ARMAND PRODUCTS COMPANY

K_2CO_3

Potassium Carbonate Handbook

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Background

In 1986, two strong manufacturers in the chemical industry combined in a joint venture to form the Armand Products Company.

From the Occidental Chemical Corporation came the manufacturing capability for the production of the highest quality chemicals, with marketing and technical service expertise for product support.

From the Church & Dwight Co., Inc. came the sales and R&D functions to maintain the high level of customer service that our customers require.

As a market leader, the Armand Products Company provides the facilities and manpower needed to meet the requirements of our customers. Our two plants and four reactors run in the continuous mode, assuring an uninterrupted supply of potassium carbonate. An additional reactor is dedicated to the production of potassium bicarbonate. This manufacturing facility is located in Muscle Shoals, AL.

Armand Products Company has full integration with a dedicated production source of potassium hydroxide, the key raw material for making potassium carbonate. Potassium bicarbonate is produced by further carbonation of potassium carbonate. The Tennessee Valley Authority (TVA), one of the largest electricity generators in the United States, supplies power to the Muscle Shoals facility at a reasonably stable cost.

In order to meet the requirements of the many diverse applications, each product is available in several different grades. Various package forms are also available, ranging from bags to railcars. Smaller packages are available directly from the plant or at many warehouse and distribution points across the country.

Armand Products' commitment to quality and consistent performance is shown in its continued good standing as an ISO 9001:2015 certified and OSHA Star facility.

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I. Introduction

Potassium carbonate is one of the most important inorganic compounds used in industry even though it is as old as recorded history. Potassium carbonate was leached from ashes in Pompeii and mixed with slaked lime for soapmaking. The increase in the use of this alkali paralleled the growth of western civilization. So much wood was consumed in the production of potassium carbonate that the forests of Europe were threatened. At the time of the French Revolution, LeBlanc's invention allowed sodium carbonate to be substituted on a general basis.

The potassium carbonate that was recovered from ashes became the prime potassium compound before 1870. During these early times, potassium hydroxide (KOH, caustic potash) was made from potassium carbonate by reaction with calcium hydroxide. However, the recovery of potassium chloride in 1860 from "rubbish salt" in the Strassfurt, Germany salt mines changed this methodology. Today potassium hydroxide is produced through the electrolysis of potassium chloride brine. Subsequently, the KOH is carbonated with carbon dioxide to form potassium carbonate.

K_2CO_3 is the chemists' short-way of representing potassium carbonate or PotCarb as it is commonly called today. Although it is known by several other names, the chemical formula is the most definitive way to confirm this compound. Some of alternate nomenclature that may be used includes: PC, carbonate of potash, pearl ash and carbonic acid, dipotassium salt.

In everyday chemical technology, the choice between potassium and sodium carbonate is decided on the economics or some desired physical / chemical property. The principal reasons to utilize potassium carbonate are:

- source of potassium ion
- buffered alkalinity
- greater solubility for potassium vs. sodium carbonate
- potassium ion is more reactive than sodium ion
- replacement for sodium sensitive applications
- enhances fluxing properties of glass
- depresses freeze point of water, allowing cold temperature applications

In the past, the use of a hydrated potassium carbonate (16% water) was preferred to the more deliquescent anhydrous form. Technology has since spurred improvements in several areas, thereby fostering the acceptance of the anhydrous granular form of potassium carbonate. These improvements include the development of other PotCarb processes and enhancements in the areas of packaging, dry bulk handling and storage facilities.

One commonly used method produces a calcined PotCarb that requires several additional processing steps after its liquid phase reaction between caustic potash and carbon dioxide. The final step being a heat treatment where the temperature is raised sufficiently to drive off the water of crystallization. Two different ion exchange methods each employ a multi-step process that requires several raw materials and also yields several by-products. A more practical approach for supporting the use of anhydrous potassium carbonate is the development of the fluidized bed reactor. This process allows the direct, one-step manufacturing of an anhydrous material that needs no further refinement. Typically there is much greater customer demand for dry material, however, since PotCarb it is readily soluble, aqueous solutions do not present a challenge. Armand Products is well positioned to meet the demands of today's customers with its four state-of-the-art fluidized bed reactors and its liquid PotCarb capabilities.

II Markets and Uses of Potassium Carbonate

The potassium carbonate market is divided between the glass industry and other numerous applications. Product is shipped throughout the United States and into international regions.

Potassium carbonate has a wide variety of uses including potassium bicarbonate, video glass, specialty glass, ceramics, agrochemicals, fertilizers, potassium silicate, food, detergents and cleaners, photographic chemicals, gas purification, animal feed, rubber additives, polymer catalysts, fire suppression, cement, and textiles.

A more complete summary of the various uses of potassium carbonate is given in the following table:

POTASSIUM CARBONATE APPLICATIONS

GLASS	CHEMICALS	DYES PIGMENS	FOOD	CLEANERS	GAS PURIFICATION	OTHER
Source of K_2O for alkali barium, lead or strontium silica glasses used in the production of television tubes, illuminating ware, tubing, laboratory glass, optical glass, tableware, and giftware. Used alone or with Na_2CO_3. **CERAMICS** Used especially in titanium dioxide frits for appliance industry	Inorganic chemicals Potassium salts of phosphates, silicates, persulphates, permanganates, and potassium cyanide antioxidants Oxalic acid Dehydrating agents Corrosion inhibitor Fertilizers Catalysts Rocket fuels Gums Adhesives Terephthalic acid	Inks Dry Colors Dyeing Textiles Printing Fabrics	Chocolate ”alkalyzing” or ”Dutch” processing of cocoa powder Effervescent mineral water Special leavening agents Brewing beer Raisin drying Alfalfa drying Cattle feed additive Ramen noodles	Washing Bleaching Boiler compounds Liquid soaps Metal cleaners	Removal of carbon dioxide and other acid gases by absorption in a solution of potassium carbonate. With the following materials: 1. natural gas 2. synthesis 3. hydrogen 4. synthetic natural gas 5. petrochemical products 6. dry ice 7. chemicals from hydrocarbon gases 8. Molten carbonates for removal of sulfur dioxide from flue gases	Tanning leather Perfume and toilet articles Refrigeration Fire Extinguishers Photography Flameproofing Electroplating Molten salts Fireproof coolant as in anti-freeze for exposed steel columns Rubber additives

III. Manufacturing Process

Armand Products' Potassium Carbonate is manufactured in a fluidized bed reactor at its production facility in Muscle Shoals, AL. This results in a product that is anhydrous, making it unnecessary to perform any further processing to eliminate hydrated water (calcining). Armand Products' Potassium Carbonate (PotCarb) is a white, dense, free-flowing granular material which is easy to handle and store.

The process starts with potassium chloride, obtained from the Canadian province of Saskatchewan. Through an electrolytic conversion of the KCl salt, potassium hydroxide (caustic potash, KOH), chlorine (Cl_2) and hydrogen (H_2) are produced. The hydrogen is a fuel source while the chlorine has numerous important and varied applications. Liquid caustic potash and carbon dioxide are the only raw materials required for producing PotCarb.

The dry potassium carbonate can easily be dissolved in water to form a liquid solution. Typically a 47% solution is recommended as this capitalizes on the highest concentration with the lowest freezing point (3°F). This minimizes handling problems during colder weather.

The chemical equation for this process is simply:

$$2\,KCl + 2\,H_2O \rightarrow 2\,KOH + H_2 + Cl_2$$

$$2\,KOH + CO_2 \rightarrow K_2CO_3 + H_2O$$

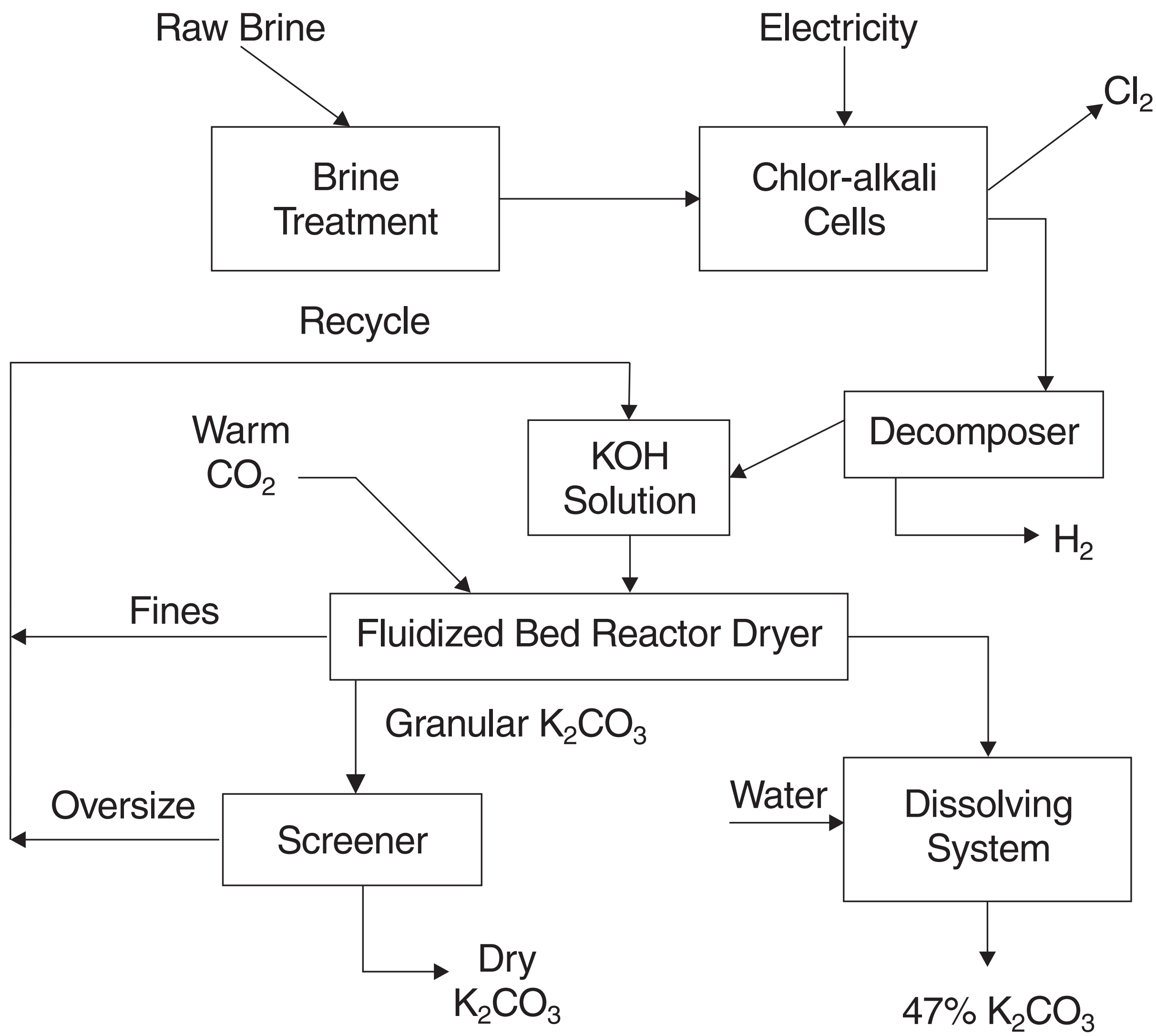
The favorable logistics of this production facility cover a wide spectrum of advantages:

- Largest domestic facility, utilizing four reactors in two plants;
- Dry and liquid forms available in various packaging units;
- Vertically integrated with on-site production of liquid caustic potash (KOH), the key raw material;
- Geographical location in proximity to Gulf and Eastern coast ports, allowing for timely export shipments;
- Electric power for raw material conversion and plant operations is readily available from a nearby TVA plant.

As a side note, potassium carbonate cannot be made by the Solvay process used for sodium carbonate (Na_2CO_3).

The flow diagram for the manufacturing process is shown on the next page:

Flow Chart of Manufacturing Process



IV. Grades of Potassium Carbonate

Armand Products produces potassium carbonate in granular and liquid form to satisfy the requirements of its customers. Each material conforms to the high standards of chemical purity and physical properties essential for our extensive and varied customer base. Armand Products' anhydrous potassium carbonate is white, dustless, dense, free-flowing granular product. Several grades of dry PotCarb that differ in their typical granulation ranges are available. In addition, a water solution in the form of a 47% solution is typically provided.

Requests can be made through the Armand Products' Technical Service staff for the following literature:

Sales Specification
product grade sheets listing parameters and their limits

Technical Information
data sheets covering chemical and physical properties

The chemical parameters included in the standard sales specifications for Anhydrous Potassium Carbonate are:

K_2CO_3	KOH
KCl	H_2O
Na	K_2SO_4
As	Fe
Hg	Ni
Heavy Metals (as Pb)	

Physical Properties:
(not specification items)

Particle Size
Typically material is between 18 and 80 mesh for granular and through 325 mesh for ground material.

Bulk Density
75 - 84 lb/ft^3 (granular, varies by grade); 37 lb/ft^3 (extra fine)

Melting Point
891°C

Solubility in Water
112 grams in 100 grams water at 20°C

Appearance
White, granular, free-flowing

V. Materials of Construction

Iron, steel, stainless steel, rubber lined steel or phenolic lined steel is recommended.
If heated to 120°F, the potential for iron contamination exists.
Polyethylene drums can be used for liquid storage and handling at ambient temperatures.
Aluminum, zinc, brass, bronze, and copper are NOT recommended due to the potential for a reaction.

Experience with the various polymeric materials of construction is limited. Additional data based on customers' own experiences should be considered. The weight of the PotCarb must be factored into the structural constraints of polymeric materials. The following information is therefore offered as a starting point:

- Polyethylene and propylene can be used if not exposed to temperatures beyond that recommended for each polymer. Possible deformation of polymer surface may occur. Caution is advised during processes that generate heat, e.g., solubilization of dry potassium carbonate.
- Polyester is NOT recommended.
- Polyvinyl chloride (PVC) has been reported to be acceptable but caution is advised for possible embrittlement. Regular inspection and proper usage procedures may allow good service from this material.

A. ANHYDROUS POTASSIUM CARBONATE

Piping

Pneumatic systems require 4 inch minimum piping with four foot radius curves. Gravity feed systems should be 6 inch diameter or larger.

Valves

Butterfly or slide gate valves are typical, however, the butterfly type are preferred for humid conditions.

Blowers

Pneumatic unloading requires blowers that are generally 500 - 600 CFM and operate at 10 lbs. of pressure. In humid conditions, the transfer air should be pulled through a desiccant bed to provide dry transfer air. The dryer should be checked periodically and replaced as needed.

Storage Silos

Capacity should hold at least 1.5 times the size of a normal shipment. A dry air purge in wet humid conditions will ease the handling of product. A 45° slope to the silo bottom is recommended. A bag house is recommended for filtering out dust from the transfer air during the pneumatic unloading of product. Storage silos should be steel, lined steel or stainless. A more thorough discussion of the recommendations for bulk storage can be found in section VIII.D.

B. LIQUID POTASSIUM CARBONATE

Pipelines

A two inch minimum pipeline with a 3 inch minimum on the suction side of the pump is recommended. Long sections of outdoor piping should be heat traced and insulated. Experience has demonstrated that flanged connections with alkali-resistant gaskets will minimized the potential for leaks. All piping should be installed with a slight slope to ensure complete drainage. Loops and pockets are to be avoided.

Pumps

Centrifugal and rotary types with all iron construction may be used. A deep packing gland is desired to prevent leakage at the pump shaft. Graphite/asbestos packing material is recommended for potassium carbonate solution service.

Valves

Globe, angle, gate, and plug valves may be employed to control flow rates and for line shutoffs. Valve construction of cast iron, steel, and stainless steel (Type 316 and 304) is recommended.

Storage Tanks

Fabrication specifications require at least 3/8 inch wall thickness for units larger than 10,000 gallon capacity and a 1/4 inch wall for smaller capacities. The withdrawal pipe connection should be a few inches above the bottom of the tank. To facilitate tank cleaning, a drain connection should be installed at the lowest point in the tank.

