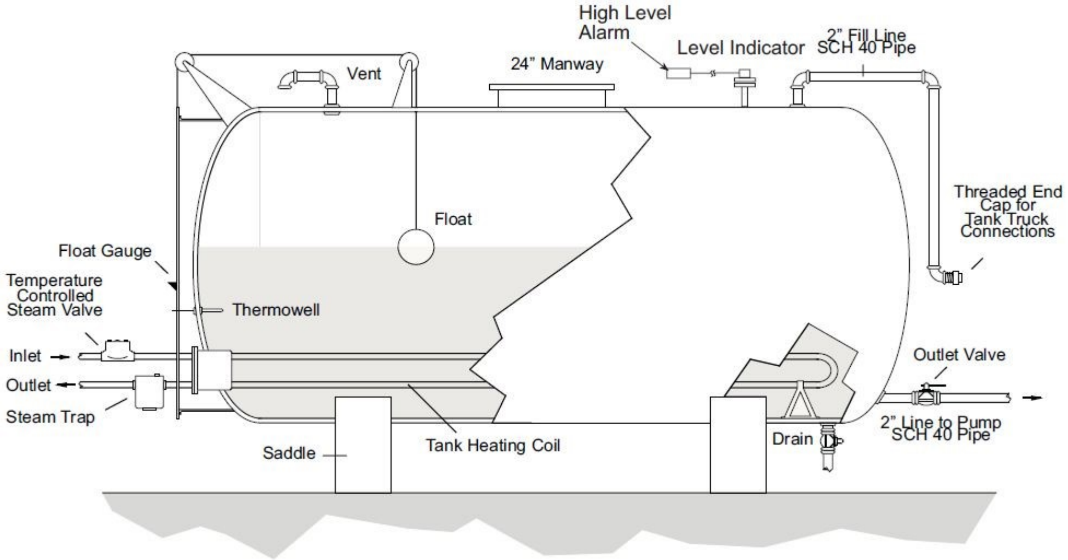
The pump location should receive careful consideration. For ease of operation, keep the suction lines as short as possible. A recirculating line will help prevent excess wear on the pump and, in many cases, can assist in controlling flow rates.

The pump seal area should have a liquid impervious shield installed.

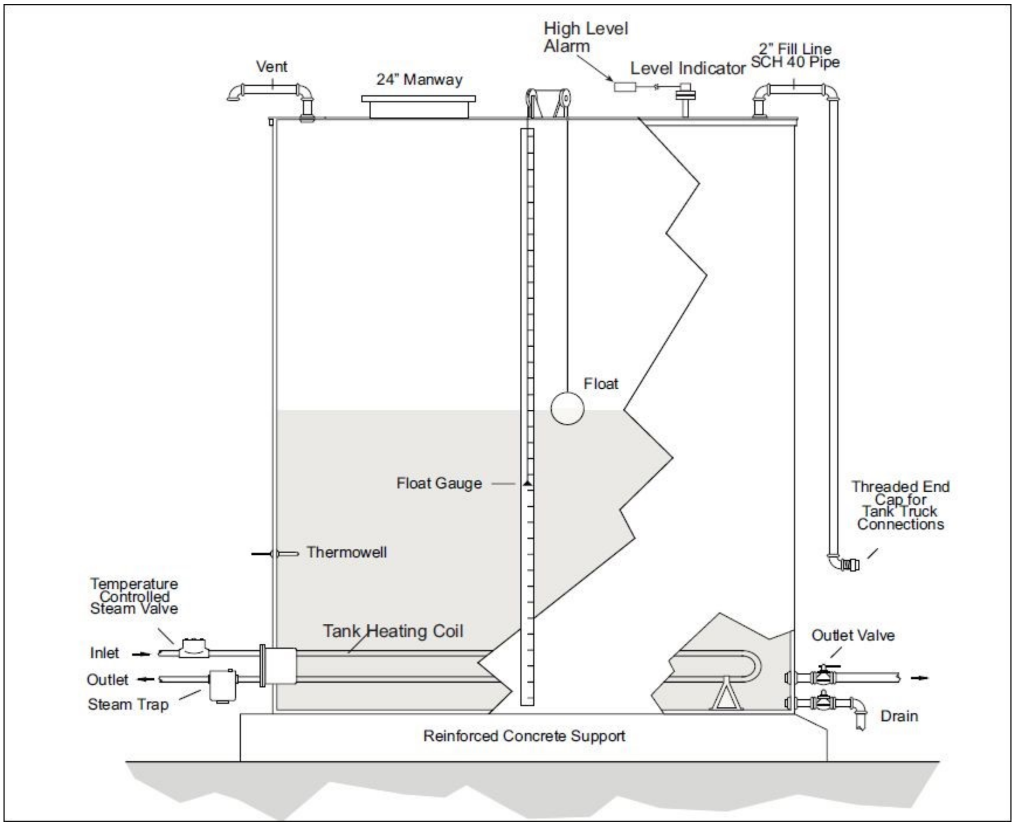
METERS

Caustic potash solutions can be metered through standard rotameters having non-glass tubes and nickel or stainless steel floats. Magnetic, Coriolis or orifice-type meters are preferred for strong, hot solutions. They should be made of corrosion resistant materials such as stainless steel, alloy 20, Monel®, or nickel.

TYPICAL STORAGE TANK INSTALLATION



*NOTE: All tanks should be located within a diked area.



TECHNICAL DATA

Table 1—Density and Solids Content of Caustic Potash Solution at 15.6°C (60°F)

%KOH	%K_2O	Specific Gravity	Degrees Baumé	Degrees Twaddell	KOH g/L	KOH lb/gal	KOH lb/ft^3	Density lb/gal	Density lb/ft^3
0	0.00	1.000	0.00	0.0	0.00	0.00	0.00	8.34	62.43
1	0.84	1.009	1.29	1.8	10.09	0.08	0.63	8.41	62.99
2	1.68	1.018	2.56	3.6	20.36	0.17	1.27	8.49	63.55
3	2.52	1.027	3.81	5.4	30.81	0.27	1.92	8.56	64.12
4	3.36	1.036	5.04	7.2	41.44	0.35	2.58	8.64	64.68
5	4.20	1.045	6.38	9.0	52.25	0.44	3.26	8.72	65.24
6	5.03	1.055	7.56	11.0	63.30	0.53	3.95	8.80	65.86
7	5.88	1.064	8.72	12.8	74.48	0.62	4.65	8.88	66.43
8	6.72	1.073	9.86	14.6	85.84	0.72	5.35	8.95	66.99
9	7.56	1.083	11.11	16.6	96.57	0.81	6.08	9.04	67.61
10	8.40	1.092	12.22	18.4	109.20	0.91	6.81	9.11	68.17
11	9.23	1.102	13.42	20.4	121.22	1.01	7.56	9.20	68.80
12	10.07	1.111	14.49	22.2	133.32	1.11	8.32	9.27	69.36
13	10.91	1.121	15.65	24.2	145.73	1.22	9.10	9.35	69.98
14	11.75	1.131	16.79	26.2	158.34	1.32	9.88	9.44	70.61
15	12.59	1.140	17.81	28.0	171.00	1.43	10.68	9.51	71.17
16	13.43	1.150	18.91	30.0	184.00	1.54	11.49	9.60	71.79
17	14.27	1.160	20.00	32.0	197.20	1.64	12.31	9.68	72.42
18	15.11	1.170	21.07	34.0	210.60	1.75	13.13	9.76	73.04
19	15.95	1.180	22.12	36.0	224.20	1.87	14.00	9.85	73.67
20	16.79	1.190	23.15	38.0	238.00	1.99	14.84	9.93	74.29
21	17.63	1.200	24.17	40.0	252.00	2.10	15.72	10.01	74.92
22	18.47	1.210	25.16	42.0	266.20	2.22	16.60	10.09	75.54
23	19.31	1.220	26.15	44.0	280.60	2.34	17.50	10.18	76.16
24	20.15	1.230	27.11	46.0	295.20	2.46	18.41	10.26	76.79
25	20.99	1.240	28.06	48.0	310.00	2.59	19.35	10.35	77.41
26	21.83	1.250	29.00	50.0	325.00	2.71	20.27	10.43	78.04
27	22.67	1.261	30.01	52.2	340.47	2.84	21.24	10.52	78.72
28	23.51	1.271	30.92	54.2	355.88	2.97	22.20	10.60	79.35
29	24.35	1.282	31.90	56.4	371.78	3.10	23.19	10.69	80.04
30	25.19	1.292	32.77	58.4	387.60	3.23	24.17	10.78	80.66
31	26.02	1.303	33.72	60.6	403.93	3.37	25.19	10.87	81.35
32	26.86	1.314	34.65	62.8	420.48	3.50	26.22	10.96	82.03
33	27.70	1.324	35.48	64.8	436.92	3.64	27.25	11.05	82.66
34	28.54	1.334	36.30	66.8	453.56	3.78	28.29	11.13	83.28
35	29.38	1.345	37.19	69.0	470.75	3.92	29.36	11.22	83.97
36	30.22	1.356	38.07	71.2	488.16	4.07	30.45	11.31	84.66
37	31.06	1.367	38.93	73.4	505.79	4.22	31.55	11.41	85.34
38	31.90	1.378	39.78	75.6	523.64	4.37	32.66	11.50	86.03
39	32.74	1.389	40.61	77.8	541.71	4.52	33.79	11.59	86.72
40	33.58	1.401	41.50	80.2	560.40	4.67	34.96	11.68	87.46
41	34.42	1.412	42.31	82.4	578.92	4.82	36.11	11.77	88.15
42	35.26	1.423	43.10	84.6	597.66	4.98	37.28	11.87	88.84
43	36.10	1.435	43.95	87.0	617.05	5.14	38.49	11.97	89.59
44	36.94	1.446	44.72	89.2	636.24	5.31	39.68	12.06	90.27
45	**37.78**	**1.457**	**45.48**	**91.4**	**655.65**	**5.47**	**40.89**	**12.16**	**90.96**
46	38.62	1.468	46.23	93.6	675.28	5.63	42.12	12.25	91.65
47	39.46	1.480	47.03	96.0	695.60	5.80	43.39	12.35	92.40
48	40.30	1.492	47.92	98.4	716.16	5.97	44.66	12.45	93.15
49	41.14	1.504	48.59	100.8	736.96	6.14	45.96	12.55	93.89
50	**41.98**	**1.516**	**49.35**	**103.2**	**758.00**	**6.32**	**47.28**	**12.65**	**94.64**