Rubber or phenolic based epoxies can be used for lining steel storage tanks where prevention of iron contamination is critical. However, rubber does not withstand high temperatures.

Storage tanks in cold climates should be insulated with 2 inches of polyurethane insulation to maintain pumpable conditions and should have an internal or external heat source to make temperature adjustments.

Some plastics are also acceptable for small tank construction (generally less than 10,000 gallons). Polypropylene, polyethylene, PVC, CPVC and FRP can be used. Polypropylene is the most commonly used plastic for storage tanks.

Further discussion with our Technical Service staff on the appropriate materials of construction and equipment for handling potassium carbonate is encouraged.

VI. Shipping Packages

SHIPMENT OF POTASSIUM CARBONATE

Potassium carbonate can be shipped in various types and sizes of packaging. Armand Products' Technical Service is available to discuss all possible options and can assist the customer in determining the most advantageous method to receive this product. Examples of the typical containers available are:

- 50 and 100 pound multiwall paper bags
- 400 pound fiber drums
- 2000 pound bulk bags
- hopper and pneumatic trucks
- hopper and pneumatic railcars

Factors that determine the type of package include the quantity to be used, as well as the customer's location, unloading system and storage facilities. Since bulk shipments can be made in trucks and railcars, it is better to determine the most advantageous delivery method through an economic survey for each individual case.

BAG AND DRUM SHIPMENT

The multiwall kraft bags used by Armand Products are constructed with a polyethylene moisture barrier to better protect product quality during storage. Fiber drums can be furnished with a polyethylene liner upon request. Bag and drum specifications are available from Armand Products' Technical Service department.

Although receipt of the material in bags or drums may not offer the economic advantage of bulk, there are other factors which may prompt the choice of this type of packaging. The use of bags or drums simplifies distribution when potassium carbonate is used in small quantities at several locations. Individual packages also eliminate the necessity of batch weighing potassium carbonate for various requirements. No expensive or elaborate equipment is needed to unload or handle bagged or drummed potassium carbonate shipments. The use of drums and bags makes potassium carbonate available to even the smallest industrial consumer.

Trucks and rail cars for this service are checked internally for the presence of moisture, previously loaded material, and general surface deterioration.

BULK BAG SHIPMENT

This larger form of individual packaging offers a unique advantage for those that meet the space requirements but do not have the capability to unload and store truck or rail car shipments. Typically each bag is filled with 2,000 pounds of potassium carbonate. For truckload quantities however, different weights can be filled to meet the customer's batch requirements. These bulk bags are made of a woven polypropylene fabric with a polyethylene liner as a moisture barrier. This is considered a one trip package to ensure the safe handling integrity of the bag and to maintain product quality. Bulk bags filled with a metric ton (2205 pounds) of product have become the preferred package for export shipments.

BULK SHIPMENT

Bulk quantities of potassium carbonate may be shipped in covered hopper cars, pneumatic unloading cars, hopper trucks, or self-unloading pneumatic trucks. The choice depends on the quantity used, as well as the customer's location, unloading system and storage facilities. Details of the various options can be discussed with Technical Service personnel.

Covered hopper cars are specially designed to handle products such as potassium carbonate and provide the most satisfactory method of bulk shipment. These self-discharging cars are equipped with weatherproof hatches and three or four bottom outlet gates which provide protection from outside contamination. The bottom outlets are normally of the Enterprise or sliding gate type, measuring 13 x 24 or 13 x 42 inches, with a clearance of 6 to 9 inches over the rail that will vary with car design. Hopper cars, normally available in 70 and 95 ton capacities, can be readily adapted to mechanical unloading systems.

Pressure differential (PD) railroad cars are available to customers who have an appropriate pneumatic system in place to handle unloading. Those who have a potential interest in receiving PD bulk railcar shipments are invited to discuss this option with Armand Products' Technical Service.

Motor truck shipments have been found practical for bulk potassium carbonate, particularly where the distance is not excessive and the customer's consumption or location will not allow rail deliveries. The amount of material that can be carried is usually restricted by the road limitations of the states through which the haul is made, with a typical maximum weight of 45,000 pounds. Truck designs can vary but hopper bottom discharge or self-unloading pneumatic trucks are typical.

The development of self-unloading trailers now enables consumers of as little as 100 tons per year to receive potassium carbonate in bulk without the necessity of providing costly unloading equipment. The tank-type trailer available for this service carries a self-contained unloading system which blows the material into the customer's bin. (Figure 1, Page 15) It is operated by the driver, who makes the complete delivery. The receiving equipment required by the customer is relatively simple in structure and is easy to install. It consists of a vertical transmission line made with a four inch standard pipe and four foot radius bends with a tank vent through a filter system to remove dust.

Since shipments of potassium carbonate will be 20-23 tons by this service, a minimum storage capacity of 30-35 tons will usually be adequate for most plants. Conventional silos, tanks or existing storage facilities can easily be adapted for this system. The facility must be moisture-tight and requires a storage tank vent dryer.

Anhydrous and liquid forms of

VII. Precautions in Handling Potassium Carbonate

potassium carbonate are recognized as OSHA hazardous substances. This chemical material is alkaline and may cause severe irritation by all routes of exposure.

Before starting to work with PotCarb, the user should be aware of its properties, understand what safety precautions to follow, and know how to act in case of contact. Some general guidelines are listed below.

- Read and understand the current MSDS for complete and updated information.
- Locate and periodically check eyewash fountains and safety showers in all areas where potassium carbonate is handled.
- Seek medical attention immediately after first-aid measures are applied.

ROUTES OF EXPOSURE

PotCarb can be irritating to the skin and to a severe degree if in contact with the eyes. Tissue destruction may follow if not properly treated. Inhalation of airborne concentrations of dust, mist or spray may cause damage to the upper respiratory tract and even to the lung tissue which could result in chemical pneumonia, depending upon the severity of exposure. Ingestion may cause irritation to the mucous membranes of the mouth, throat, esophagus and stomach, with severity dependant on the quantity involved.

There are no known chronic effects associated with potassium carbonate. However, acute consequences may occur, depending on the length of exposure and whether proper treatment is rendered in a timely fashion.

OVERVIEW OF FIRST AID MEASURES

Immediately administer the first aid measure described below, then seek professional medical attention.

Eyes - Flush eyes with large amounts of water for at least 15 minutes, holding lids apart to ensure flushing of the entire surface. Washing eyes within several seconds is essential to achieve maximum effectiveness.

Skin - Wash contaminated areas with plenty of water. Remove contaminated clothing and footwear. Do not remove goggles if the eyes are not affected. Wash clothing before reuse. Discard footwear which is contaminated on the inner surface.

Inhalation - Remove to fresh air. If breathing is difficult have a trained person administer oxygen. If respiration stops give mouth-to-mouth resuscitation.

Digestion - Never give anything by mouth to an unconscious person. If swallowed, do not induce vomiting. Give large quantities of water or several glasses of milk if available. If vomiting occurs spontaneously, keep airway clear.

PROTECTIVE EQUIPMENT

OSHA requires employers to supply protective equipment for employees. When handling potassium carbonate, the following protective equipment is recommended:

- Wear suitable goggles for eye protection during the handling of potassium carbonate in any form. The goggles should be close-fitting to prevent the entry of liquids, yet provide adequate ventilation to prevent fogging.
- Wear gloves coated with rubber, synthetic elastomers, PVC or other plastics to protect the hands while handling potassium carbonate. Gloves should be long enough to cover well above the wrist.
- Potassium carbonate causes leather to disintegrate. For this reason, wear rubber boots. Wear the bottoms of trouser legs outside the boots. Do NOT tuck in.
- Wear cotton clothing for some protection of the body. Wear rubber aprons or a rain suit for additional protection.
- Wear shirts or jackets with long sleeves and with the collar tightly fastened.
- Wear hard hats to aid in the protection of the head, face and neck.
- Wear NIOSH-approved respirators for protection from dusts and mists.

Additionally, care should be taken to avoid the simultaneous presence of potassium carbonate and lime dust. This chemical combination when in contact with moisture in the form of water or perspiration will cause the formation of a very irritating and corrosive material, namely caustic potash (KOH). Workmen must carefully wash and remove dust from one of these chemicals before working in an area where the other chemical is being handled.

VIII. Handling of Anhydrous Potassium Carbonate

A. BAG AND DRUM HANDLING

An efficient system for unloading bagged or drummed potassium carbonate depends on the volume to be handled and the distance it must be carried. Lightweight two-wheel hand trucks, with a wide lip on which bags are laid horizontally, are commonly used where a small volume is involved. Larger tonnage may justify roller conveyors, either freeturning or powered, or portable belt conveyors. Power lift trucks and pallets may be used to store bags or drums and distribute them to points of usage.

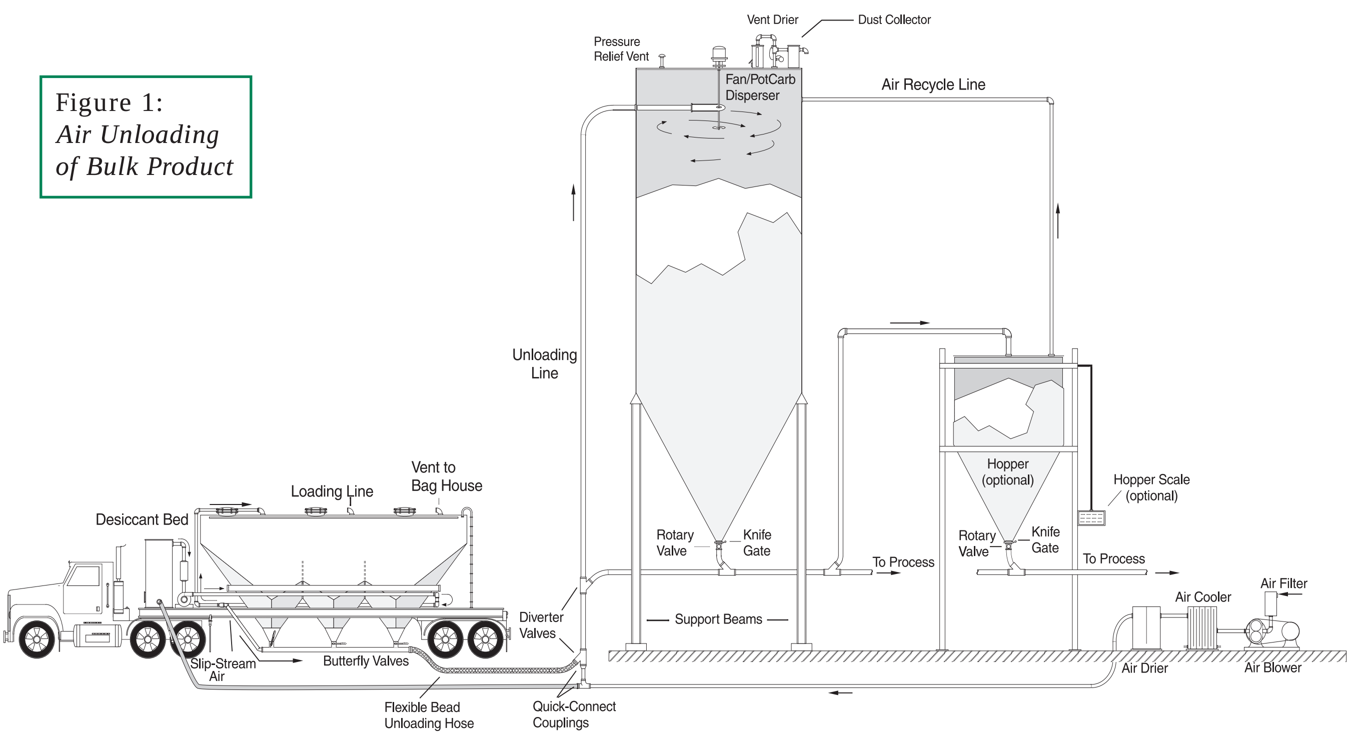
Bags should be piled flat to avoid package distortion and load insecurity. Since potassium carbonate is hygroscopic, the best possible storage conditions should be provided. Damp floors, leaky roofs, and moist storage conditions in general must be avoided. Moisture absorption will cause lumping of potassium carbonate and consequently a reduction in the alkalinity. To minimize this condition, the quantity of material stored should be limited and the oldest material should be used first. For drum users, a good in-plant practice is to keep the cover secured except when withdrawing material. This minimizes moisture pick-up by the hygroscopic potassium carbonate.

BULK MATERIAL HANDLING

Granular anhydrous potassium carbonate can readily be transferred in a conventional conveying system or a pneumatic conveying system. In a conventional system, atmospheric conditions have only a negligible effect on moisture pickup during product handling. However, the anhydrous material must be shielded from rain and snow. It is recommended that the product be transferred in one continuous operation and used before caking occurs.

In pneumatic conveying (see Figure 1), potassium carbonate

Figure 1:
Air Unloading of Bulk Product



may be transferred from truck or rail car with ambient air in dry climates (low dew points). In humid (high dew point) areas like the Mississippi Valley and coastal regions, dry air for pneumatic transfer is suggested. For in-plant pneumatic transfer of anhydrous potassium carbonate, it is always advisable to use clean, dry air.

In preparing for receipt of anhydrous potassium carbonate by bulk hopper car, provisions should also be made to receive emergency shipments by bulk truck. In addition, it is strongly recommended that a roof be provided over the bulk transfer area to provide protection from the weather.

Armand Products' Technical Service is available to assist the customer in selecting the most advantageous method to receive potassium carbonate and to supply information on equipment in the material handling field.

B. MECHANICAL SYSTEMS

This type of transfer equipment consists of a belt or screw conveyor in combination with a bucket elevator or a drag flight chain conveyor. A schematic diagram of this equipment is given in Figure 2. A permanent undertrack unloading system is recommended when bulk rail receipts are at least 800-1,000 tons per year. A portable above track conveying unit is recommended where bulk material receipt is only 400-500 tons per year.

The screw conveyor is versatile, relatively inexpensive, simple to maintain and readily made dust-tight. It can convey horizontally, vertically, or on an incline and can be fitted with multiple inlet and discharge openings. Screw conveyors are not usually used for long-distance movement and are limited in length to 50-100 feet. The grinding action of a screw conveyor on friable materials

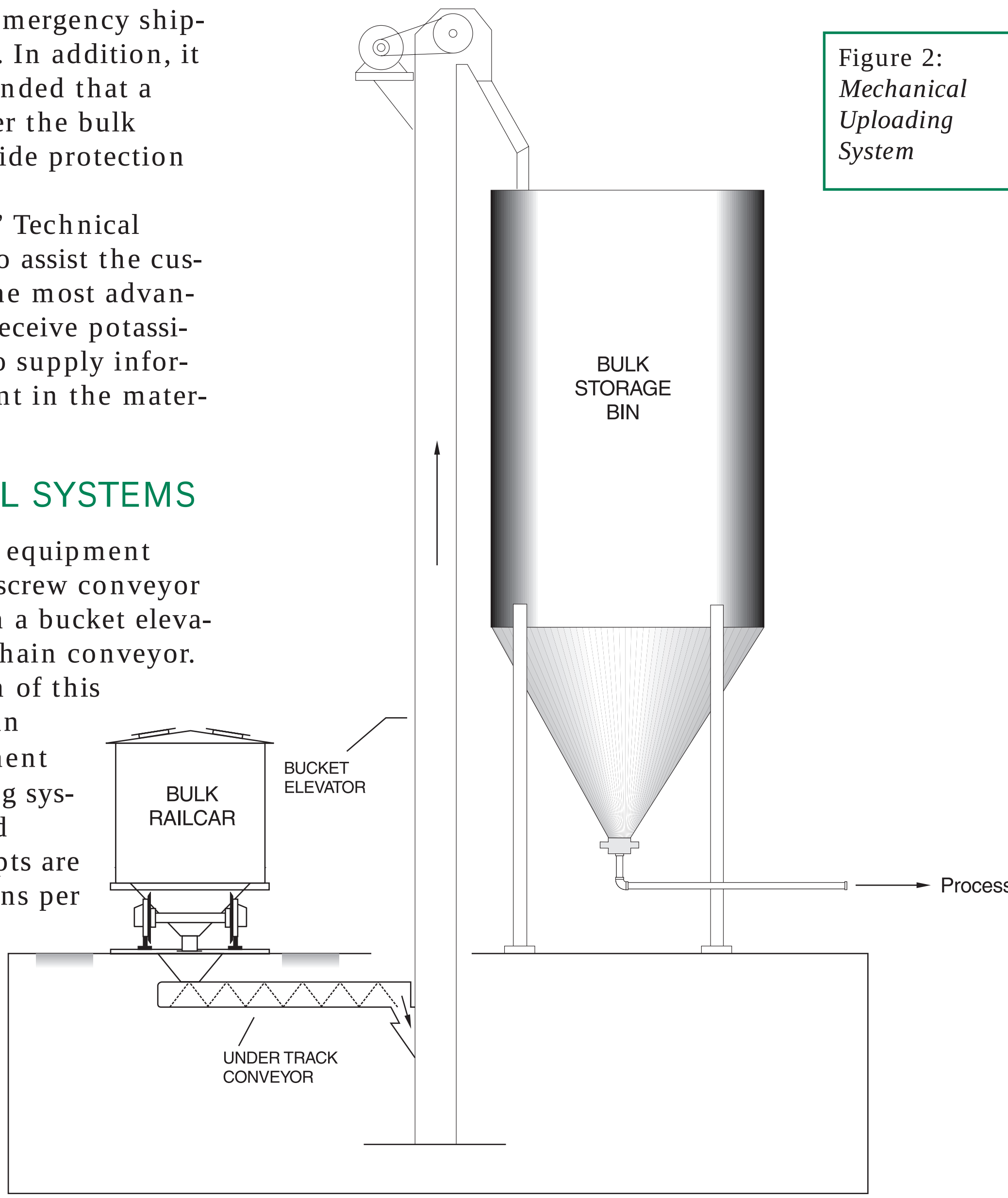


Figure 2: *Mechanical Uploading System*

increases particle degradation. In many cases where particle size is important for granular potassium carbonate, the use of a screw conveyor should be avoided if possible.

Lubricants would contaminate the potassium carbonate due to the location of bearings in the screw conveyor. Therefore, either white iron or Stellite bearings is recommended. The outboard bearing drive end of the conveyor should be located outside the trough to decrease maintenance and increase bearing and drive life. Discussion with an equipment manufacturer regarding the capacity, size and r.p.m. of screw conveyors would facilitate better potassium carbonate service.

The belt conveyor is recommended primarily where large tonnages are to be conveyed long distances. The initial expense and horsepower requirements of belt conveyors are quite low. Particle degradation is kept to a minimum where belt conveyors are used. They can be used to convey up, down or on an incline at a maximum operating angle of 10° for potassium carbonate. All bearings and idlers on a belt conveyor for potassium carbonate should be antifriction and dustproof. Discussion with an equipment manufacturer regarding the capacity, size and r.p.m. of screw conveyors would facilitate better potassium carbonate service.

Although dusting and contamination may become excessive at loading and discharge points, the use of special housings will overcome this problem. Where wind or moisture presents a serious problem, the increased cost of weather-tight housing may increase the total investment above that required for other types of conveyors.

A bucket elevator consists of an endless rubber belt or chain to which are attached buckets for elevating material vertically or along steep inclines. It has a receiving hopper or boot and provisions for discharging the load. For potassium carbonate service, the rubber belt type is preferred to the chain type thereby avoiding metallic contamination. Loading should be on the vertical leg rather than forcing the bucket to scoop the material from the boot. The added cost of a deeper elevator pit required for vertical loading will be offset by a much smoother and continuous operation.

There are three general types of bucket elevator discharges: centrifugal, continuous and positive. The centrifugal elevator discharges material by centrifugal force, operating at high speeds to assure that the buckets empty completely and to prevent the "backlogging" or recycling of material. Some particle degradation of granular potassium carbonate occurrs due to the centrifugal force used to fling the material into the discharge chute.

The continuous elevator is designed for slow speed operation and ordinarily should not exceed 125 feet per minute. Its buckets are mounted continuously so that material is discharged from one bucket over the front of the preceding one which acts as a moving chute or guide to the fixed discharge spout. The continuous elevator has a higher initial cost when compared to a centrifugal elevator. However, its slower speed causes less wear on buckets, belts and wheels, translating into a lower maintenance cost.

Although the continuous elevator operates at slower speed than the centrifugal, it has about the same capacity because of the greater number of buckets. The continuous elevators are especially well adapted where degradation of material is to be minimized and where extreme dust conditions are to be avoided.

The positive discharge bucket elevator is also designed for slow speeds of approximately 120 feet per minute. The interval spaced buckets are completely inverted by snubbing the chains after they have passed over the head pulley, giving them an opportunity for complete discharge. Since the positive discharge elevator is a low-capacity unit for the cost involved, it does not replace the centrifugal or continuous elevators in normal granular potassium carbonate service.

The continuous flow conveyor is a unit which carries materials en masse through a dustproof and weathertight duct, completely filling its cross section. In general, the conveyor medium is an endless chain upon which are mounted flights at suitable intervals or a chain with the flight cast as an integral part of the link. The flights can be either solid or open finger type. The conveyor can operate on a horizontal, vertical or incline plane and in any combination thereof. Speed is usually limited to about 80 feet per minute, although slower speeds are preferred. Because the conveyor is completely enclosed and lubrication points can be located outside the unit, the material conveyed is not subject to external contaminations. The conveyor is not completely self-cleaning, so where more than one material is handled, contamination could become a problem. When handling deliquescent materials like potassium carbonate, special care is required to remove any residual material between product transfers.

Another type of en masse conveyor is known as the “zipper”. It consists of a flat, endless rubber belt with two attached side belts designed to fold into a semi-circular enclosure. The outer edges of the side belts are made with rubber teeth which interlock when the conveyor is closed. It is dust-tight and will handle granular potassium carbonate without degradation.

C. PNEUMATIC CONVEYING

The pneumatic conveyor differs altogether from other machines in that it depends on a high velocity air stream to transport materials. If the velocity is too low, the material will drag and build up, particularly on long horizontal runs and in elbows. The pneumatic conveyor is higher in capital investment and power cost than a mechanical conveying system. Some product degradation can be expected, however, the extent of particle breakage is nominal for Armand Products’ dense granular potassium carbonate when the system is properly operated. Advantages that help offset its high cost include dust suppression, flexibility, low handling losses and self-cleaning. Dry air must be used in the pneumatic transfer of potassium carbonate when moisture pickup is detrimental and also where the humidity factor is high.

There are three types of pneumatic conveying systems: vacuum, pressure and combination vacuum-pressure.

With a proven record of efficient performance for low cost unloading of bulk cars, the vacuum system is especially effective in picking up material from many points and delivering to one remotely located process or storage area. Hook-up of a bulk rail car and startup of the system is quickly and easily done.

The vacuum type pneumatic unloading system requires little accessory equipment. The system consists of a transport pipe attached to an adapter mounted on the Enterprise gate of the hopper car. Approximately 20 feet of flexible pipe should be available to make the proper connection to the car.

The transport pipe delivers potassium carbonate to a receiver above the storage bin where the air and potassium carbonate are separated. The air is exhausted to the atmosphere through a dust filter or preferably a bag house while the potassium carbonate is discharged through an airlock into storage. A blower on the exhaust side of the separator or dust collector provides vacuum in the system. The suction system is capable of transporting potassium carbonate economically for distances up to 500 feet.

The pressure system (Figure 3) is an economical, compact system, for transferring product from one area to several points, frequently at considerable distances. This system is extremely flexible and can be adapted to many specific needs including unloading, in-plant transfer and recirculation.

Pressure type pneumatic systems usually require additional equipment at the inlet end of the transport pipe. Generally, a blower or fan is used to furnish compressed air which is forced into the line through an injection nozzle. In a modified version of the system, potassium carbonate is fed by screw conveyor into an air stream which carries the potassium carbonate to storage. Another pressure system utilizes a fluidization chamber, where a predetermined volume of potassium carbonate is dropped into the vessel, fluidized under pressure and blown through the line. This unloading method is an intermittent and cyclic operation.

The combination vacuum-pressure (Figure 4) is considerably more sophisticated. Combining features of both types described above, this system expands the range of usage and is capable of meeting multiple needs. Stationary or portable systems can be operated under push-button control. Railcars can be unloaded simultaneously from various sidings with simultaneous delivery to many points, without contamination and at a high volume throughput.

Potassium carbonate can be transported a considerable distance by this combination system. This also eliminates the inlet equipment necessary for a straight pressure system. Although the unloading distance can be extended over that of a suction system, the power requirements are likely to be excessively large for the longer application.

Figure 3: *Pressure System*

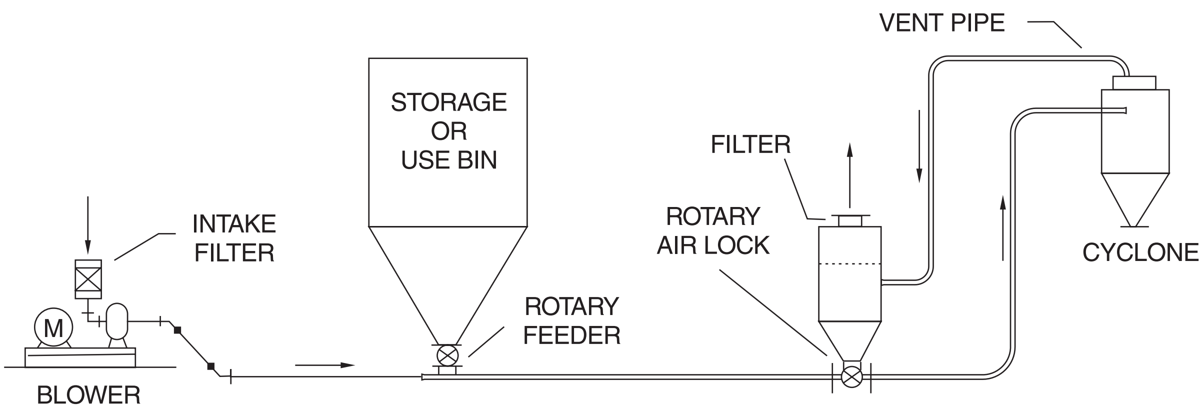
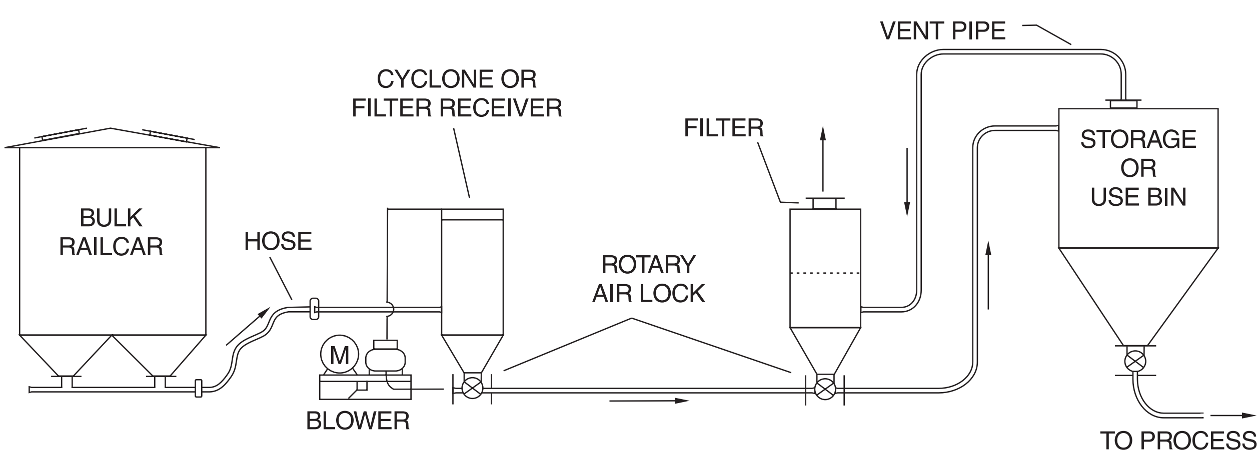


Figure 4: *Combination Vacuum-Pressure System*



D. BULK STORAGE

Storage facilities for potassium carbonate can be constructed of concrete or steel. A moisture seal is required to eliminate penetration of water which both contaminates the product and causes it to lump. Storage capacity should be at least 1.5 to 2 times the amount of potassium carbonate received in a single shipment. This safety factor insures an adequate supply of potassium carbonate at all times and allows for unforeseen transportation delays. Storage bin capacity should be calculated on a basis of 82 lbs./cu. ft. for Armand Products' granular potassium carbonate. Bins may be cylindrical with cone bottom or rectangular with V-slanted bottoms. The slopes of the bin bottoms should be approximately 45 degrees.

Prefabricated storage bins offer the most economical and versatile choice for bulk storage of potassium carbonate. These bins are readily available in a variety of sizes and shapes with galvanized, black or stainless steels as the common materials of construction. The standard vertical types are cylindrical with the bottom designed for either center or side draw-off. They are easily assembled in the field on their own supports with the assistance of the vendor or may be assembled directly by the customer. They must be made weatherproof to protect the potassium carbonate from moisture.

Concrete silos are of three types: stave, monolithic or poured, and block or tile. The stave type is lowest in cost and is formed of precast concrete staves, set up in stepped tiers and bonded with outside turnbuckle rods. The interior walls are plastered for a smooth surface, and the exterior is brush-coated with waterproof cement. Cone bottoms are easily adapted to this type of silo. Monolithic silos are of poured concrete with circumferential reinforcing steel. Concrete block and hollow tile silos are more expensive than either the stave or monolithic. Block silos are formed with chambered precast concrete blocks, and hollow tile silos are of precast chambered tile with reinforcing rods grouted into recesses between the tiers. Care should be exercised when choosing a silo of this last design.

A vent dryer is recommended for all dry bulk potassium carbonate storage facilities. A typical dryer for this application can be fabricated by the customer or purchased from a vendor.

IX. Handling of Liquid Potassium Carbonate

A. SHIPMENTS OF 47% POTASSIUM CARBONATE

Liquid 47% potassium carbonate is typically shipped in drums, tank truck or rail car. It is also available by consignment as a barge load, shipped from the Armand Products' plant in Muscle Shoals, AL. Each form of transportation has its own advantages. The type of service you select will depend upon such factors as size and location of storage, rate of consumption, plant location and freight rates. Armand Products' Technical Service staff is well qualified to survey your present facilities and recommend the economical form of transportation best suited to your particular requirements.

The unloading and handling of 47% liquid potassium carbonate is simple. The freight for transporting this solution is higher than for the granular, anhydrous form. It is necessary to have enough tank capacity to accommodate a 16,000 gallon rail shipment or a 4,000 gallon tank truck shipment in addition to whatever level of inventory the customer maintains.

B. UNLOADING LIQUID POTASSIUM CARBONATE FROM TANK CARS

Placement of Railcar for Unloading

1. DOT requires setting the handbrake and blocking the wheels after the car is properly spotted.
2. DOT regulations also state that caution signs must be placed on the track or car to give warning to persons and switching crews approaching the car from the open end(s) of the siding. Caution signs must be left up until the car is empty and disconnected from the unloading line. Signs must be made of metal or other suitable material, at least 12 x 15 inches in size, and bear the words, "Stop - Tank Car Connected" or "Stop - Men At Work".
3. Place derail attachments at the open end(s) of the siding, approximately one car length (50 ft.) away.