Table 2—Specific Conductance (Electrical) of Caustic Potash Solutions

%KOH	γ_{18}	$\gamma_{19.1\text{-}23.4}$	γ_{25}	γ_{30}	$\gamma_{50.5\text{-}52.4}$	$\gamma_{80.1\text{-}80.2}$
42.123	0.4212					
37.871	0.4790					
33.642	0.5221					
29.417	0.5434					
25.216	0.5403					
20.998	0.5106					
20.926	0.5199					
18.216		0.5301[4]			0.7958[5]	1.0861[9]
16.797	0.4558					
12.605		0.4044[3]			0.6202[7]	0.8291[10]
12.596	0.3763					
10.311	0.3286					
8.779		0.2968[2]			0.4563[6]	0.6089[10]
8.429	0.2723					
5.374	0.1876					
5.339		0.1870[1]			0.3021[8]	0.3962[9]
5.151				0.2160		
4.199	0.1464					
2.736	0.1001					
2.629				0.1165		
1.1413	0.04334					
0.5927	0.02309					
0.28069			0.01284			
0.07996			0.003689			
0.052608			0.002434			
0.041281			0.0019155			
0.035893			0.0016723			
0.030193			0.0014125			
0.019356			0.0009144			
0.011927			0.0005675			
0.006534			0.0003124			
0.0044968			0.00021495			
0.0032901			0.00015654			

Note: The exact temperatures used for the values with superscripts are as follows:

1	19.1°C	6	50.6°C
2	20.7°C	7	51.3°C
3	21.6°C	8	52.4°C
4	23.4°C	9	80.1°C
5	50.5°C	10	80.2°C

The specific conductivity "γ" is expressed in ohm^{-1}·cm^{-1}.

Table 3—Dielectric Constants for Caustic Potash Solutions

%KOH	0	0.0004771	0.000951	0.004771	0.00951
$\varepsilon_{20.0°C}$	80.50	80.28	80.08	78.71	
$\varepsilon_{21.0°C}$	80.15			78.30	76.37

Table 4—Diffusion Coefficient between water and Caustic Potash Solutions

%KOH	0.06	0.11	0.28	0.56	1.11	2.74	5.42	10.25
D (cm^2/day)	1.903	1.889	1.872	1.854	1.843	1.841	1.855	1.892

Table 5—Dissociation Voltage for Caustic Potash Solutions

%KOH	0.0695	0.695	5.4	19.0	40.0
E (volt)	1.46	1.45	1.40	1.38	1.32

Note: A platinum cathode and an amalgamated zinc plated anode were used.

Table 6—Equivalent Conductance of an Infinite Dilution of Caustic Potash

°C	°F	λ_∞
18	64.4	238.7
18	64.4	239
25	77.0	274.9
25	77.0	274.9
25	77.0	238.92
25	77.0	285.46
90	194.0	599

The equivalent conductance "λ" is expressed in $cm^2 \cdot ohm^{-1} \cdot mol^{-1}$.

Table 7—Heat of Neutralization at 20°C

Acid	KOH ΔH (cal/mol)	NaOH ΔH (cal/mol)
HCl	14014	13895
HBr	13988	13843
HI	13915	13779
HNO_3	14086	13836

Table 8—Heat of Solution of Caustic Potash

°C	°F	Mole H_2O/Mole KOH	Heat of Solution kcal/mol	 BTU/lb
11.4	52.5	260	12.46	399.9
18	64.4	250	13.29	426.5
100	212	260	16.8	539.2

Table 9—Index of Refraction of Caustic Potash Solutions

at 17.5°C (63.5°F)		at 18°C (64.4°F)	
%KOH	n	%KOH	n
0.1785	1.33358	0.5927	1.3344
0.9219	1.33513	1.1413	1.33552
2.7984	1.33896	2.7361	1.33872
4.7183	1.34275	5.374	1.34398
6.681	1.34650	10.311	1.35366
8.659	1.35021	20.926	1.37306
10.507	1.35388		
12.401	1.35750		
14.305	1.36109		
16.189	1.36464		
17.323	1.36675		

Table 10—Molar Magnetic Rotation for Caustic Potash Solutions (14.78%) at 0°C

Wave Length (Å)	4360	5460	5780
[M]	2.88	2.75	2.87

Table 11—Molar Magnetic Susceptibility of Caustic Potash Solutions

-21.2×10^{-6} cm^3/mol

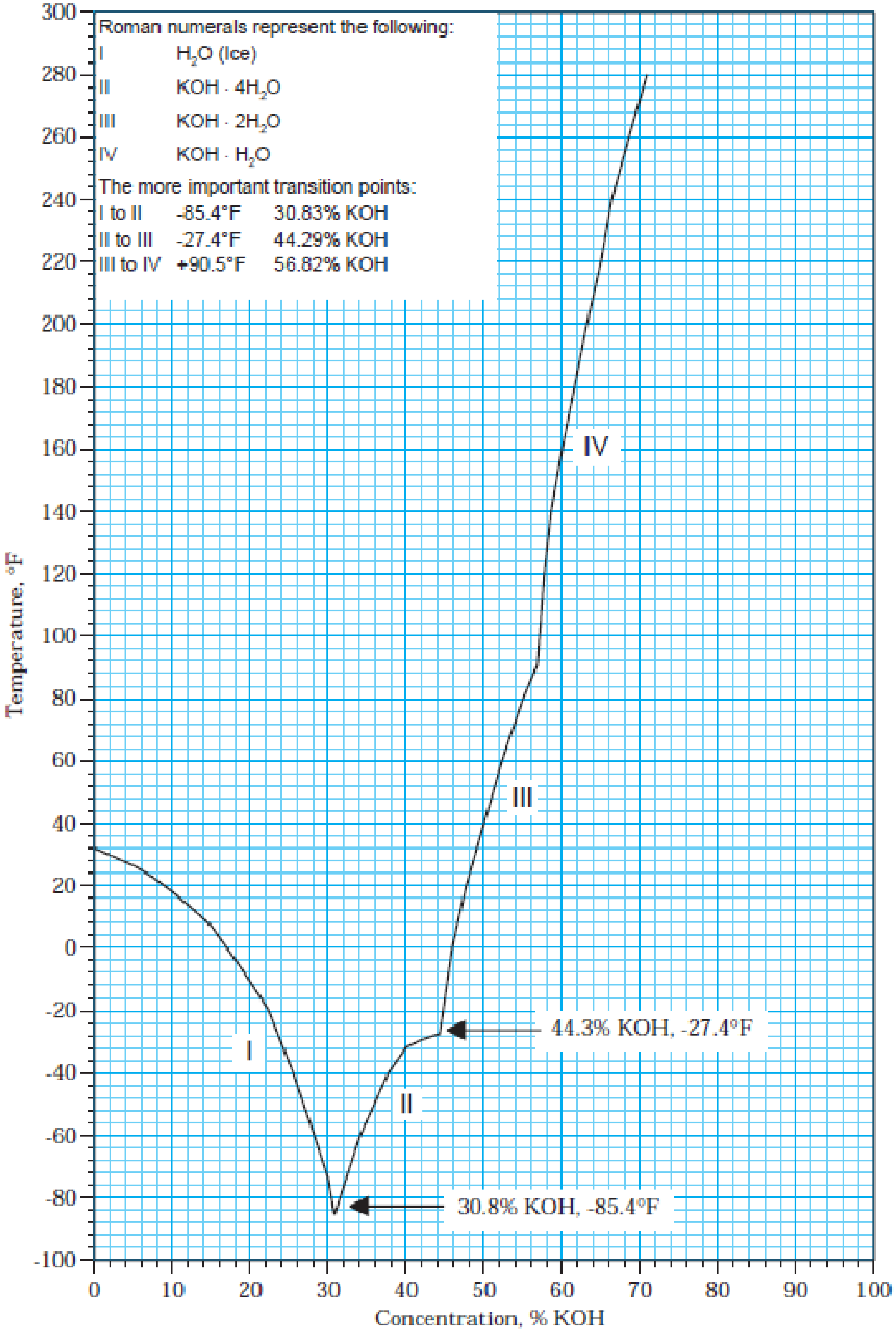
-22.0×10^{-6} cm^3/mol

Table 12—Molar Refraction, R_M (Lorentz-Lorentz), Sodium D-Lines, for Caustic Potash Solutions at 18°C (64.4°F)