Unloading Precautions

1. Entrust only responsible and well-supervised employees with the unloading of liquid potassium carbonate. It is recommended that a worker be present during the entire time that a car is being unloaded.
2. Provide workers with chemical splash goggles, hard hats, and rubber or rubber coated gloves to protect against eye and skin exposure. A safety shower and eyewash fountain must be located in the unloading area.
3. Unload only in daytime or when adequate lighting is available. Caution workers to exercise care.
4. Before starting to unload, make certain that the tank car and storage tank are vented and verify that the storage tank has sufficient capacity for the delivery.
5. Do not allow entry into the car under any circumstances.
6. If the tank car needs to be moved when partially unloaded, DOT regulations require disconnecting all unloading lines and replacing all car closures.

7 A suggested method for sampling is to withdraw intermittent samples from a 1/2 inch sample line fitted with a valve and 1/4 inch nipple which is connected to a vertical portion of the unloading line.

8 Armand Products' liquid potassium carbonate is shipped in well insulated and specially lined tank cars. Linings in these liquid tank cars will withstand temperatures up to 225°F. To prevent damage to the linings, steam should not be added directly into the tank cars under any circumstances.

9 If compressed air (20 psig max.) is used in the unloading operations, inspect all fittings for leaks or other defects before unloading. Dome fittings should be inspected carefully. If leaks are found, suspend unloading operations until they are fixed.

Handling in Cold Weather

Under normal weather conditions, 47% liquid potassium carbonate will remain fluid. Since Armand Products' liquid PotCarb is loaded hot into well insulated tank cars, this product should arrive at the destination in a liquid state. Under severe winter conditions, 47% potassium carbonate will begin to crystallize below 8°F. Although frozen material has not presented a problem in the past, the Technical Service staff is available for assistance should such a situation occur.

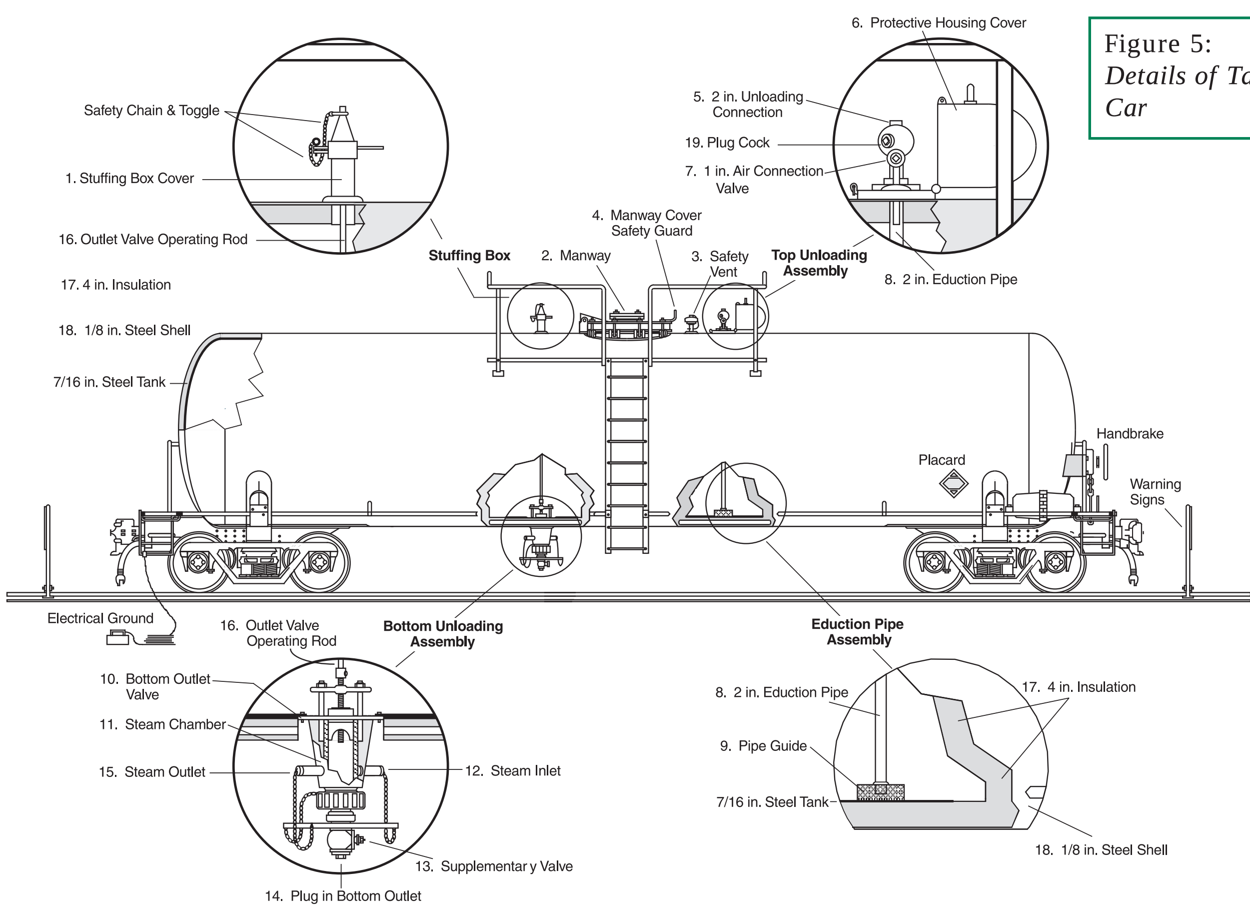


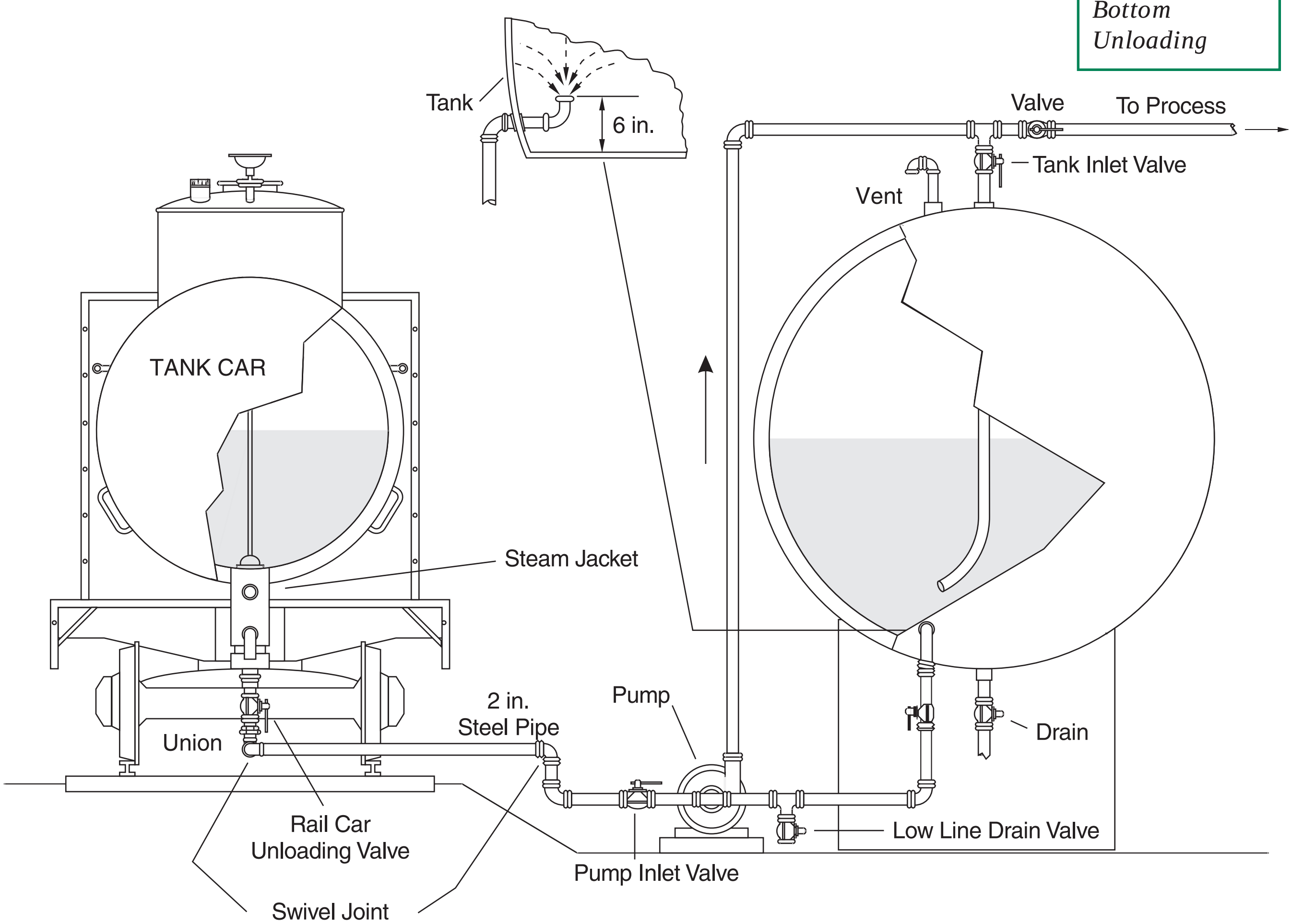
Figure 5: *Details of Tank Car*

Unloading through Bottom Outlet Valve (Figures 5 and 6)

Liquid potassium carbonate is usually unloaded through the bottom outlet valve. Most cars are also equipped with eduction pipes that allow for unloading through the dome if desired. Both methods are described in this handbook.

1. Open the dome cover and ascertain whether the contents of the car are liquid. Keep the dome cover at least partially open during the entire unloading operation to vent the tank car. This prevents a vacuum from being created in the car.
2. Ensure that the bottom outlet valve (10) Figure 5 is closed tightly. The valve rod (16) which operates the bottom outlet valve has a wheel on it which is located either under the dome or just outside the dome of the car. When located outside of the dome, the wheel is reversed and serves as a cap during transit.
3. Remove the pipe plug (14) and then carefully open supplementary valve (13) in order to drain out any liquid into a bucket that may have seeped by the bottom outlet valve (10) during transit. If the supplementary valve cannot be opened, apply steam from a steam lance on the valve's exterior to free it for opening.

Figure 6: *Bottom Unloading*



4 Attach the unloading line to the outlet side of supplementary valve.

5 Check the unloading line to see that all valves are in the proper position for unloading.

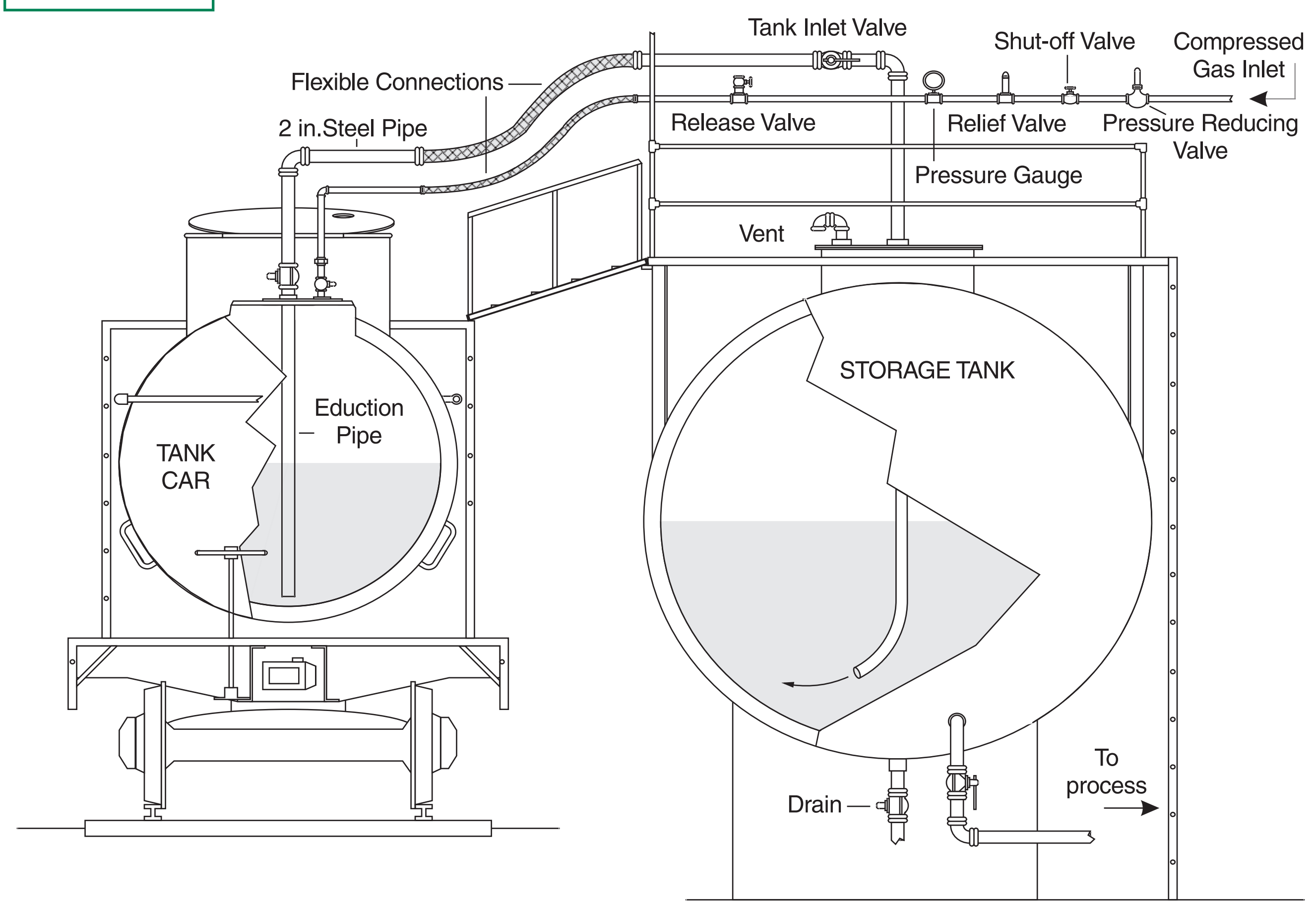
6 Open the bottom outlet valve by turning valve rod (16) to allow contents to flow by gravity to pump or tank.

7 Compressed air can be used to speed up the flow or to transfer the liquid to the storage tank without the use of a pump. Check that the rupture disk is intact. The pipe plug on the air connection valve (7) must be removed and a flexible air line connected for this purpose. This line should be equipped with a release valve, oil trap, pressure relief valve set at 20 psig, pressure reducing valve set at 18 psig and a shutoff valve. When using compressed air to assist in the transfer of product, the dome cover should be securely closed before the application of air pressure.

8 When the car and unloading line are empty, shut off the air supply and open the release valve if air pressure has been used in unloading.

9 Detach the unloading line at the car after the following conditions are met: tank car is empty and at atmospheric pressure; discharge pipe has completely drained; and if used, air line is disconnected. Prepare car for returning according to procedure under “Preparing Empty Cars For Return.”

Figure 7: *Top Unloading*



Top Unloading with Air Pressure (Figures 5 and 7)

1 Open the dome cover and ascertain that the contents of the car are liquid. Check that the rupture disk is intact.
2 Close the dome cover and fasten securely, making certain that it is airtight.
3 Check storage tank to see that it has sufficient capacity and is well vented. Removal of cover on top of storage tank is advisable for venting.
4 Connect the unloading line to a 2-inch unloading connection (5) on the eduction pipe, after removing cover (5). A flexible steel hose connection for unloading is recommended since the car may rise of as much as two inches during unloading.
5 Connect the flexible air supply line to the 1-inch air inlet valve (7, Figure 6). This line should be equipped with a release valve, oil trap, pressure relief valve set at 20 psig, pressure reducing valve set at 18 psig and a shutoff valve.
6 Apply air pressure slowly until there is a normal flow of liquid to the storage tank, after which the pressure should be adjusted and maintained until the tank car is completely empty. The sound of air rushing through the unloading line and a drop in air pressure indicates that the tank car is empty.
7 When the unloading line is empty, shut off air supply, open the release valve and allow the discharge pipe to drain well.
8 After proper draining and with the tank car at atmospheric pressure, disconnect the air supply line at the railcar.
9 Open the manway cover and determine whether the car is empty, however, do not enter the railcar to make an inspection. If the car is empty, only then should the unloading line be disconnected. Replace manway and valve covers tightly.
10 Take care not to spill any product on the car as it will endanger trainmen handling the empty car on its return and may cause damage to the car.
11 Prepare car for return as highlighted in the following section.