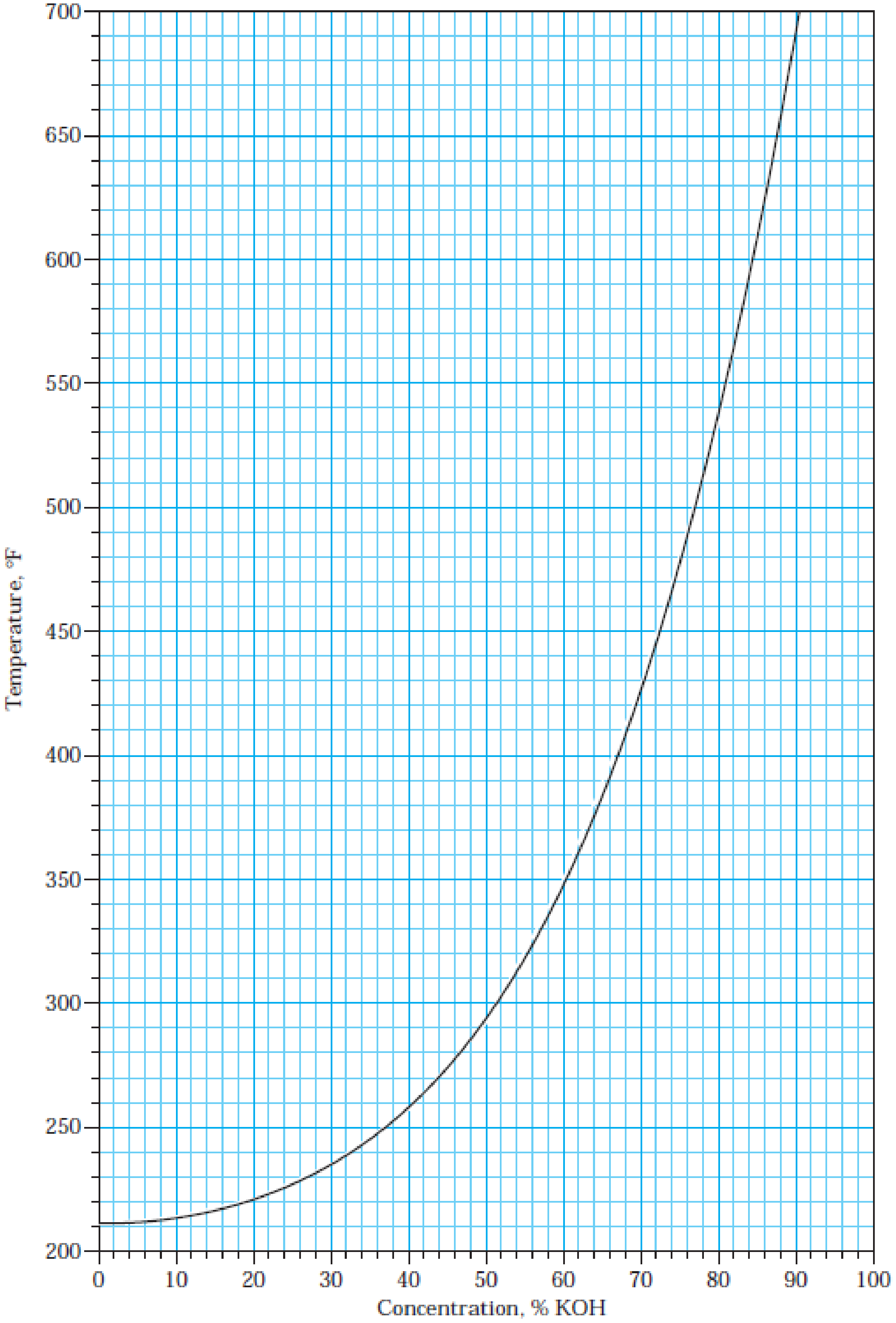
%KOH	0	1.1	2.7	5.3	10.3	19.0
R_M (cm^3/mol)	7.36	7.31	7.28	7.28	7.29	7.19

TECHNICAL DATA

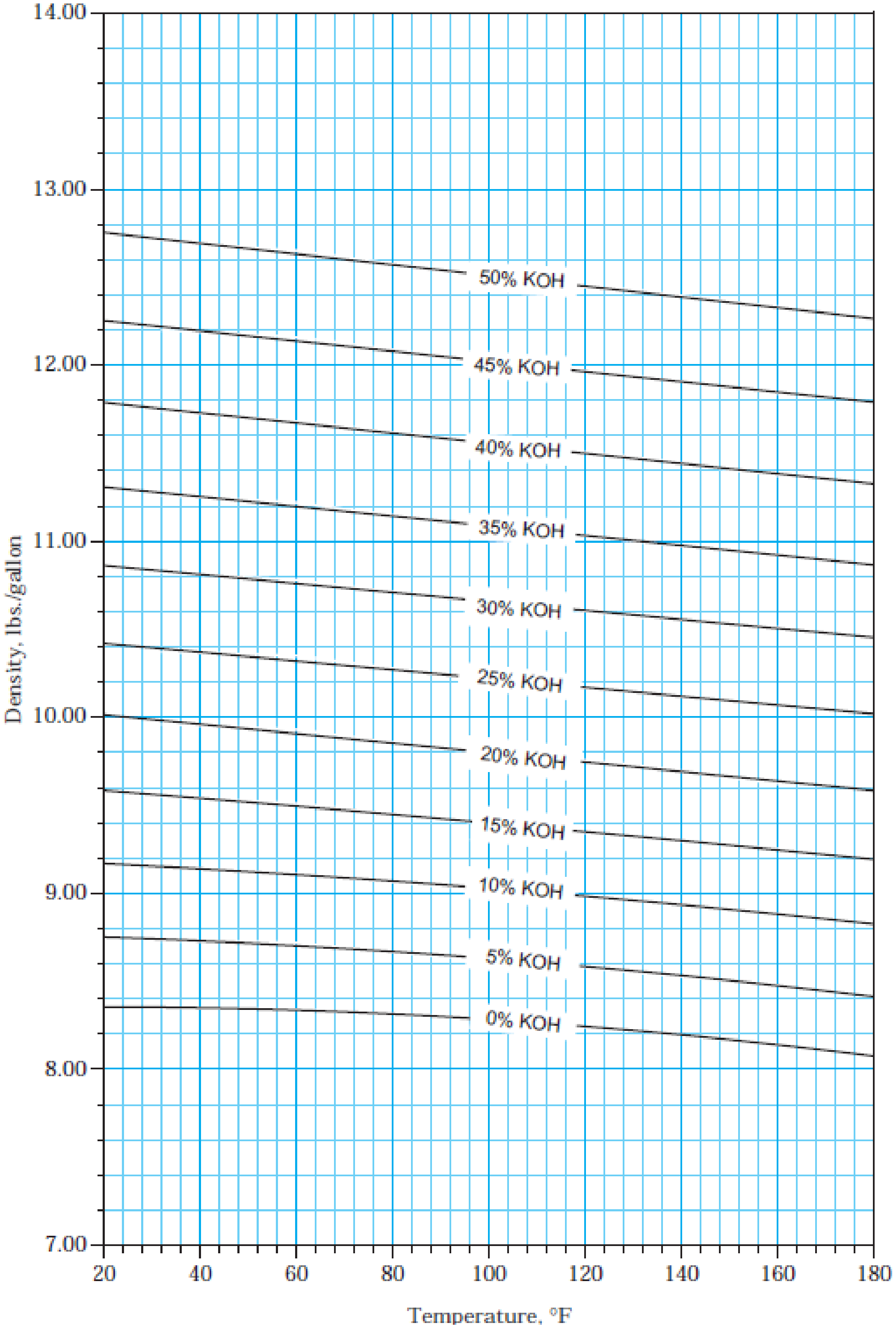
Graph 1—Freezing Points of Aqueous KOH Solutions



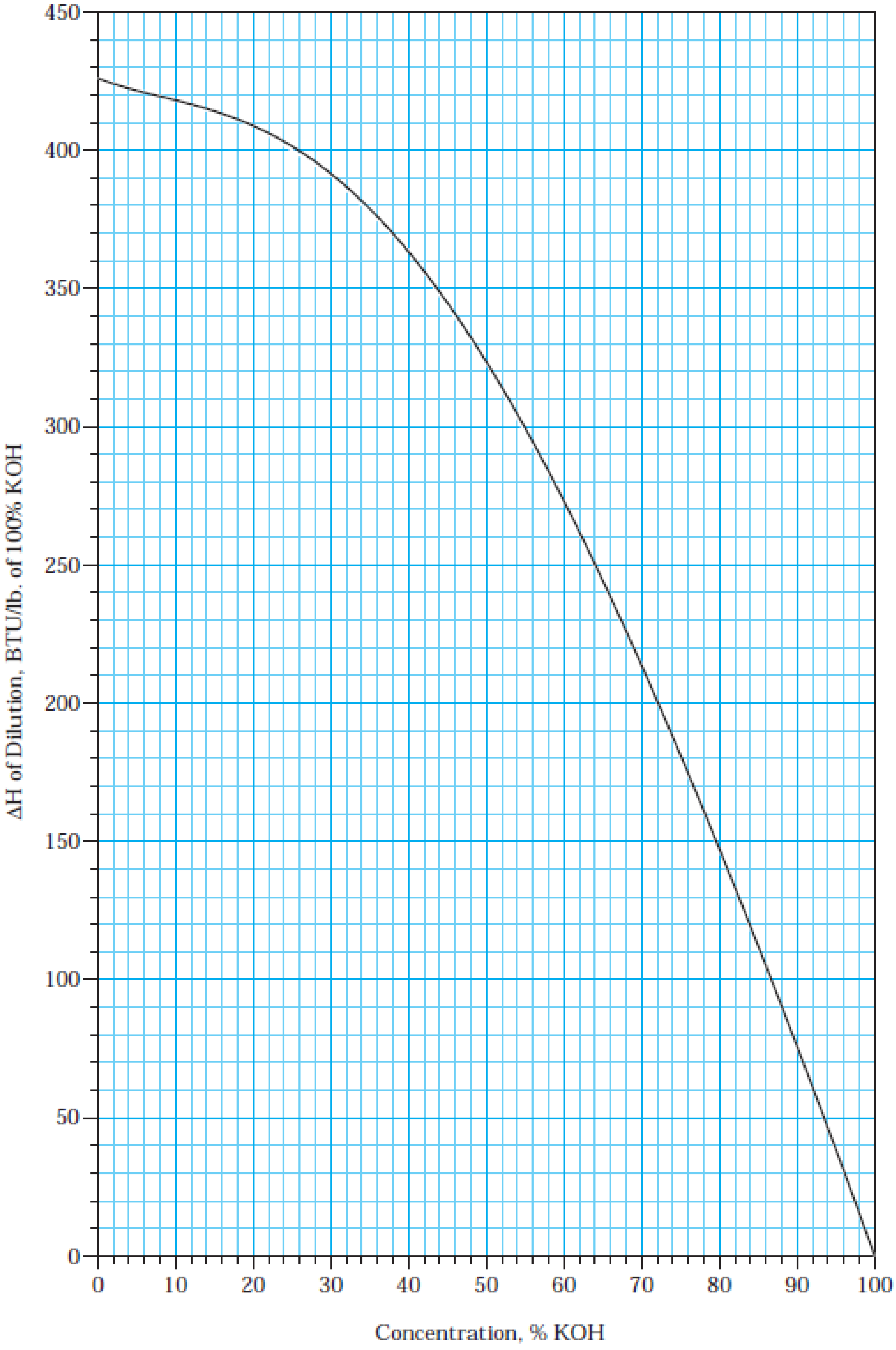
Graph 2—Boiling Points of Aqueous KOH Solutions at Atmospheric Pressure