Preparing Empty Tank Car for Return

1 Close bottom outlet valve and supplementary valve.
2 Disconnect the unloading line and replace the bottom outlet plug. Do not replace closures on steam openings.
3 Close the dome cover and fasten securely.
4 Return the empty tank car promptly in accordance with the shipper’s instructions. Follow the shipper’s routing directions in all instances.

C. UNLOADING LIQUID POTASSIUM CARBONATE IN TANK TRUCKS

The transportation of 47% liquid potassium carbonate is accomplished in 4,000-gallon tank trucks meeting DOT regulations. The consignee should determine if sufficient tank capacity is available to accept the shipment. Any special instructions concerning the delivery must be conveyed by the consignee to the truck driver before permission is given to unload. Each truck driver is completely familiar with the equipment used and under normal unloading conditions does not require manpower assistance from the consignee. However, it is always a good idea to have a consignee operator near the unloading area. Customers are asked to report any problems that result from the carrier failing to follow proper unloading procedures as well as any personal protective gear or specified safety requirements.

Tank trucks containing liquid potassium carbonate are commonly unloaded by one of three methods:

Gravity

Flow of material direct to storage or to an unloading pump furnished by the consignee.

Truck Mounted Pump

Tank truck equipment is available which has an all iron or nickel pump mounted on the tank truck. The pump is driven by a tractor powered take-off or an auxiliary gasoline engine. At least a 2-inch pump line should be used. If this type of unloading is desired, arrangements should be made with your Armand Products Company sales representative at the time of your original order.

Compressed Air

Unloading by air pressure is another option where the consignee can provide the air supply system. Equipment on the air supply line to the truck includes: pressure reducing valve (PRV), pressure gauge, pressure relief valve (set 2-3 psi above PRV setting) and a pressure release valve. All associated equipment should be properly maintained and periodically tested. An air hose of appropriate length to reach the truck dome is required if the customer's air supply is used.

Another option is to request that the carrier come equipped with a self-contained air compressor system mounted on the truck. If this is the desired unloading mechanism, arrangements should be made through your Armand Products' salesperson at the time of your original order.

Tank Truck Unloading Facilities

1. An eye wash and safety shower should be located nearby and periodically tested. If these are not available then clean, potable water flowing through a hose must be readily accessible.
2. A storage tank of 6,000 gallons capacity is suggested to provide adequate capacity for typical tank truck shipments and a sufficient reserve supply between shipments. Figure 8 provides general information on both horizontal and vertical tanks for storage of truck

shipments. Steam coils are not required for these tanks except in those areas where the temperature will drop below 10°F for extended periods of time. In fringe areas, insulation of the tank should suffice.

3 A permanent 2-inch unloading line from a convenient truck unloading area to the top of the storage tank should be installed. Connection between the truck and the permanent unloading line is furnished through the use of an alkali-resistant heavy duty rubber hose carried on the truck. The distance should be measured to determine the appropriate length required on the truck.

4 The permanent unloading line should be equipped with a 2-inch male pipe fitting (kamlock type) to facilitate connecting the rubber hose from the tank truck. Cap the end of the permanent unloading line when not in use.

5 A ¾ inch valve connection is recommended on the unloading line at the truck end for use in flushing out the line with air, water or steam. It can also be used as a drain.

Unloading Procedure

1 Purge out the eye wash and shower to remove rust that may have accumulated.

2 Connect one end of the unloading hose to the customer's storage tank fill pipeline.

3 Depending on the severity of the cold weather, the fill line, the unloading hose or truck outlet may need to be preheated with steam.

4 Check the unloading line to be sure that it is open.

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

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

7 Open the valves on the truck discharge line.

8 Monitor in person the entire unloading process until the truck is empty.

9 If compressed air is used, allow the air to flush out the lines to the storage tank and then cut off the air supply.

10 When a pump is used, it is advisable to flush out the unloading line before disconnecting the hose. If water is available, a small quantity can be added to the truck while the pump is running to flush out the line. Air or water can be used to flush out the line into the storage tank. If no water is available or incomplete flushing is suspected, great caution should be exercised when disconnecting lines.

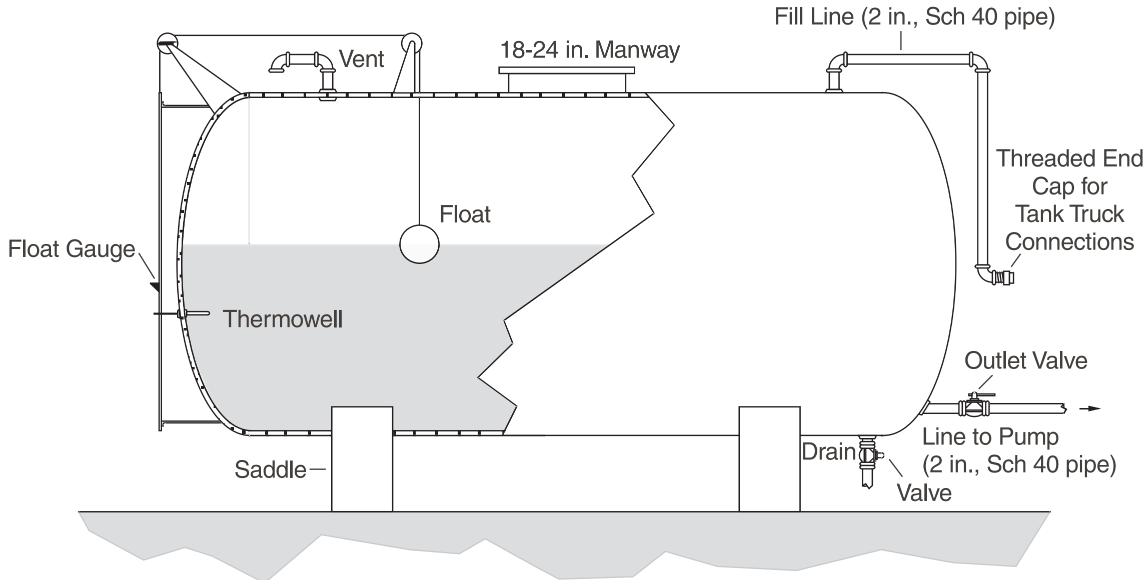
11 Close the valve on the storage tank fill line.

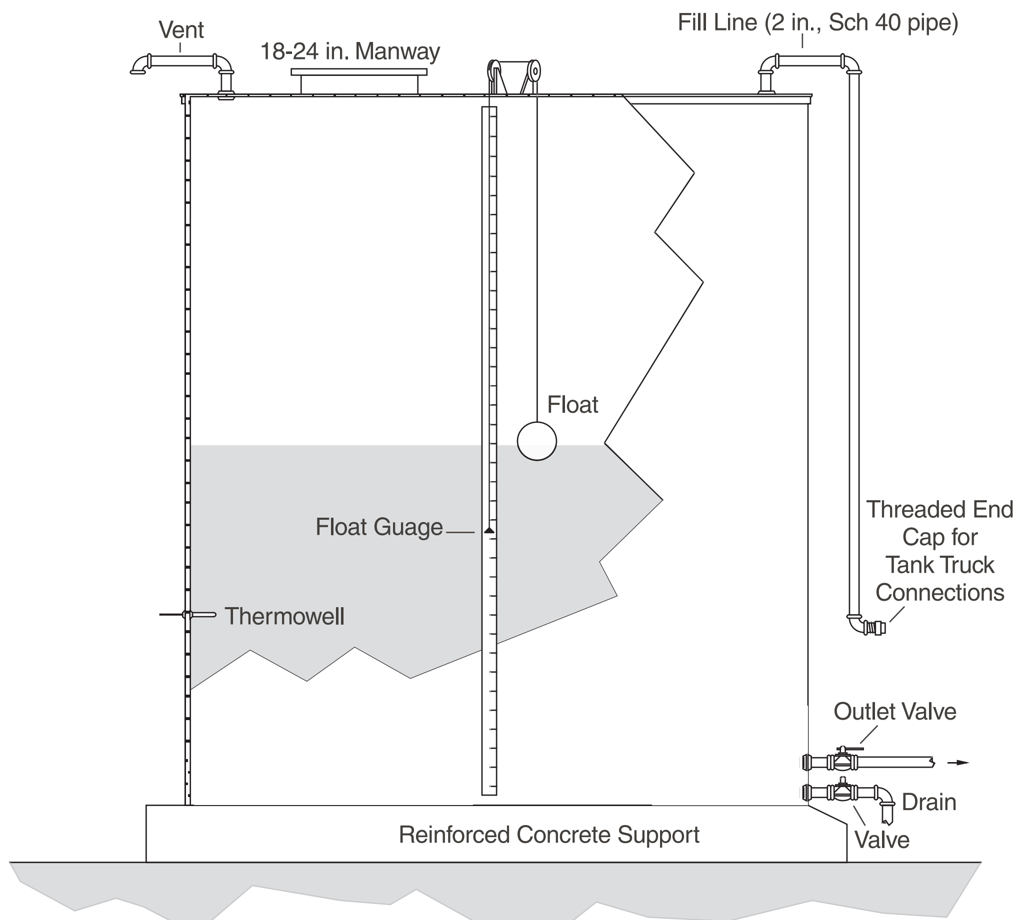
12 Close all valves on the tank truck.

13 If the customer's fill line is fitted with a drain, this should be disconnected and any residue discharged into a proper container.

14 Unload potassium carbonate with adequate safeguards for spill control. Proper containment and disposal of released product should be handled in accordance with federal, state and local regulations.

Figure 8:
Installation of Tanks for Liquid Product





D. DILUTION OF POTASSIUM CARBONATE SOLUTION

The references to various tables and figures will provide important data for the dilution of an aqueous solution of potassium carbonate:

Table 4, page 36 - weight of potassium carbonate solutions at 60°F; used in calculating and estimating capacities of process equipment.
Table 5, page 37 - density of a potassium carbonate solution as a function of temperature and concentration.
Graph 1, page 40 - dilution chart for 47% potassium carbonate with water at 60°F.
Graph 2, page 41 - dilution chart for various concentrations of potassium carbonate with water at 60°F.
Graph 3, page 42 - gross weight in pounds per gallon of potassium carbonate solution including its density at 60°F.

Dilution Calculations

The matter of diluting liquid potassium carbonate to a given concentration is frequently confusing. Dilutions can be simplified to the following formula:

A x (B - C) / C = D
A = Specific Gravity of Strong Solution
B = % K_2CO_3 in Strong Solution
C = % K_2CO_3 in Weak Solution
D = Volume of water to add to each volume of strong solution.

Example 1

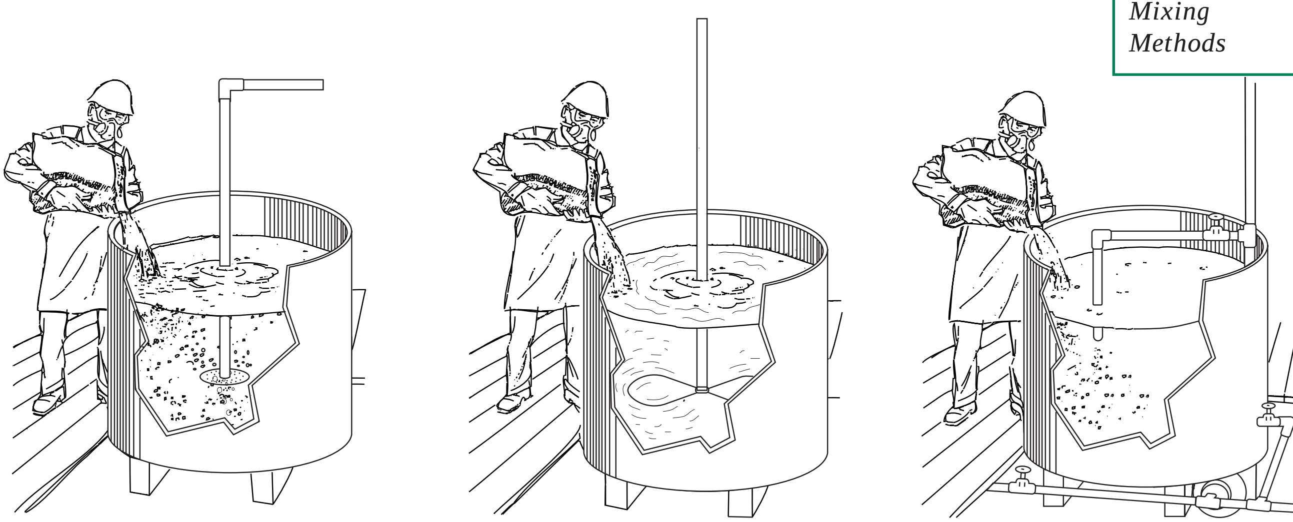
Dilution of 47% liquid potassium carbonate to 15% potassium carbonate solution:

Specific Gravity of 47% liquid at 60°F = 1.496 (from Table 4)

Thus, [1.496 x (0.47 - 0.15)] / 0.15 = 3.19 volumes of water to add to each volume of 47% liquid potassium carbonate.

It should be noted that volumes of potassium carbonate solutions and water are not additive. When one gallon of water and one gallon of potassium carbonate solution are mixed, the resultant yield is somewhat less than two gallons. This fact is quite significant when dealing with large volumes.

Figure 9: *Alternative Mixing Methods*



Example 2

(using Graph 2)

To make a 100 gallon solution containing 2.5 pounds K_2CO_3 per gallon, two solutions containing 1.5 and 6.0 pounds of K_2CO_3 per gallon are used. The volume of each solution is determined by drawing the line “AB” connecting 1.5 lbs./gal. with the 6.0 lbs./gal. This intersects 2.5 lbs./gal. at point “C”. Reading down to the “Volume of Water or Weak Liquor” we find 77 gallons (1.5 lbs./gal.). Next, reading up to the “Volume of Strong Liquor Used” we find 23 gallons (6.0 lbs./gal.).

DISSOLVING ANHYDROUS POTASSIUM CARBONATE

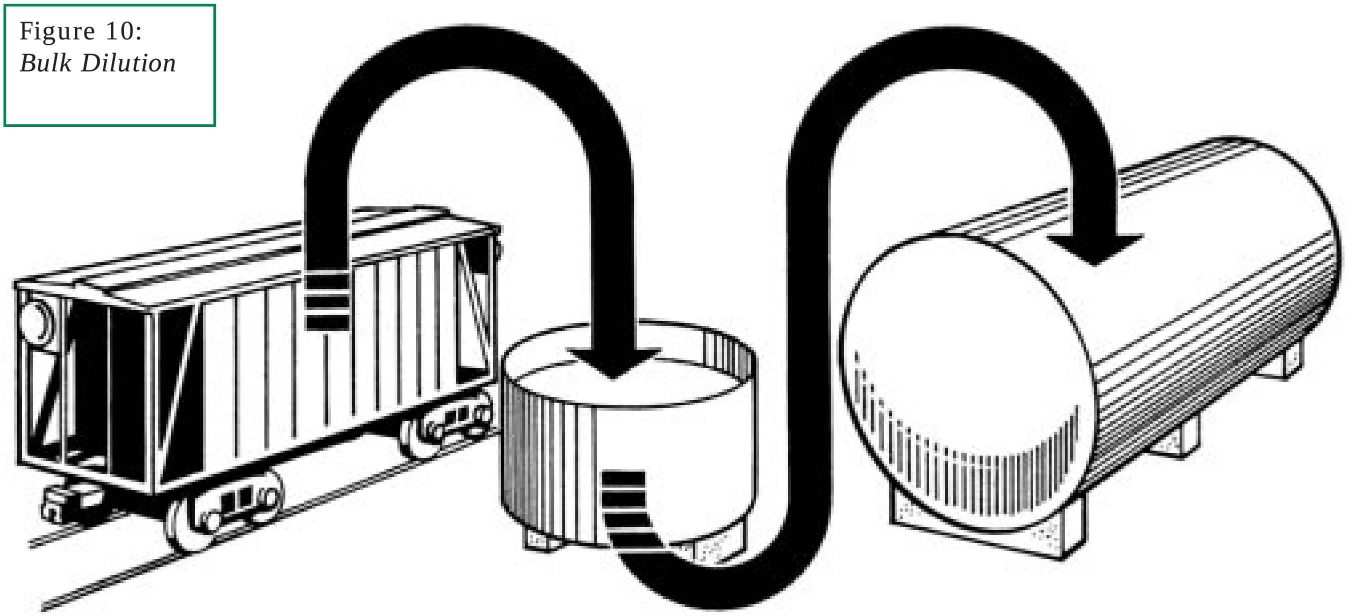
Anhydrous potassium carbonate is readily soluble in water. When large quantities of granular potassium carbonate are placed in quiescent solutions, the granular material falls to the bottom and forms a layer of hydrate. This layer dissolves quite slowly, forming an area of heavy concentration. There also is local overheating which may cause an attack on the tank lining or coating.

Good practice calls for slowly adding the granular material to a well agitated solution of water (see Figure 9). Agitation is best supplied by a propeller type agitator, or under some conditions, by using a circulating pump.

SOLUTION STORAGE WITH DRY BULK DELIVERY

A user of potassium carbonate in solution form may want to explore another possibility. Bulk trucks of anhydrous material can allow the dissolution of the PotCarb in water as it is transferred to the storage tank (see Figure 10). With pneumatic truck delivery, anhydrous potassium carbonate is discharged into a slurrifier located on top of the storage tank. The slurrifier is a pipe held in vertical position with dry material added at the top and

Figure 10: *Bulk Dilution*



solution added tangentially to the pipe with the resultant slurry discharged from the bottom of the pipe into the top of the storage tank. Dissolving of the potassium carbonate is completed in the storage tank. A vent on the storage tank is required to let the anhydrous PotCarb transport air escape. Armand Products' Technical Service is available for discussion should the customer consider such a system.

The slurrifier system works best with a hot solution (140-190°F). This helps to insure that the potassium carbonate dissolves rapidly and minimizes any problem of solids build-up in the tank bottom. The dissolving rate is approximately doubled when the temperature of solution is raised from 60°F to 150°F A considerable amount of heat is evolved in the charging process due to the heat of solution. For a 30% solution, a temperature rise of 35°F will take place.

X. Technical Data

PROPERTIES OF POTASSIUM CARBONATE

TABLE 1:

CHEMICAL AND PHYSICAL PROPERTIES OF POTASSIUM CARBONATE

Chemical Name:	Potassium Carbonate	
Chemical Formula:	K_2CO_3	
Molecular Weight:	138.20	
Acid Equivalent:	1 lb. K_2CO_3 = 0.528 lb. HCI	
Alkali Equivalent:	100% K_2CO_3 = 68.16% K_2O	
Electrical Conductivityof Molten K_2CO_3		
	1.95 ohm^{1} cm^{1} at 1652°F (900°C)	
	2.12 ohm^{1} cm^{1} at 1742°F (950°C)	
	2.26 ohm^{1} cm^{1} at 1832°F (1000°C)	
Dielectric Constant:	4.96 at 18° and 166.6 Kcs/sec.	
Heat of Fusion:	56.4 cal./g. or 101.5 BTU/lb.	
Heat of Formation:	K_2CO_3	1982 cal./g. at 25°C
		3568 BTU/lb. at 77°F
	$K_2CO_3 \cdot 0.5\ H_2O$	
		1429 cal./g. at 25°C
		2572 BTU/lb. at 77°C
	$K_2CO_3 \cdot 1.5\ H_2O$	
		1715 cal./g. at 25°C
		3087 BTU/lb. at 77°F
Heat of Solution:	K_2CO_3	47.6 cal./g. (Evolved)
		85.7 BTU/lb. (Evolved)
	$K_2CO_3 \cdot 0.5\ H_2O$	
		28.9 cal./g. (Evolved)
		52.0 BTU/lb. (Evolved)
	$K_2CO_3 \cdot 1.5\ H_2O$	
		2.6 cal./g. (Absorbed)
		4.7 BTU/lb. (Absorbed)
Infrared Absorption Band:		6.9 µ and 11.4 µ
Melting Point:	1636°F (891°C)	
pH of 47% K_2CO_3:	12.63	
Specific Gravity:	2.428 at 66°F (19°C)	
Specific Heat:	0.216 BTU/lb.-°F over range 73-210°F	
	0.216 cal./g.-°C over range 2-99°C	
Solubility in Water:		
— at 68°F	112 grams per 100 grams	
— at 212°F	156 grams per 100 grams	
Transitions:	Second order at 410°C and 465°C	

TABLE 2:

DISSOCIATION PRESSURE OF POTASSIUM CARBONATE IN MILLIMETERS MERCURY

Reaction: $K_2CO_3 \rightarrow K_2O + CO_2$

Temperature (°C)	D.P. (mm Hg)
900	0
950	1.2
970	1.68
1000	2.1
1100	7.4
1200	10.3
1300	15.1
1400	35.6

TABLE 3:

SOLUBILITY OF POTASSIUM CARBONATE IN SELECTED ORGANIC SOLVENTS

Solvent	Category	Solubility (ppm)	
		Anhydrous K_2CO_3	Solution**
Kerosene	Hydrocarbons	ND*	ND*
Carbon Tetrachloride	Chlorinated Hydrocarbons	ND*	ND*
O-Dichlorobenzene	Chlorinated Hydrocarbons	0.2	0.2
Trichloroethylene	Chlorinated Hydrocarbons	0.1	< 0.1
Perchloroethylene	Chlorinated Hydrocarbons	ND*	< 0.1
Acetone	Ketones	1.3	17
Methyl Ethyl Ketone	Ketones	3.7	7.2
Ethyl Acetate	Esters	1.2	0.6
Butyl Acetate	Esters	25	5.4
Methyl Cellosolve Acetate	Esters	45	6.7
Diethanolamine	Amine	29	27
Methanol	Alcohols	16,500	16,440
Ethanol	Alcohols	904	234
Isopropanol	Alcohols	4.0	21
Ethylene Glycol	Glycols	15,300	46,100

* ND = None Detected   ** Solution: 25% H_2O + 75% K_2CO_2 Dry Basis

TABLE 4:

DENSITY OF POTASSIUM CARBONATE SOLUTIONS AT 60°F (15.6°C)

% K_2CO_3	% K_2O	Specific Gravity 60/60°F	Degrees Baume Am. Std.	Degrees Twaddell	K_2CO_2 Grams per Liter	K_2CO_2 Pounds per Gallon	Total Weight of Solution in Pounds per Gallon	K_2CO_2 Pounds per Cu. Ft.	Total Weight of Solution in Pounds per Cu. Ft.
0	0.0	0.999	—	—	0.0	0.0	8.328	0.0	62.30
1	0.68	1.001	0.15	0.2	10.0	0.084	8.353	0.62	62.49
2	1.36	1.011	1.58	2.2	20.6	0.167	8.437	1.26	63.12
3	2.04	1.021	2.99	4.2	30.6	0.256	8.520	1.91	63.74
4	2.73	1.031	4.36	6.2	41.2	0.344	8.604	2.57	64.37
5	3.41	1.041	5.72	8.2	52.0	0.434	8.687	3.25	64.99
6	4.09	1.051	7.04	10.2	63.0	0.526	8.771	3.94	65.61
7	4.77	1.060	8.21	12.0	74.2	0.619	8.846	4.63	66.18
8	5.45	1.070	9.49	14.0	85.6	0.714	8.929	5.34	66.80
9	6.13	1.080	10.74	16.0	97.2	0.811	9.013	6.07	67.42
10	6.82	1.090	11.97	18.0	109.0	0.910	9.096	6.81	68.05
11	7.50	1.100	13.18	20.0	121.0	1.010	9.180	7.55	68.67
12	8.18	1.110	14.37	22.0	133.5	1.112	9.263	8.32	69.30
13	8.86	1.120	15.54	24.0	145.6	1.215	9.346	9.09	69.62
14	9.54	1.129	16.57	25.8	158.1	1.319	9.422	9.87	70.48
15	10.22	1.138	17.59	27.8	170.8	1.425	9.497	10.66	71.05
16	10.91	1.148	18.70	29.6	183.7	1.533	9.580	11.47	71.67
17	11.59	1.158	19.79	31.6	196.9	1.643	9.664	12.29	72.29
18	12.27	1.168	20.86	33.6	210.2	1.754	9.747	13.13	72.92
19	12.95	1.178	21.92	35.6	223.8	1.868	9.830	13.97	73.54
20	13.63	1.188	22.95	37.6	237.6	1.983	9.914	14.83	74.17
21	14.31	1.199	24.07	39.8	251.8	2.101	10.006	15.72	74.85
22	15.00	1.209	25.07	41.8	266.0	2.220	10.089	16.61	75.48
23	15.68	1.219	26.06	43.8	280.4	2.340	10.173	17.50	76.10
24	16.36	1.230	27.11	46.0	295.1	2.463	10.264	18.43	76.79
25	17.04	1.240	28.06	48.0	310.0	2.587	10.348	19.35	77.41
26	17.72	1.251	29.10	50.2	325.2	2.714	10.440	20.31	78.10
27	18.40	1.262	30.11	52.4	340.7	2.843	10.531	21.27	78.79
28	19.08	1.273	31.10	54.6	356.4	2.974	10.623	22.25	79.47
29	19.77	1.284	32.08	56.8	372.3	3.107	10.715	23.25	80.16
30	20.45	1.295	33.04	59.0	388.5	3.242	10.807	24.26	80.85
31	21.13	1.306	33.98	61.2	404.9	3.379	10.899	25.27	81.53
32	21.81	1.317	34.91	63.4	421.4	3.517	10.990	26.31	82.22
33	22.49	1.328	35.82	65.6	438.2	3.657	11.082	27.36	82.91
34	23.17	1.340	36.79	68.0	455.6	3.802	11.182	28.44	83.66
35	23.86	1.351	37.68	70.2	472.8	3.946	11.274	29.52	84.34
36	24.54	1.362	38.54	72.4	490.3	4.092	11.366	30.61	85.03
37	25.22	1.374	39.47	74.8	508.3	4.242	11.466	31.74	85.78
38	25.90	1.386	40.39	77.2	526.6	4.395	11.566	32.88	86.53
39	26.58	1.398	41.29	79.6	545.2	4.550	11.666	34.04	87.28
40	27.26	1.410	42.16	82.0	563.9	4.706	11.766	35.21	88.03
41	27.95	1.422	43.04	84.4	583.0	4.865	11.867	36.40	88.78
42	28.63	1.434	43.89	86.8	602.2	5.026	11.967	37.60	89.52
43	29.31	1.446	44.73	89.2	621.8	5.189	12.067	38.82	90.27
44	29.99	1.459	45.62	91.8	641.9	5.357	12.175	40.08	91.09
45	30.67	1.471	46.43	94.2	661.9	5.524	12.275	41.32	91.83
46	31.35	1.483	47.23	96.6	682.2	5.693	12.376	42.59	92.58
47	32.04	1.496	48.08	99.2	703.0	5.867	12.484	43.90	93.40
48	32.72	1.509	48.91	101.8	724.3	6.045	12.593	45.22	94.21
49	33.40	1.522	49.74	104.4	745.7	6.223	12.701	46.56	95.02
50	34.08	1.535	50.54	107.0	767.5	6.405	12.810	47.92	95.83
51	34.76	1.549	51.40	109.8	790.0	6.592	12.926	49.32	96.70
52	35.44	1.562	52.18	112.4	812.2	6.778	13.035	50.71	97.52

TECHNICAL DATA

TABLE 5:

SPECIFIC GRAVITY OF POTASSIUM CARBONATE SOLUTIONS AS A FUNCTION OF TEMPERATURE

TEMPERATURE (°F)

Conc.*	F.P.**	10	20	30	40	50	60	70	80	90	100
0	32	—	—	—	0.9999	0.9997	0.9990	0.9980	0.9966	0.9950	0.9931
5	29	—	—	1.057	1.046	1.044	1.042	1.040	1.038	1.035	1.033
10	25	—	—	1.096	1.094	1.092	1.090	1.088	1.086	1.083	1.081
15	21	—	1.146	1.145	1.143	1.141	1.139	1.137	1.135	1.132	1.130
20	12	—	1.199	1.197	1.194	1.192	1.190	1.188	1.185	1.182	1.179
25	3	1.252	1.251	1.248	1.245	1.244	1.242	1.240	1.237	1.234	1.231
30	-3	1.308	1.306	1.303	1.301	1.298	1.296	1.293	1.290	1.287	1.284
35	-16	1.366	1.363	1.360	1.358	1.355	1.352	1.350	1.347	1.344	1.341
40	-34.6	1.423	1.422	1.419	1.416	1.414	1.411	1.408	1.405	1.402	1.399
45	-1	1.486	1.484	1.481	1.479	1.476	1.473	1.470	1.467	1.463	1.460
47	8	1.511	1.509	1.506	1.503	1.500	1.498	1.495	1.492	1.489	1.486
50	18	—	1.547	1.545	1.542	1.540	1.537	1.534	1.531	1.528	1.525

Conc.*	110	120	130	140	150	160	170	180	190	200	210
0	0.9910	0.9986	0.9860	0.9832	0.9803	0.9771	0.9739	0.9704	0.9669	0.9631	0.9591
5	1.031	1.028	1.025	1.022	1.019	1.016	1.013	1.010	1.006	1.002	0.998
10	1.079	1.076	1.073	1.070	1.067	1.064	1.061	1.058	1.054	1.050	1.046
15	1.127	1.124	1.121	1.118	1.115	1.112	1.109	1.105	1.101	1.097	1.093
20	1.176	1.173	1.170	1.167	1.163	1.160	1.157	1.153	1.149	1.145	1.143
25	1.228	1.225	1.222	1.219	1.215	1.212	1.209	1.205	1.201	1.197	1.195
30	1.281	1.278	1.276	1.273	1.270	1.267	1.264	1.260	1.256	1.252	1.248
35	1.338	1.335	1.332	1.329	1.325	1.322	1.319	1.316	1.312	1.308	1.304
40	1.396	1.393	1.390	1.387	1.384	1.381	1.378	1.375	1.371	1.367	1.363
45	1.457	1.454	1.451	1.448	1.445	1.442	1.439	1.435	1.431	1.427	1.423
47	1.482	1.478	1.476	1.473	1.470	1.467	1.464	1.460	1.456	1.452	1.448
50	1.522	1.518	1.515	1.511	1.507	1.504	1.501	1.497	1.493	1.487	1.483
55	—	1.583	1.580	1.577	1.573	1.570	1.567	1.563	1.559	1.555	1.550

* Conc. = Concentration (% K_2CO_3) ** F.P. = Freezing point (°F)

TABLE 6:

ELECTRICAL CONDUCTIVITY OF AQUEOUS K_2CO_3 SOLUTIONS AT 15°C

K_2CO_3 (wt. %)	Conductance (mho/cm x 10^4)	Equivalent Conductance
5	561	74.2
10	1038	65.7
20	1806	52.4
30	2222	39.4
40	2168	26.5
50	1469	13.2

TECHNICAL DATA

TABLE 7:

HEAT CAPACITY OF POTASSIUM CARBONATE SOLUTIONS

K_2CO_3 (wt. %)	Cp* (cal./g.-°C)
5	0.94
10	0.89
15	0.84
20	0.80
25	0.75
30	0.72
35	0.68
40	0.66
45	0.63

* Good over range from 21-52°C

TABLE 8:

pH OF DILUTE POTASSIUM CARBONATE SOLUTIONS

Commercial K_2CO_3 (wt. %)	pH at 22°C
0.01	10.48
0.05	10.91
0.10	11.08
0.25	11.25
0.50	11.37
0.75	11.43
1.00	11.49
2.00	11.58
3.00	11.63
5.00	11.68
10.00	11.75

TABLE 9:

REFRACTIVE INDEX AND MOLAR REFRACTION OF POTASSIUM CARBONATE SOLUTIONS

K_2CO_3 (wt. %)	ND^{25}	RD*
0	1.3325	8.349
5	1.3415	8.357
10	1.3514	8.362
15	1.3624	8.370
20	1.3751	8.381

* Measured at Sodium D Wavelength (5893A°)

TABLE 10:

SURFACE TENSION OF POTASSIUM CARBONATE SOLUTIONS AT 20°C

K_2CO_3 (wt. %)	Dynes/cm
0	72.0
10	75.1
20	78.6
30	83.8
35	87.3
40	91.4
45	96.6
50	103.8

TECHNICAL DATA

TABLE 11:

VISCOSITY OF POTASSIUM CARBONATE SOLUTIONS AT 20°C

K_2CO_3 (wt. %)	Viscosity (centipoise)*
5	3.0
10	3.3
15	3.7
20	4.3
25	4.9
30	5.6
35	6.3
40	7.5
45	9.6
47	10.4
50	11.5

* Viscosities determined by Brookfield Method.