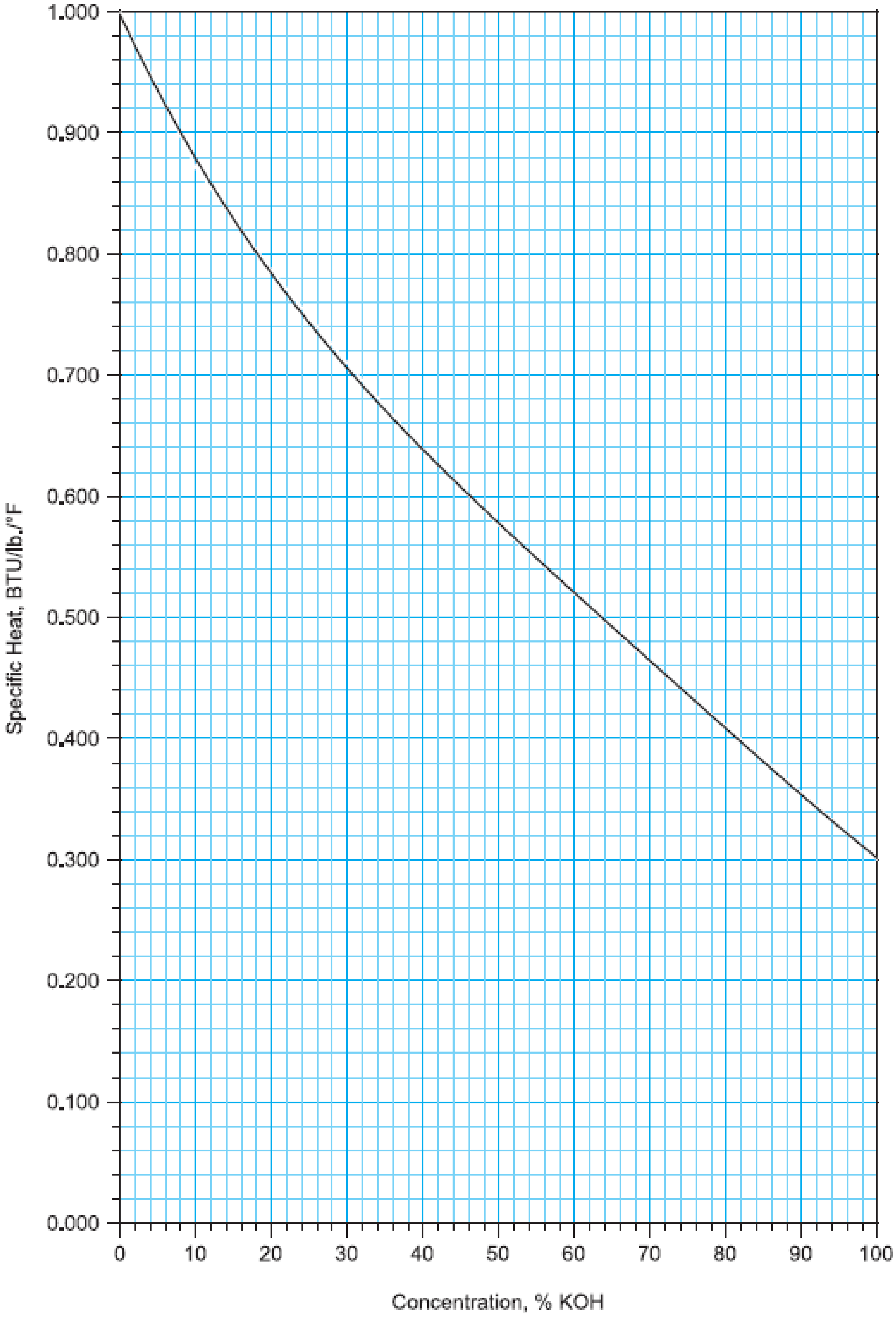
Graph 3—Density of KOH Solutions at Various Temperatures



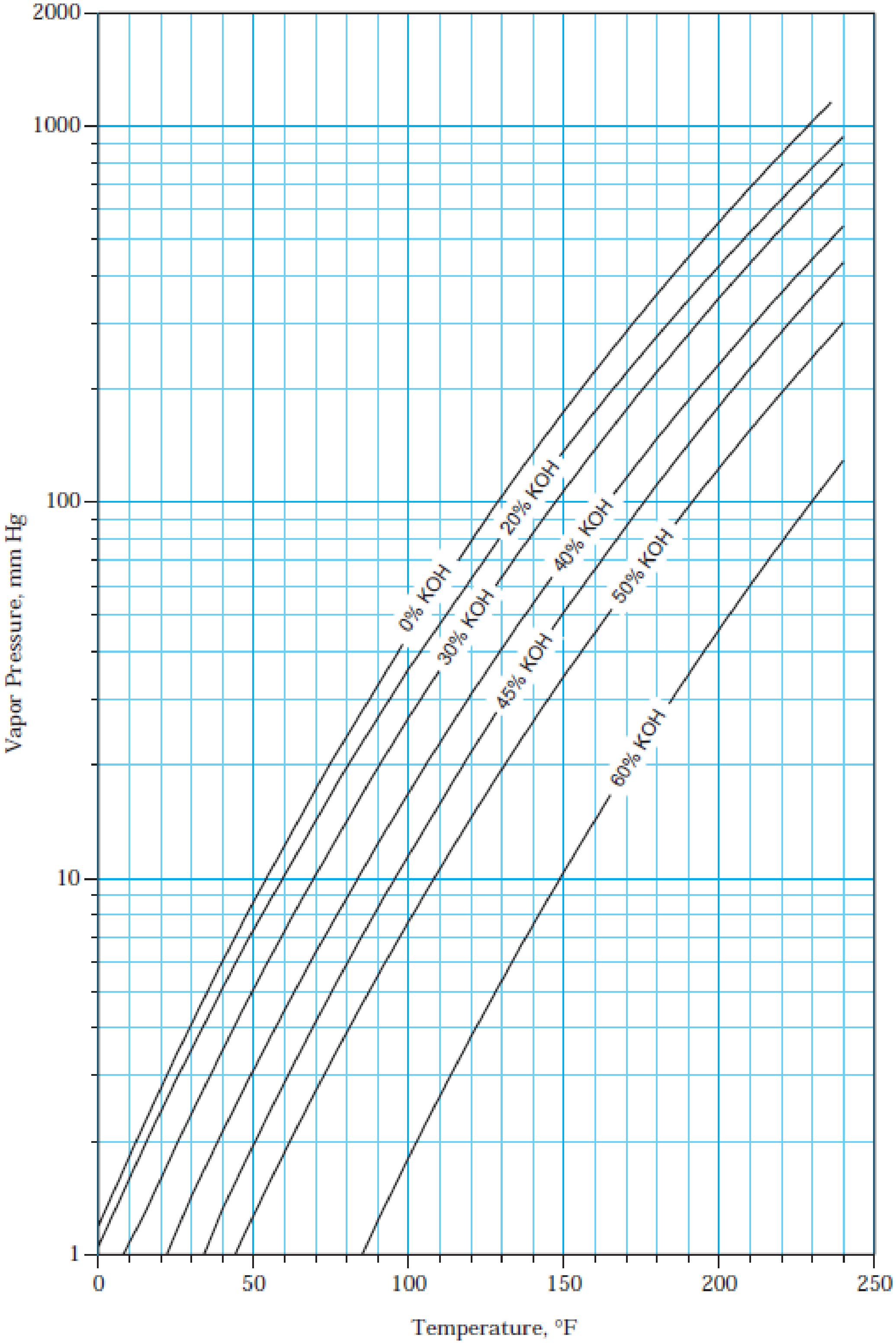
Graph 4—Heat of Solution for Aqueous KOH Solutions at 64.4°F



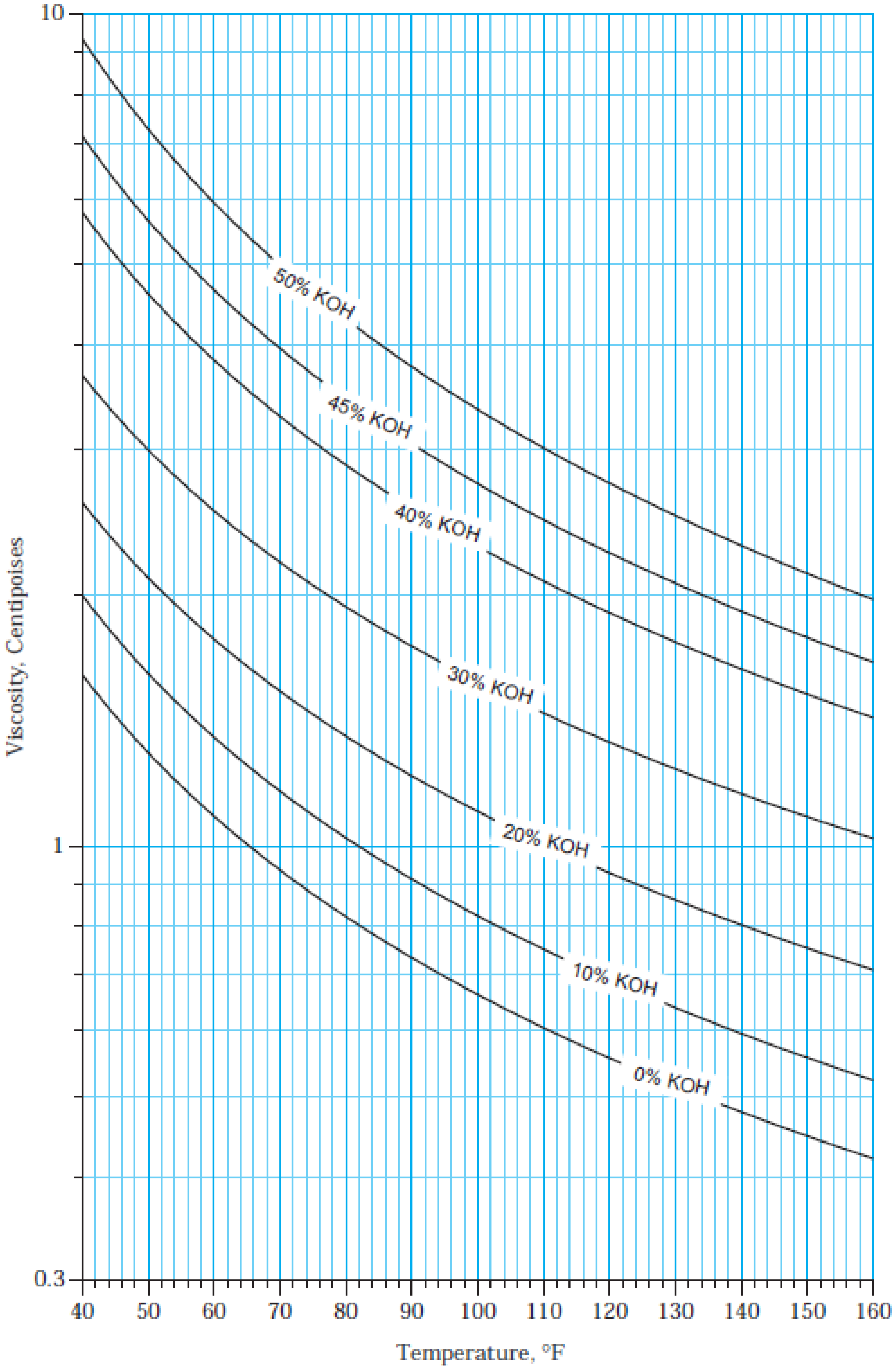
Graph 5—Specific Heat of Aqueous KOH Solutions at 64.4˚F



Graph 6—Vapor Pressure of Aqueous KOH Solutions



Graph 7—Viscosity of Aqueous KOH Solutions



DETERMINATION OF THE TOTAL ALKALINITY OF CAUSTIC POTASH

PURPOSE AND THEORY

The accurate determination of the total alkalinity value for caustic potash is important. It is used to calculate the weight of product shipped and ultimately the amount of money billed for the shipment.

Total alkalinity in caustic potash products is determined by titration of a sample with a standardized solution of 1N hydrochloric acid. Modified methyl orange indicator is used to determine the titration endpoint.

This procedure is based on ASTM E291, Standard Test Methods for Chemical Analysis of Caustic Soda and Caustic Potash.

APPARATUS

100mL Buret; Class A Volumetric, (Fisher Scientific Cat.#: 03-700-22D or equivalent)

Analytical Balance; 200g capacity, 0.1mg readability (Mettler ML204 or equivalent)

250mL Erlenmeyer Flasks; (Fisher Scientific Cat.#: 10-090B or equivalent)

Magnetic Stirrer; (Fisher Scientific Cat.#: 14-493-120SQ or equivalent)

Magnetic stirring bars; (Fisher Scientific Cat.#: 14-513-60 or equivalent)

REAGENTS

1N Hydrochloric Acid; measure 83.0mL of ACS Reagent grade concentrated hydrochloric acid into a graduated cylinder and transfer it to a 1L volumetric flask containing approximately 500mL of deionized water. Dilute to volume with additional water, mix well and store in a tightly closed container. A prepared solution of 1N HCl can also be purchased (Fisher Scientific Cat.# SA48-1 or equivalent). Hydrochloric Acid must be standardized to ±0.0001N before use.

Sodium Carbonate; anhydrous, 99.5% min., (Fisher Science Cat.#: S263-500 or equivalent.) Dry at 250° C in a platinum or porcelain crucible for 4 hours. Store in a desiccator.

Modified Methyl Orange Indicator; dissolve 0.14g of Methyl Orange (Fisher Cat#: M216-25) and 0.12g of Xylene Cyanol FF (Fisher Cat.#: BP125-100) in deionized water and dilute to 100mL.

Water, Deionized & Carbon Dioxide free; boil and cool the deionized water or purge it with nitrogen for two hours.

SAFETY

Refer to the SDS for the proper handling procedures for each of the chemicals listed in this procedure.

Caustic potash is a strong base. Hydrochloric acid is a strong acid. These chemicals are corrosive to body tissue and can cause immediate and severe burns to eyes. Wear proper gloves, proper eye protection and other protective clothing when handling these chemicals.

A. STANDARDIZATION OF 1N HYDROCHLORIC ACID

1. Weigh 4.2g of sodium carbonate to the nearest 0.0001g into a weighing dish. Carefully transfer to an Erlenmeyer flask. Add 75mL of deionized water and swirl to dissolve. Add three drops of the modified methyl orange indicator and titrate with the HCl solution to a steel gray color change.

2. The following formula is used to calculate the normality of the HCl.

   Let:

   N = Normality of HCl
   W = Weight (g) of Na_2CO_3 used
   V = Volume (mL) of HCl required to endpoint.
   Milliequivalent weight of Na_2CO_3 = 0.053

   N = W / 0.053 / V

   Determine the normality by averaging the result of at least three titrations.

B. ANALYSIS

1. In a clean, dry Erlenmeyer flask, accurately weigh, to the nearest 0.001g an amount of sample described in the table below. Weighing should be performed as rapidly as possible.

   The sample sizes are:

45% KOH	8-9g
50% KOH	7-8g
Anhydrous KOH	3-4g

2. Immediately add 50 mL of deionized water, making sure the sides of the beaker are washed down.

3. Add 3 to 4 drops of modified methyl orange indicator and carefully add the magnetic stirring bar.

4. Titrate the sample to a steel gray color with 1N HCl. Samples should be titrated as soon as possible to avoid pick up of carbon dioxide from the air.

5. Record the volume of acid required to reach this color. Estimate the Buret reading to the nearest 0.02mL.

C. CALCULATIONS

The following are formulas used to calculate total alkalinity.

Let:

W = Weight (g) of sample titrated
N = Normality of HCl
V = Volume (mL) of HCl required
Milliequivalent wt. of KOH = 0.05611

$$\%KOH = \frac{(V)\ (N)\ (0.05611)\ (100)}{W}$$

EXAMPLE

8.470g of caustic potash required the addition of 68.62mL of 1.0011N HCl to reach the modified methyl orange endpoint.

$$\%KOH = \frac{(V)\ (N)\ (0.05611)\ (100)}{W}$$

$$\%KOH = \frac{(68.62)\ (1.0011)\ (5.611)}{8.470}$$

%KOH = 45.51%

QUALITY ASSURANCE

With each batch of samples being analyzed, at least one of the samples should be analyzed in duplicate. On a regular basis, samples that have been previously analyzed for total alkalinity should be reanalyzed and the results compared with OxyChem specifications.

Hydrochloric acid should be restandardized at least monthly.

DETERMINATION OF POTASSIUM HYDROXIDE IN CAUSTIC POTASH

PURPOSE AND THEORY

The potassium hydroxide content of caustic potash is determined by adding barium chloride to a prepared sample and titrating with 1 N HCl to the phenolphthalein end point. The results are reported as percent KOH on a sample weight basis.

APPARATUS

100mL Buret; Class A Volumetric, (Fisher Scientific Cat.#: 03-700-22D or equivalent)

Analytical Balance; 200g capacity, 0.1mg readability (Mettler ML204 or equivalent)

250mL Erlenmeyer Flasks; (Fisher Scientific Cat.#: 10-090B or equivalent)

Magnetic Stirrer; (Fisher Scientific Cat.#: 14-493-120SQ or equivalent)

Magnetic stirring bars; (Fisher Scientific Cat.#: 14-513-60 or equivalent)

REAGENTS

1N Hydrochloric Acid; the preparation of this reagent is described in the method for: “Determination of Total Alkalinity”.

1% Phenolphthalein Indicator; dissolve 1g of phenolphthalein (Aldrich Cat.#: 105945 or equivalent) in 100mL of methanol.

10% Barium Chloride; dissolve 120g of $BaCl_2.2H_2O$ (Fisher Scientific Cat.#: B34-500 or equivalent) in 880mL of deionized water.

Water, Deionized & Carbon Dioxide free; boil and cool the deionized water or purge it with nitrogen for two hours.

SAFETY

Refer to the SDS for the proper handling procedures for each of the chemicals listed in this procedure. Caustic potash is a strong base.

Hydrochloric acid is a strong acid. These chemicals are corrosive to body tissue and can cause immediate and severe burns to eyes. Wear proper gloves, proper eye protection and other protective clothing when handling these chemicals. Barium chloride is highly toxic. Avoid inhaling barium chloride dust.

A. STANDARDIZATION OF 1N HYDROCHLORIC ACID

Standardization procedure is described in the method for: “Determination of Total Alkalinity”.

B. ANALYSIS

1. In a clean, dry Erlenmeyer flask, accurately weigh, to the nearest 0.001g an amount of sample described in the table below. Weighing should be performed as rapidly as possible.

   The sample sizes are:

45% KOH	8-9g
50% KOH	7-8g
Anhydrous KOH	3-4g

2. Immediately add 100 mL of barium chloride solution, making sure the sides of the beaker are washed down.

3. Add 3 to 4 drops of phenolphthalein indicator and carefully add the magnetic stirring bar.

4. Titrate the sample with 1N HCl until the pink color changes to water white. The sample should be titrated as soon as possible to avoid pick up of carbon dioxide from the air.
5. Record the volume of acid required to reach this color. Estimate the Buret reading to the nearest 0.02mL.

C. CALCULATIONS

The following are formulas used to calculate %KOH.