TABLE 12:

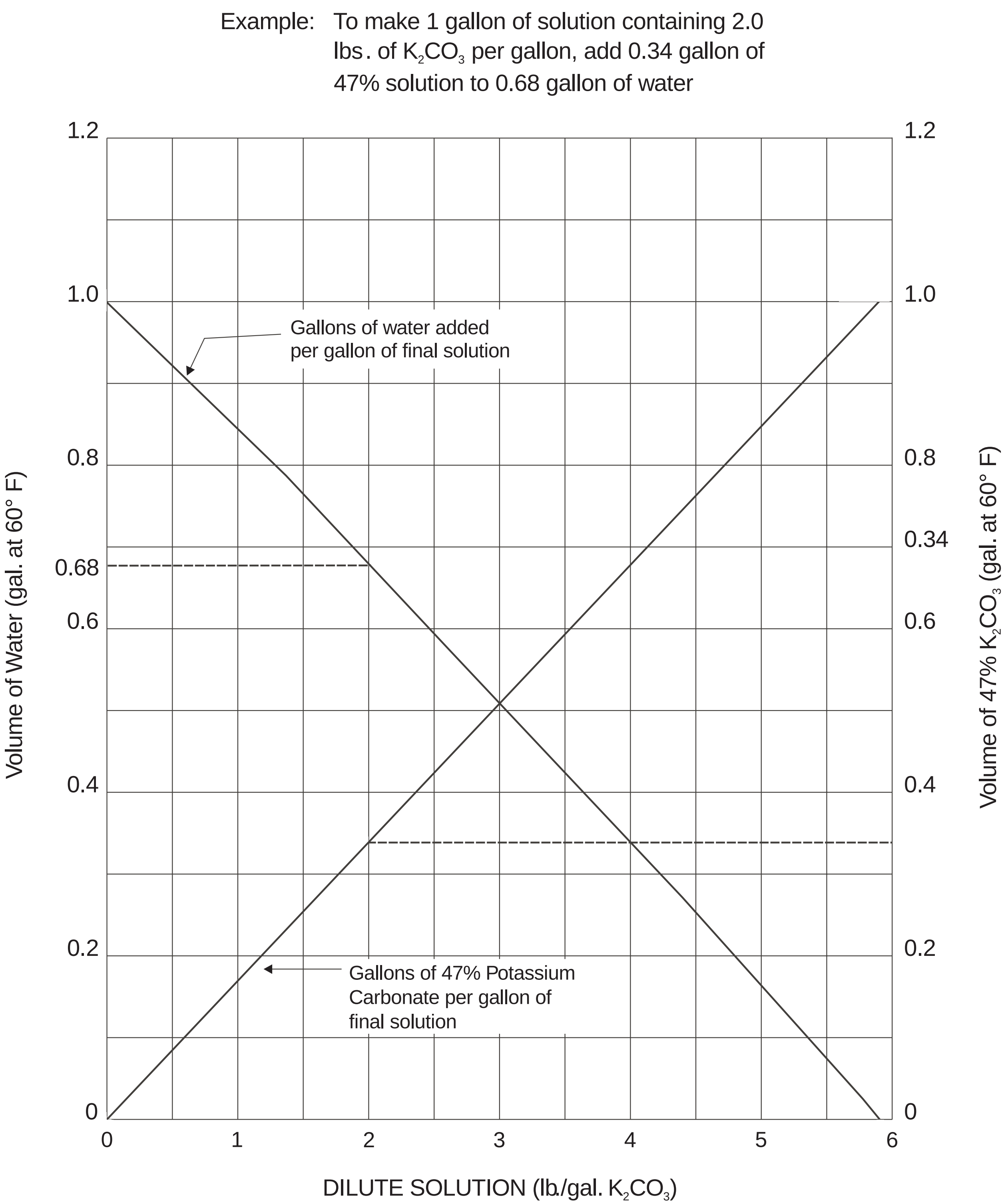
VAPOR PRESSURE, WATER AT 20.5°C

K_2CO_3 (wt. %)	Water Vapor Pressure*
0	18.1
19.6	16.8
46.2	11.8
54.7	8.6

* Pressure measured in millimeters of mercury, water at 20.5°C.