Let:

W = Weight (g) of sample titrated
N = Normality of HCl
V = Volume (mL) of HCl required
Milliequivalent wt. of KOH = 0.05611

$$\%KOH = \frac{(V)\,(N)\,(0.05611)\,(100)}{W}$$

EXAMPLE

8.470g of caustic potash required the addition of 68.52mL of 1.0011N HCl to reach the phenolphthalein endpoint.

$$\%KOH = \frac{(V)\,(N)\,(0.05611)\,(100)}{W}$$

$$\%KOH = \frac{(68.52)\,(1.0011)\,(5.611)}{8.470}$$

$$\%KOH = 45.44\%$$

QUALITY ASSURANCE

For each batch of samples being analyzed, at least one of the samples should be analyzed in duplicate. On a regular basis, samples that have been previously analyzed for total alkalinity should be reanalyzed and the results compared with OxyChem Specifications.

Hydrochloric acid should be restandardized at least monthly.

DETERMINATION OF POTASSIUM CARBONATE IN CAUSTIC POTASH (Gravimetric)

PURPOSE AND THEORY

The potassium carbonate content of a sample of caustic potash is determined by a direct gravimetric method. The method involves acidification of the caustic potash sample with dilute sulfuric acid, boiling, and weighing the carbon dioxide evolved. Accurate results can be obtained when the potassium carbonate content is 0.01% or greater. This method should be used to analyze samples of liquid caustic potash containing 0.01% or greater. This method should be used to analyze samples of 45 or 50% caustic potash containing 0.01 to 0.25% K_2CO_3.

This procedure is based on ASTM E291, Standard Test Methods for Chemical Analysis of Caustic Soda and Caustic Potash. Although it is included as a reference for anyone who may wish to perform the analysis, it should be noted that the procedure is rather lengthy, may show poor precision and is susceptible to error because of air intrusion into the apparatus.

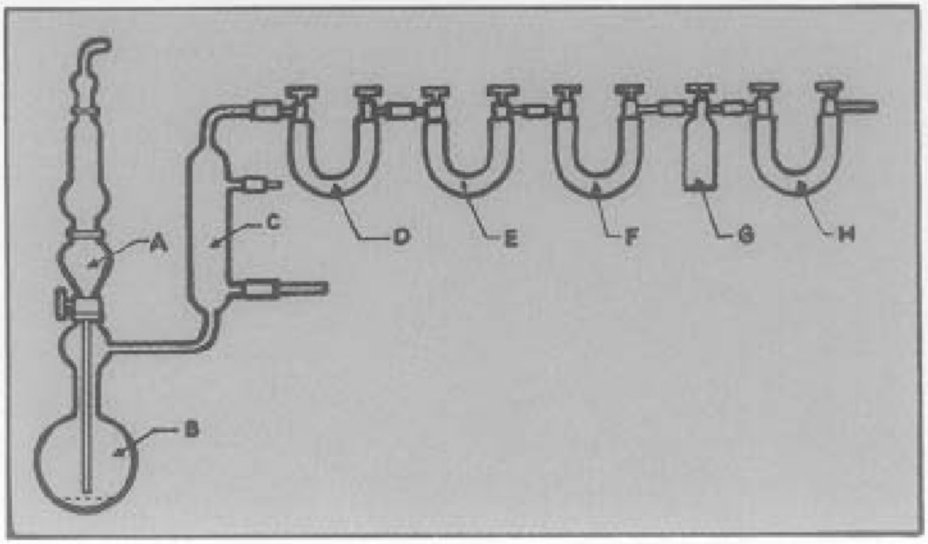
OxyChem typically utilizes a carbon analyzer in inorganic mode for the rapid and precise determination of carbonate content of caustic potash. For further information regarding the carbon analyzer method, please contact Technical Service.

APPARATUS

See the CO_2 train sketch below.

Air for sweep is drawn in through “A”. This air must be scrubbed free of CO_2. The ground-glass jointed tube fitted into the top of “A” should be packed with 8-20 mesh ascarite with a layer of anhydrous granular copper sulfate on top.

U-tube “D”
Add a few glass beads and 5 to 10mL of concentrated H_2SO_4. The acid takes up the bulk of the moisture passing through condenser “C” and should be changed often depending on frequency of use.



U-tube "E"
Pack with dehydrated copper sulfate pumice. This packing material is prepared by soaking pulverized pumice having the grain size of wheat in saturated copper sulfate solution drying at 150-180°F. The product must be kept in a well stoppered bottle.

U-tube "F"
Pack with anhydrous magnesium perchlorate. This removes all final traces of moisture carried through the system.

Ascarite - Absorbing Tower "G"
Pack inside tube with 8-20 mesh ascarite. Over the top layer add about 0.25 inch of magnesium pechlorate and cover with absorbent cotton. The cotton will prevent loss of weight due to carry-over of dust particles. After tower is packed, it should be hooked into the system and swept with CO_2-free air for a period of 15 to 20 minutes.

U-Tube "H"
Pack with 8-20 mesh ascarite.

REAGENTS

Sulfuric Acid; 12N with 27.8g/L $FeSO_4 \cdot 7H_2O$

Sulfuric Acid, concentrated.

Ascarite II; 8-20 mesh (sodium hydroxide coated silica)

Magnesium Perchlorate, anhydrous

Copper (II) Sulfate, anhydrous

Water, Deionized & Carbon Dioxide free; boil and cool the deionized water or purge it with nitrogen for two hours.

SAFETY

Caustic potash as dust or mist is intensely irritating to the respiratory system, skin, and eyes. Become familiar with the first aid measures recommended in this handbook.

When preparing 12N sulfuric acid, the concentrated acid must be poured slowly into water with constant stirring.

Wear safety glasses with side shields when handling caustic potash samples or acid solutions.

1. Sample Preparation

50% liquid caustic potash will solidify at 40°F. If the sample is solidified at the time of analysis, it may be thawed out by placing the container in hot water until no solids are present. The lip of the bottle may be wiped before the sample is poured into a weighing bottle.

No special preparation is required for anhydrous samples.

Carbonate and moisture pickup should be avoided by rapid sample handling.

In all cases, samples for carbonate analysis should be the first taken from the sample bottle to minimize carbon dioxide pickup from the atmosphere.

2. Analysis

The train must be conditioned daily before any samples are run. This is done by making a regular determination using a sample that contains carbonate. Following this, a blank should be run on the train to make sure the train is leak free. This is done by making a regular determination but omitting the sample. If the ascarite weighing tower gains more than 0.2mg in weight during the blank run, the train probably has a leak.

After the train has been conditioned and found to be leak free, the samples are run as follows:

1. Two absorbing towers (G) must be conditioned and weighed prior to analysis. These will be called G1 and G2 in the procedure. The use of two towers will enable the analyst to conserve time when performing more than one analysis.
2. Weigh a sample of at least 20g (50% basis) or large enough to contain 5mg of CO_2 into a flask "B" using an analytical balance. Add 4 or 5 glass beads and 80mL of CO_2-free deionized water and immediately place the flask into its proper position in the train.
3. Add 50mL of 12N sulfuric acid to funnel "A".
4. Place tared tower G1 between U-tubes "F" and "H".
5. Open the system starting at U-tube "H" and working back to "D".
6. Open cock on funnel "A" and allow acid to run into flask "B" and immediately hook vacuum line to tube "H". Adjust the flow of air to 4 to 5 bubbles per second through the tip of the stem of funnel "A".
7. Apply heat to flask "B" and bring to a boil. Hold "B" contents to boiling point for 3 minutes and remove heat.
8. Sweep the system for 20 minutes. While this is being done, the next sample can be weighed into another flask (B), and the beads and distilled water added. This flask is then stoppered and set aside until needed.

9. At the end of 20 minutes, the vacuum line is removed, tower G1 is shut off and removed and tower G2 placed into position. The cock on funnel “A” is closed and 50mL of 12N sulfuric acid is again added to funnel “A.
10. ”Flask “B” is removed, the stem of funnel “A” is washed down with deionized water and the new sample is placed into position.
11. Tower G2 is opened and the procedure is repeated beginning at Step 6.
12. When G1 is removed from the train, a period of 20 minutes will condition the sample for weighing. During this 20 minute sweep time, another sample is prepared and tower G1 is reweighed in order to determine the weight of CO_2 found in the first sample. Tower G1 is then ready for Run No. 3.

CALCULATIONS

Report results as percent K_2CO_3 calculated to the nearest 0.01.

Let:

$W(CO_2)$ = Weight of CO_2 evolved
$W(S)$ = Weight of sample

$$\%K_2CO_3 = \frac{(W(CO_2))\ (3.136)\ (100)}{W(S)}$$

Note: Molecular weight of K_2CO_3 / Molecular weight of CO_2 = 138 / 44 = 3.136

EXAMPLE

A 50g sample was used and 0.0080g of CO_2 was absorbed in tower “G”, then:

$$\%K_2CO_3 = \frac{(0.0080)\ (3.136)\ (100)}{50}$$

$\%K_2CO_3 = 0.05\%$

DETERMINATION OF POTASSIUM CHLORIDE IN CAUSTIC POTASH

PURPOSE AND THEORY

Chloride is a contaminant in all grades of caustic potash. Potassium chloride is present at <60 ppm in 45% caustic potash. Higher concentrations of this compound can have undesirable effects in many applications of the product. Consequently, accurate determination of this impurity is most important.

When acid solutions of silver ion and an alkali thiocyanate are mixed in the presence of a ferric salt, the thiocyanate has a selective action toward silver, resulting in the formation of silver thiocyanate. Any excess of thiocyanate not required by the silver reacts with ferric salt to form reddish-brown ferric thiocyanate. This color indicates the completion of the reaction.

An excess of silver nitrate and the ferric indicator is added to a sample of caustic potash that has been acidified with nitric acid. Any chloride that is contained in the sample will react with the silver nitrate to form a silver chloride precipitate. The silver nitrate that is remaining in the sample solution after this reaction is titrated with a standardized solution of ammonium thiocyanate. The equations involved are:

$AgNO_3 + KCl$ " $AgCl + KNO_3$

$AgNO_3(excess) + NH_4CNS$ " $AgCNS + NH_4NO_3$

$6NH_4CNS + Fe_2(SO_4)_3$ " $2Fe(CNS)_3 + 3(NH_4)_2SO_4$
(reddish brown color)

This procedure is based on ASTM E291-09, Standard Test Methods for Chemical Analysis of Caustic Soda and Caustic Potash.

OxyChem typically utilizes turbidimetric determination, potentiometric titration (similar to ASTM E291, with modifications to account for the low chloride content of membrane grade caustic potash) or ion chromatography. For further information regarding any of these methodologies, please contact Technical Service.

APPARATUS

25mL Buret; Class A Volumetric (Fisher Scientific Cat.#: 03-724-10A or equivalent)

20mL Pipet; Class A Volumetric, (Fisher Scientific Cat.# 13-650-2N or equivalent)

500mL Erlenmeyer flasks; (Fisher Cat.#: 10-090C or equivalent)

Magnetic Stirrer; (Fisher Scientific Cat.#: 14-493-120SQ or equivalent)

Magnetic stirring bars; (Fisher Scientific Cat.#: 14-513-60 or equivalent)

Analytical Balance; 200g capacity, 0.1mg readability (Mettler ML204 or equivalent)

REAGENTS

Water, Deionized

0.1N Silver Nitrate; accurately weigh 16.99g of ACS Reagent grade silver nitrate (dried at 110°C for 1 hr) and transfer to a 1L volumetric flask. Dilute to volume with deionized water, mix well and store in a tightly closed amber container. Silver nitrate and its aqueous solutions are photodecomposed by light and should be stored in a dark place.

0.1N Ammonium Thiocyanate; accurately weigh 7.612g of ACS Reagent grade ammonium thiocyanate and transfer to a 1L volumetric flask. Dilute to volume with deionized water, mix well and store in a tightly stoppered glass bottle. The thiocyanate solution must be standardized to within ±0.0001N prior to use.

Ferric Indicator, *saturated;* dissolve 8g of $NH_4Fe(SO_4)_2 \cdot 12H_2O$ (Aldrich Cat.# 221260 or equivalent) to 20mL of deionized water and add a few drops of concentrated nitric acid

1% Phenolphthalein Indicator; dissolve 1g of phenolphthalein (Aldrich Cat.#: 105945 or equivalent) in 100 mL of methanol.

Nitric Acid, 1:1 (v/v); slowly pour 500mL of ACS Reagent grade nitric acid in 500mL of deionized water as it is stirring. Allow the solution to cool.

SAFETY

Refer to the SDS for the proper handling procedures for each of the chemicals listed in this method.

Caustic potash is a strong base and nitric acid is a strong acid. These chemicals are corrosive to body tissue and can cause immediate and severe burns to eyes. Wear proper gloves, proper eye protection and other protective clothing when handling these chemicals.

Silver Nitrate is a strong oxidizing agent. Wear rubber gloves when handling. Contact with skin causes a black discoloration. Keep away from heat, sparks and open flames.

A. STANDARDIZATION OF 0.1N SILVER NITRATE

Since this procedure determines the chloride content of a sample by comparing the amount of unreacted silver nitrate remaining in a sample with the amount that is remaining in a reagent blank, the exact normality of the silver nitrate need not be known. If a reagent blank is not used, silver nitrate standardization is essential. A manual titration method is described in ASTM-E200, Standard Practice for Preparation, Standardization and Storage of Standard Solutions for Chemical Analysis.

B. STANDARDIZATION OF 0.1N AMMONIUM THIOCYANATE

1. Use a volumetric pipet to transfer 20.00 mL of freshly standardized 0.1 N silver nitrate into a 250 mL Erlenmeyer flask containing 50 mL deionized water, 5 mL of 1:1 nitric acid and 1 mL of ferric indicator. Titrate the AgNO3 with the NH4SCN solution until the first permanent reddish-brown color appears and persists after vigorous shaking for 15 seconds. Record the volume of NH4SCN required. Repeat the above procedure on at least three more solutions of silver nitrate.
2. Use the following formula to calculate the normality of the ammonium thiocyanate solution:

   N1 = (N2) (V2) / (V1)

   where:

   N1 = Normality of NH_4SCN
   N2 = Normality of $AgNO_3$
   V1 = Volume of NH_4SCN required
   V2 = Volume of $AgNO_3$ added
3. Determine the normality by averaging the results of at least three titrations.

C. PROCEDURE

1. In a clean dry Erlenmeyer flask, accurately weigh, to the nearest 0.01g, 80g of 45 or 50% liquid caustic potash or 40 to 80g of anhydrous caustic potash. Weighing should be performed as rapidly as possible.
2. Immediately add 100mL of deionized water, making sure the sides of the beaker are washed down.
3. Add 2 drops of 1% phenolphthalein indicator and carefully neutralize the sample with 1:1 nitric acid.

   Caution: The sample solutions generate considerable heat when being neutralized with acid. The flask should be continuously cooled in an ice bath while the acid is slowly added. After the phenolphthalein endpoint has been reached (color changes from pink to colorless), add an additional 5.0mL of acid.
4. Allow the solution to cool to room temperature and add a stirring bar to the flask.
5. Using a volumetric pipet add 20.00mL of 0.1N silver nitrate, also add approximately 1mL of the ferric indicator solution.

   Note: Sample solutions should be titrated within several minutes of adding the silver nitrate. The silver chloride decomposes when exposure to light giving the solution a purplish color. This color can interfere with an accurate determination of the endpoint color change.

6. Prepare a reagent blank by adding two drops of phenolphthalein, 5mL nitric acid, 20.00mL silver nitrate solution and 1mL of ferric indicator to a flask containing 100 mL of deionized water and a stirring bar.
7. Place the flask containing the reagent blank on a magnetic stirrer and titrate the solution with 0.1N ammonium thiocyanate until a reddish-brown color persists for at least 15 seconds. Record the volume of NH_4SCN required to reach the color change.

   Note: From the outset of the back titration with ammonium thiocyanate, an appreciable quantity of silver ions are absorbed on the surface of the precipitates. Because of this, there is a premature appearance of the endpoint color. Vigorous stirring or shaking of the solution is essential to bring about desorption of silver ions from the precipitates so they can react with the thiocyanate.
8. Titrate the sample solution with 0.1N ammonium thiocyanate until the same color change is reached and record the volume of NH_4SCN.

   Note: As the endpoint is approached, increasing amounts of silver thiocyanate precipitating out of solution will actually increase the solubility of silver chloride. Silver chloride that has precipitated will redissolve, allowing additional silver ions to react with the thiocyanate. This causes a fading endpoint and results in low chloride values. For samples containing concentrations of chloride greater than 0.01%, it is advisable to filter the sample solution through semi-quantitative paper after the addition of silver nitrate but prior to titration with thiocyanate. Removing most of this precipitate will greatly decrease the amount of silver that can be redissolved during the titration.

   Note: The white precipitate of silver thiocyanate interferes with observation of the color change at the titration endpoint. It is sometimes helpful to stop the stirring or shaking of the sample and allow the precipitate to settle, in order to observe the color of the sample solution. If it is determined during this observation the endpoint has not yet been reached, resume vigorous stirring before addition of more NH_4SCN.

D. CALCULATIONS

The following is the formula used to calculate the percent chloride in the sample.

Let:

W = Weight of sample titrated
N = Normality of NH_4SCN
V1 = Volume of NH_4SCN required to titrate blank
V2 = Volume of NH_4SCN required to titrate sample
Milliequivalent wt. of Cl = 0.03545

$$\%Cl = \frac{(V1 - V2)\ (N)\ (0.03545)\ (100)}{W}$$

Calculate the percentage of potassium chloride as follows:

$$\%KCl = (\%Cl)\ (2.1029)$$

EXAMPLE

79.28g of 45% caustic potash required the addition of 19.54mL of 0.1005N NH_4SCN to reach the titration end-point while the reagent blank required 19.95mL of NH_4SCN to reach the same endpoint.

$$\%Cl = \frac{(V1 - V2)\ (N)\ (0.03545)\ (100)}{W}$$

$$\%Cl = \frac{(19.95 - 19.54)\ (0.1005)\ (3.545)}{79.28}$$

$$\%Cl = 0.0018$$

$$\%KCl = (\%Cl)\ (2.1029)$$

$$\%KCl = (0.00180)\ (2.1029)$$

$$\%KCl = 0.0038\% \text{ or } 38 \text{ ppm}$$

QUALITY ASSURANCE

Because of difficulties in determining the exact endpoint when using this method, only skilled laboratory personnel should attempt to perform these titrations.

On a regular basis, samples that have been previously analyzed for chloride content should be reanalyzed and the results compared.

DETERMINATION OF IRON IN CAUSTIC POTASH

PURPOSE AND THEORY

Iron can result from contamination during storage or transport of the product. Since iron is often detrimental to the end use of the product, accurate quantitation of this element is essential.

Caustic potash is neutralized with hydrochloric acid and the resulting solution buffered with sodium acetate. Hydroxylamine hydrochloride reduces any iron present in the ferric state to the ferrous state. o-phenanthroline (1,10-phenanthroline Monohydrate) forms an orange-red complex with ferrous iron. The intensity of the color is proportional to the amount of iron present. By measuring the color intensity with a spectrophotometer, the concentration of iron in a sample of caustic potash can be determined.

This procedure is based on ASTM E291, Standard Test Methods for Chemical Analysis of Caustic Soda and Caustic Potash.

APPARATUS

Visible Spectrophotometer; able to measure absorbance or percent transmittance at 510 nanometers.

Analytical Balance; 0.01g readability

1L Volumetric Flask; Class A (Fisher Scientific Cat.# 10-210-8G or equivalent)

100 mL Volumetric Flask; Class A (Fisher Scientific Cat.# 10-210-8C or equivalent)

Pipets; Volumetric, Class B;

1mL (Fisher Scientific Cat.# 13-650B or equivalent)

2mL (Fisher Scientific Cat.# 13-650C or equivalent)

5mL (Fisher Scientific Cat.# 13-650F or equivalent)

10mL (Fisher Scientific Cat.# 13-650L or equivalent)

15mL (Fisher Scientific Cat.# 13-650M or equivalent)

Indicator Paper; Hydrion, pH 3.0 to 5.5: (Fisher Scientific Cat.# 14-853-70 or equivalent)

Disposable plastic pipets; ("Dispopipet")

Cuvettes, quartz: appropriate to the spectrophotometer in use, 25mm diameter is typical but other path lengths providing detection limits suitable for the user are acceptable.

REAGENTS

Deionized Water

Hydrochloric Acid, concentrated: reagent grade (Fisher Scientific Cat.# A144SI-212 or equivalent)

Sodium Acetate, 164g/L; weigh 164.0±0.1g of sodium acetate (Fisher Scientific Cat.# S210-500 or equivalent) dissolve and dilute to 1L with deionized water in a 1L volumetric flask

Hydroxylamine Hydrochloride, 100g/L; weigh 100.0±0.1g of hydroxylamine hydrochloride (Fisher Scientific Cat.# H330-500 or equivalent) dissolve and dilute to 1L with deionized water in a 1L volumetric flask

o-Phenanthroline, 0.25%; weigh 2.50±0.05g of 1,10-Phenanthroline Monohydrate (Fisher Scientific Cat.# P70-10 or equivalent) dissolve and dilute to 1L with deionized water in a 1L volumetric flask

Iron Standard for ICP,**1000µg/mL**; (Fisher Scientific Cat.# PLFE2-2Y or equivalent)

SAFETY

Refer to the SDS for the proper handling procedures for each of the chemicals listed in this procedure.

Caustic potash is a strong base. Hydrochloric acid is a strong acid. The Iron Reference Solution is acidified with HNO_3. All of these chemicals are corrosive to body tissue and can cause immediate and severe burns to eyes. Wear proper gloves, proper eye protection and other protective clothing when handling these materials.

Refer to instrument manual for the proper use of equipment described in this method.

PROCEDURE

A. SAMPLE ANALYSIS

1. Weigh to the nearest 0.01g, 10g of anhydrous or 20g of liquid caustic potash into a 100mL volumetric flask. Record the sample weight.
2. Add deionized water to the flask till it is slightly less than half-full, and swirl to mix the solution.

3. SLOWLY and CAREFULLY, add concentrated hydrochloric acid to the flask while constantly swirling the flask. Continue to add until the solution is just acidic. Check the pH by dipping a clean glass rod into the flask and touching the rod to the pH indicator paper. If acidic, the paper will turn red. If it is still basic, the paper will be blue. If another type of indicator paper is used, verify the appropriate colors for the paper you are using.

   CAUTION: This is a reaction involving a strong base and a strong acid. Be sure to add the acid slowly and to maintain swirling so that spattering does not occur. The solution and flask will become quite warm. Cool the flask in an ice bath or under cold running water if desired.

4. If additional samples are to be analyzed, repeat steps 1 to 3 above for each sample.
5. Prepare a blank by adding about 40mL of deionized water and about 2mL of concentrated hydrochloric acid to a separate, 100mL volumetric flask.
6. Using a dispopipet, add sufficient sodium acetate solution to the flasks (the samples and the blank) to buffer the solution to pH 3.5±0.5 pH units. Check the pH by dipping a clean glass rod into the flask and touching the rod to the pH indicator paper. At the proper pH, the paper will have a light green color. If another type of indicator paper is used, verify the appropriate color for the paper you are using.
7. Pipet 5mL of hydroxylamine hydrochloride solution to each flask.
8. Pipet 5mL of o-Phenanthroline solution to each flask.
9. Fill the flasks to volume with deionized water and shake well to mix. Allow a minimum of 15 minutes for color development but complete the reading of the samples within 30 minutes.
10. Rinse a clean cuvette twice with small portions of the blank solution. Then fill the cuvette with the blank solution. Ensure there are no bubbles present and wipe off the outside of the cuvette with a soft, lint-free tissue. Place the cuvette into the spectrophotometer and zero the instrument at 0 (zero) absorbance (A) or 100% transmittance (T). Operate the spectrophotometer as directed in your instrument manual. Remove the cuvette from the instrument.
11. Fill a cuvette with the sample solution and load it into the spectrophotometer in the same fashion as described in step 10 above. Record the instrument reading as either A or T, depending upon how your calibration curve was constructed. (See section C on the next page.)

B. QUALITY CONTROL

1. Perform a duplicate analysis with each batch of samples. Simply repeat the Sample Analysis in section A above using an additional aliquot of a sample. If large numbers of samples are tested, it is suggested that a duplicate analysis is performed on one of every ten samples.
2. Perform a sample spike analysis with each batch of samples. To do so, prepare a second aliquot of a sample as directed in steps 1 and 2 of the Sample Analysis in section A. Then pipet 2mL of the 10µg/mL iron stock standard (see step 1 in section C on the next page) into the flask. Complete the sample preparation as described in the remaining steps of section A. This procedure provides about a 1µg/g (1ppm) spike. Different spike levels may be obtained by adding more or less of the iron stock standard or using different iron concentration solutions.
3. The relative percent difference between duplicates should be less than 20%.
4. The spike recovery should be in the 80 to 120% range.

C. SPECTROPHOTOMETER CALIBRATION

1. Prepare a stock 10µg/mL iron standard by pipetting 1mL of the 1000µg/mL iron standard to a 100mL volumetric flask and diluting to volume with deionized water.
2. Obtain six 100mL volumetric flasks and label them as 'Blank', '10µg', '20µg', '50µg', '100µg' and '150µg'. To each of these, add about 25mL of deionized water and 2mL of concentrated hydrochloric acid.
3. Pipet 1mL of the 10µg/mL iron stock standard (prepared in step 1 above) to the flask labeled '10µg'. Similarly, pipet 2mL of the iron stock to the flask labeled '20µg', 5mL to the flask labeled '50µg', 10mL to the flask labeled '100µg', and 15mL to the flask labeled '150µg'.

1. Complete the preparation and reading of the standards by following steps 6 through 11 in section A on the previous page.
2. Many spectrophotometers will allow storing the calibration curve directly on the instrument. If this is not the case, you may want to use a least squares regression analysis to store the calibration on a calculator or PC program. Finally, calibration curves may be drawn manually using normal graph paper if plotting concentration vs. absorbance (A) or semi-log paper if plotting concentration (linear axis) vs. transmittance (T - log axis).

CALCULATION

Calculate the ppm iron by dividing the micrograms of iron found from the calibration curve (C) by the sample weight (W):

ppm Fe = C / W

DETERMINATION OF OTHER ELEMENTS IN CAUSTIC POTASH

OxyChem typically utilizes inductively Coupled Argon Plasma-Atomic Emission Spectroscopy (ICAP-AES) for the analysis of metals or elements in caustic potash.

Hydride Generation Atomic Absorption (HG-AA) is used for the determination of antimony, arsenic and bismuth in order to obtain lower detection limits than are achievable via ICAP-AES.

For the determination of mercury, an automated cold vapor atomic absorption (CVAA) method, based on EPA Method 245.1 and ASTM E538, is utilized.

Note: OxyChem is a mercury-free producer of caustic soda and caustic potash. OxyChem last used mercury cells in 2008, becoming the first mercury-free producer of caustic potash in North America. OxyChem was already a mercury-free producer of caustic soda before 2008.

Since the operation of these instruments varies from manufacturer to manufacturer, a specific method is not included here. Follow your instrument manufacturer's instructions and recommendations for performing analysis of this type.

IMPORTANT!!!

Regardless of the brand of instrument, it is necessary to keep in mind that calibration standards must be similar to the sample being analyzed if results are to be considered accurate.

Caustic potash will form a significant amount of salt when neutralized; for example, potassium chloride if neutralized with hydrochloric acid or potassium nitrate if neutralized with nitric acid. Since the sample contains a high amount of dissolved solids, calibration standards must be prepared in a similar matrix or sample results will be in error.

One means of addressing this is to prepare standards containing the same amount of dissolved salt that a sample would contain. This requires obtaining high purity salts, which can be quite costly. Another more economical means of accomplishing the "matrix match" is to use calibration by standard addition.

In general, analytical results obtained for elements in caustic potash will be significantly higher than the actual values if the samples are analyzed using a calibration based on "clean" aqueous standards as might be used for water analysis.

BILLING FOR CAUSTIC POTASH

LIQUID CAUSTIC POTASH BILLING

Liquid caustic potash is typically sold as 45% or 50%. There is no standard billing basis. Typically 45% is billed on a 45% basis and 50% is billed on a 50% basis.

Caustic potash is billed per hundred weight, abbreviated CWT. “C” is the roman numeral for 100.

Example: A customer orders a truck of 50% caustic potash at $34.00/CWT. The net weight was 45,160 pounds and the %KOH was 50.60%.

1. Determine the billing weight in hundreds of pounds as 50%:

$$\frac{45{,}160\text{ lb}}{100\text{ lb/CWT}} \times \frac{50.60\%}{50.00\%} = 457.019\text{ CWT}$$

2. Invoiced amount:

457.019 CWT × $34.00/CWT = $15,538.65

DRY CAUSTIC POTASH BILLING

Dry caustic potash is billed per CWT with no adjustment for purity.

Example: A customer orders 45,000 pounds of caustic potash flake in 1,000 pound super saks at $34.75/CWT. The %KOH was 90.20%.

1. Determine the billing weight in hundreds of pounds:

$$\frac{45{,}000\text{ lb}}{100\text{ lb/CWT}} = 450.00\text{ CWT}$$

2. Invoiced amount:

450.00 CWT × $34.75/CWT = $15,637.50

NOTES

® Teflon is a registered trademark of DuPont de Nemours.

® Hastelloy is a registered trademark of Haynes International.

® Inconel is a registered trademark of Inco Limited.

® Monel is a registered trademark of Inco Limited.

BIBLIOGRAPHY

Cell diagrams (page 7) courtesy of www.eurochlor.org

Other pictures courtesy of www.oxychem.com

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