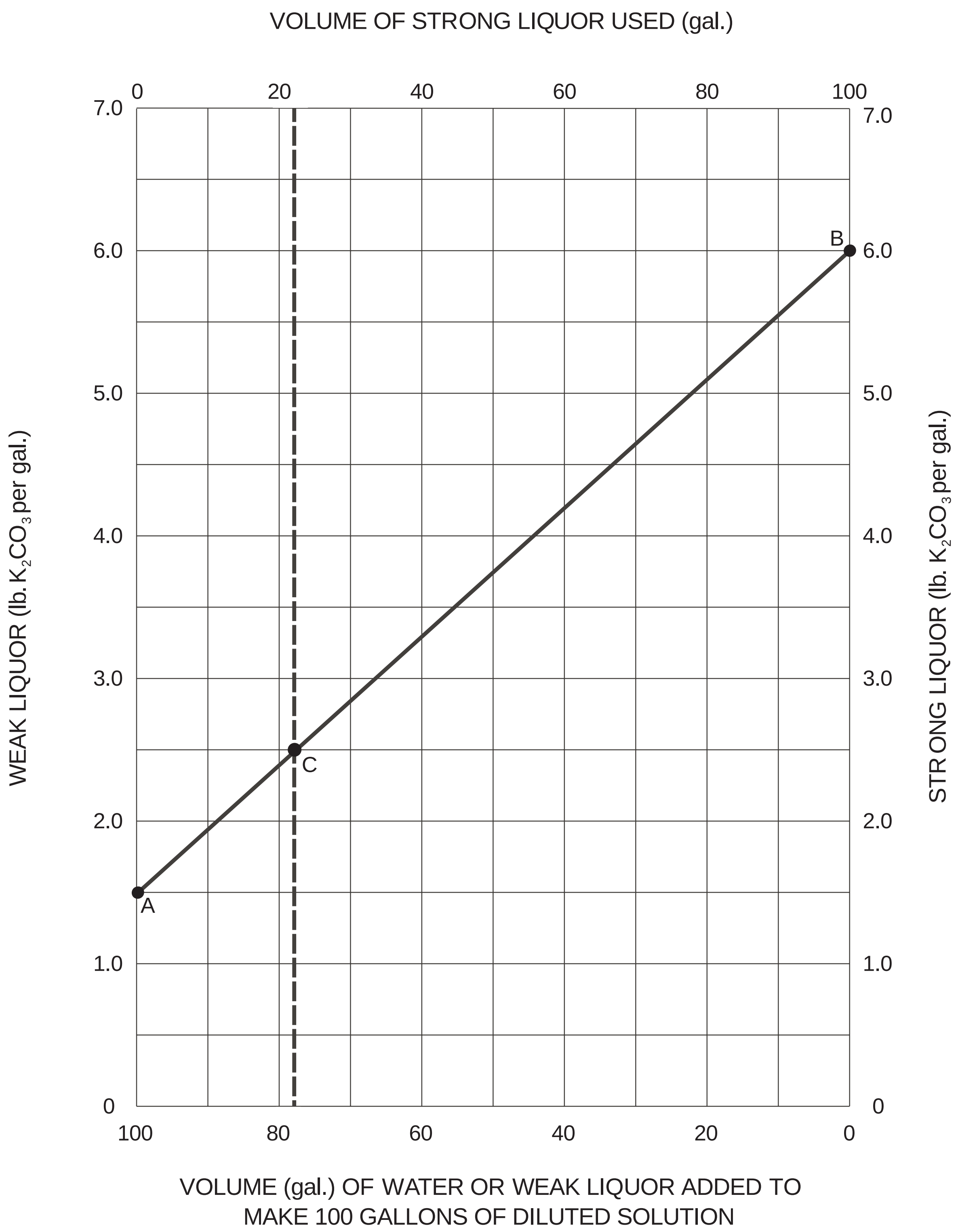
TECHNICAL DATA

Graph 1. Dilution of 47% Aqueous PotCarb at 60°F



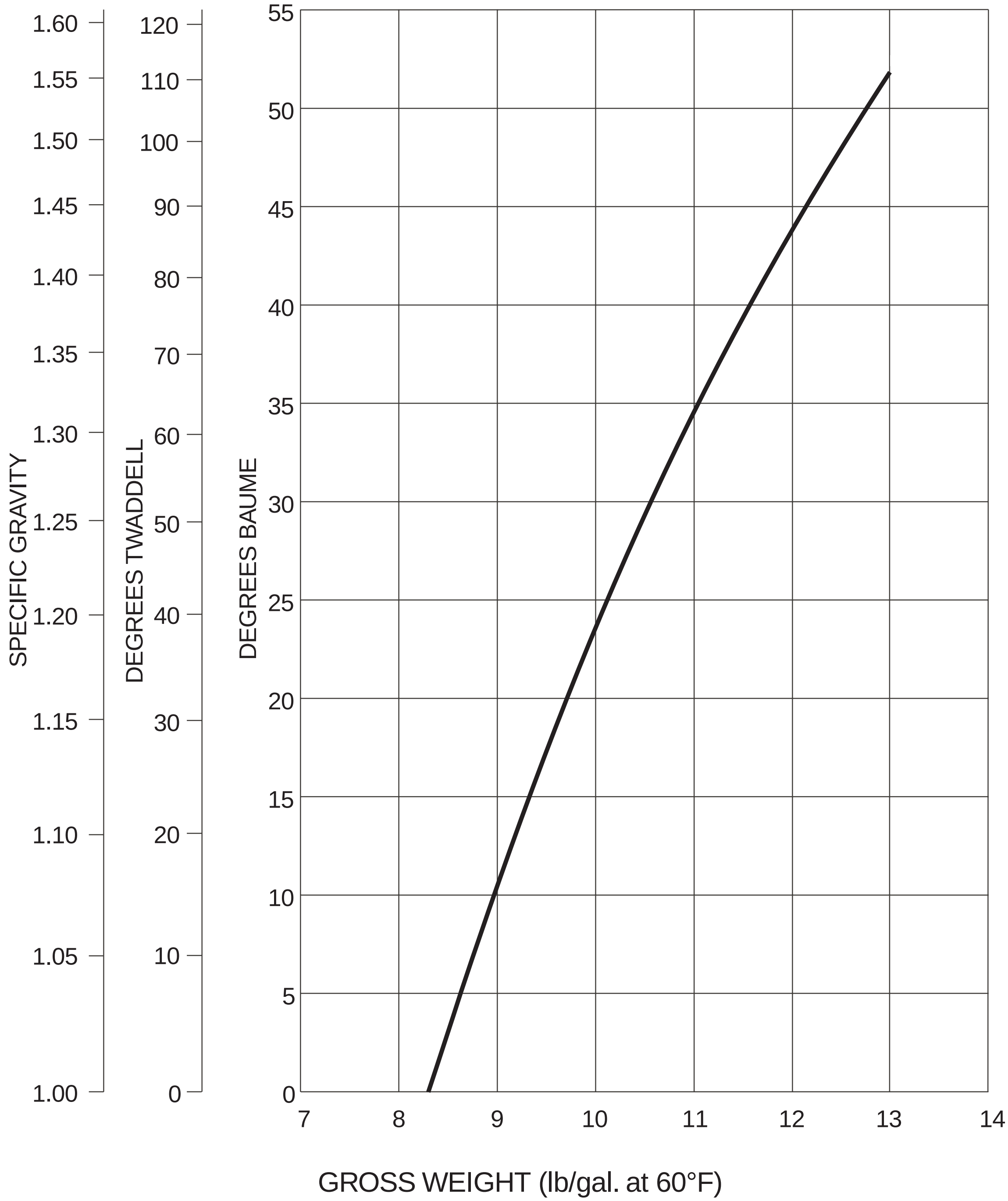
TECHNICAL DATA

Graph 2. Detemining Dilution Volumes

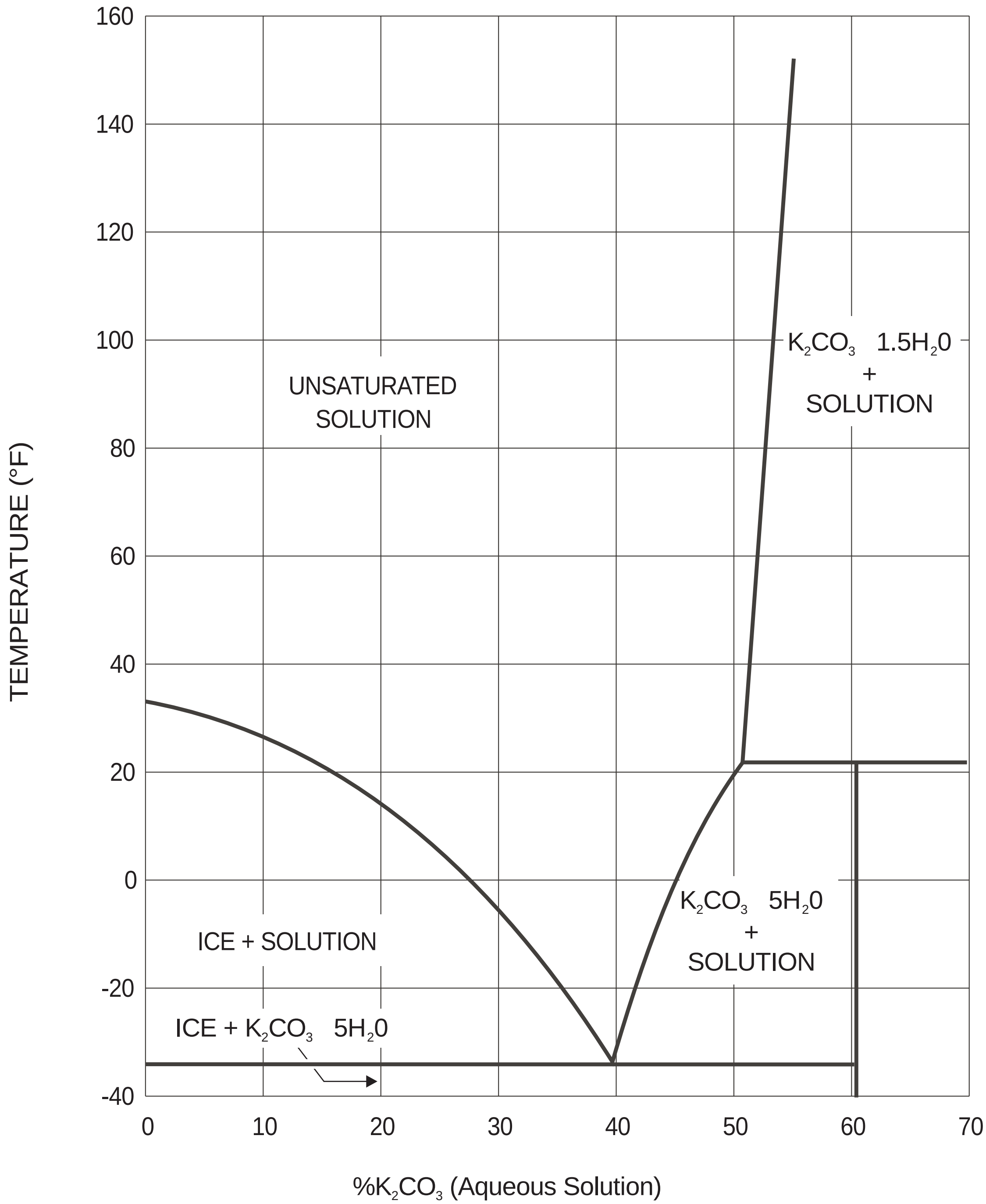


TECHNICAL DATA

Graph 3. Gross Weight of Aqueous Solutions at 60°F

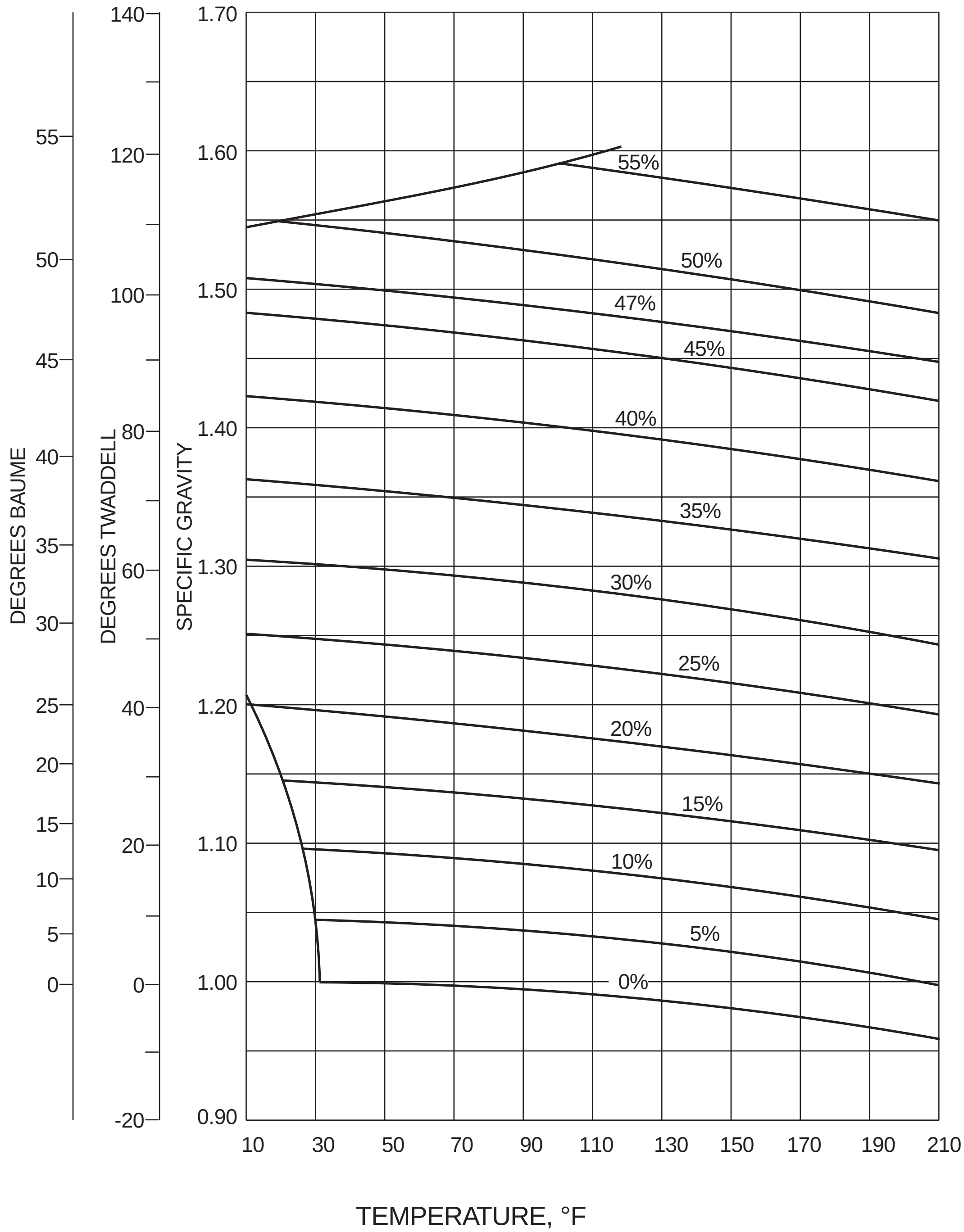


Graph 4. Solubility and Temperature Correlations

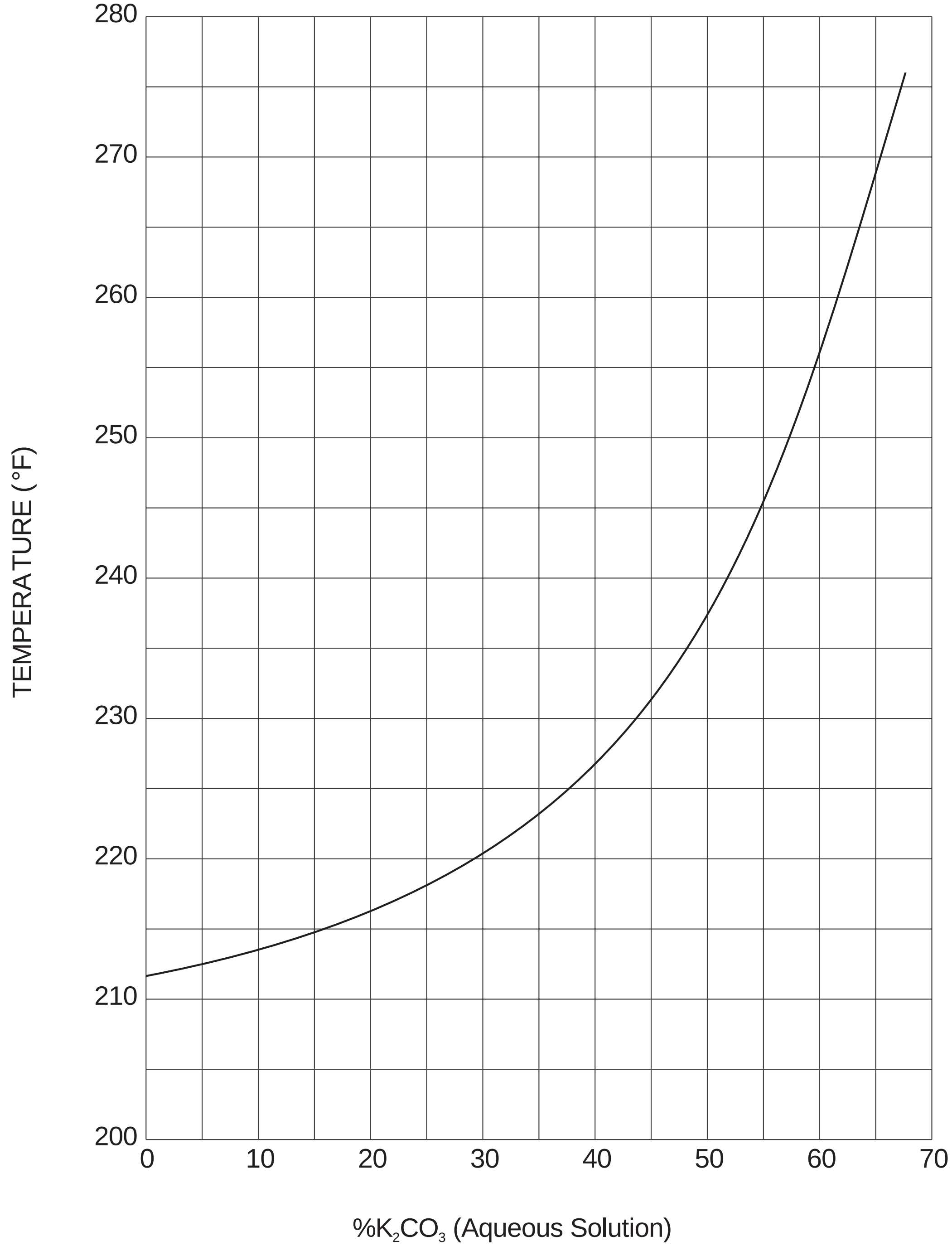


TECHNICAL DATA

Graph 5. Solution Density at Various Temperatures



Graph 6. Boiling Points at 760 mm Hg



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XI. Methods of Analysis

Methods are based upon analytical procedures as performed at the Muscle Shoals Plant Laboratory. Some variations are included and alternatives are discussed.

DETERMINATION OF POTASSIUM CARBONATE

(Total Alkalinity as Potassium Carbonate and/or Potassium Oxide)

PURPOSE AND THEORY

The alkaline strength of potassium carbonate is determined through titration with hydrochloric acid using modified methyl orange as the indicator.

Potassium carbonate with either potassium bicarbonate or potassium hydroxide present reacts with the acid to produce potassium chloride, water, and carbon dioxide.

The alkaline strength of the sample is computed from the volume of acid required for the titration and may be reported as either percent potassium carbonate or potassium oxide. When required, the exact amount of potassium carbonate can be determined by subtracting that portion of the acid required to neutralize the potassium hydroxide or potassium bicarbonate that is present in the sample.

$$K_2CO_3 + 2HCl \rightarrow 2KCl + H_2O + CO_2$$
$$KHCO_3 + HCl \rightarrow KCl + H_2O + CO_2$$
$$KOH + HCl \rightarrow KCl + H_2O$$

NOTE:
An automatic titrator may be utilized for ease of analysis, especially if this test is performed often. Contact Technical Service for further information concerning automatic titration.

PROCEDURE

This procedure should be performed as rapidly as possible to prevent adsorption/absorption of atmospheric CO_2.

1. Weigh a 4.000 to 6.000 gram sample of anhydrous potassium carbonate into a glass weighing bottle. For 47% potassium carbonate solution, use a 8.000 to 10.000 gram sample.
2. Transfer quantitatively the weighing bottle into a 250 milliliter erlenmeyer flask. Add approximately 100 ml of distilled water and four drops of the modified methyl orange indicator.
3. Titrate with standardized 1 N hydrochloric acid using a Class A 100 milliliter buret until the color of the solution changes from green to a steel gray.
4. Record the required volume of acid used to the nearest 0.1 milliliter.

CALCULATIONS

Report total alkalinity as potassium carbonate and/or potassium oxide to the nearest 0.01%.

Let: W = Weight of sample (grams)
V = Volume of HCl required (milliliters)
N = Concentration of HCl (Normality)

$$\% \text{ Total Alkalinity as } K_2CO_3 = \frac{(V)(N)(6.91)}{W}$$

$$\% \text{ Total Alkalinity as } K_2O = \frac{(V)(N)(4.71)}{W}$$

REAGENTS

Hydrochloric Acid, Standardized, 1 N Solution
Indicator, Modified Methyl Orange.

DETERMINATION OF POTASSIUM BICARBONATE OR POTASSIUM HYDROXIDE

PURPOSE AND THEORY

The bicarbonate (HCO_3) and hydroxide (OH) of potassium cannot co-exist in solution.

- If hydroxide is present, carbonate is precipitated by the addition of barium chloride. The hydroxide is determined by titration with standardized hydrochloric acid.
- If bicarbonate is present, it is converted to carbonate by the addition of an excess of standardized caustic soda solution. The carbonate is precipitated by the addition of barium chloride solution. The excess caustic soda is determined by titration with standardized hydrochloric acid.

$KHCO_3 + KOH \rightarrow K_2CO_3 + H_2O$

$K_2CO_3 + BaCl_2 \rightarrow 2KCl + BaCO_3$

PROCEDURE

This procedure should be performed as rapidly as possible to prevent adsorption/absorption of atmospheric CO_2.

A. Sample

1. Weigh a 2.000 to 3.000 gram sample of anhydrous potassium carbonate into a weighing bottle and seal with the cover. For 47% K_2CO_3 solution use a 4.000 to 6.000 gram sample. Record as “W”.
2. Transfer quantitatively the contents of the weighing bottle into a 500 ml erlenmeyer flask. Rinse bottle into the flask using distilled water and cover carefully. Add approximately 200 ml of distilled water and 4 to 6 drops of phenolphthalein indicator.
3. Add 10 ml of 0.1 N sodium hydroxide solution and 100 ml of a 10% barium chloride solution to the flask.
4. Titrate with 0.1 N hydrochloric acid until the pink color is just discharged. Record milliliters of 0.1 N HCl used as Titer “A”.

B. Blank

1. To a 500 ml erlenmeyer flask, add approximately 200 milliliters of distilled water. Add 4 to 6 drops of phenolphthalein indicator.
2. Add 10 ml of 0.1 N sodium hydroxide solution and 100 ml of a 10% barium chloride solution to the flask.
3. Titrate with 0.1 N hydrochloric acid until the pink color is just discharged. Record milliliters of 0.1 N HCl used as Titer “B”.

CALCULATIONS

KOH is present if “A” is greater than “B”.

$KHCO_3$ is present if “A” is less than “B”.

Only K_2CO_3 is present if “A” equals “B”.

$$\%\ KOH = \frac{(\text{ml "A" - ml "B"})(N)(5.61)}{W}$$

$$\%\ KHCO_3 = \frac{(\text{ml "B" - ml "A"})(N)(10.01)}{W}$$

REAGENTS

Barium Chloride, 10 % solution

Hydrochloric Acid, 0.1 N, Standardized

Sodium Hydroxide, 0.1 N, Standardized

Phenolphthalein Indicator

DETERMINATION OF POTASSIUM CHLORIDE

PURPOSE AND THEORY

This method for determination of chloride is referred to as either the Volhard or Thiocyanate-Ferric Alum method. It is applicable to the titration of chloride in acid solutions. When an acidic silver ion solution and an alkali thiocyanate are mixed in the presence of a ferric salt, the thiocyanate has a selective action toward silver, resulting in silver thiocyanate. Any excess of thiocyanate required by the silver reacts with ferric salt to form reddish-brown ferric thiocyanate, indicating the completion of the reaction.

An excess of silver nitrate is added to a nitric acid solution containing chloride and ferric indicator. The excess silver nitrate is titrated with ammonium thiocyanate.

$$AgNO + KCl \rightarrow AgCl + KNO_3$$

$$AgNO_3 \text{ (excess)} + NH_4CNS \rightarrow AgCNS + NH_4NO_3$$

$$6NH_4CNS + Fe_2(SO_4)_3 \rightarrow 2Fe(CNS)_3 + 3(NH_4)_2SO_4$$

PROCEDURE

1. Weigh a 40 gram sample of anhydrous potassium carbonate into a 250 ml erlenmeyer flask. For 47% solution, use a 50 gram sample.
2. Make up to 100 ml with distilled water and neutralize sample with 1:1 nitric acid using litmus paper as indicator. Caution: Neutralize carefully - the sample will effervesce during neutralization.
3. Add one milliliter of ferric indicator.
4. Add 20 ml of 0.05 N silver nitrate.
5. Titrate to reddish-brown endpoint with 0.05 N ammonium thiocyanate. Endpoint should hold for at least 15 seconds.
6. Minimum volumes of $AgNO_3$ and NH4CNS should be 10 ml and 5 ml, respectively.

NOTE: An automatic titrator may be utilized for ease of analysis, especially if this test is performed often. Contact Technical Service for further information concerning automatic titration.

CALCULATIONS

Let: W = Weight of sample (grams)
- N_1 = Concentration of $AgNO_3$ used (Normality)
- T_1 = Volume of $AgNO_3$ required (milliliters)
- N_2 = Concentration of NH_4CNS used (Normality)
- T_2 = Volume of NH_4CNS required (milliliters)

$$\% \text{ KCl} = \frac{(T_1N_1 - T_2N_2)(7.455)}{W}$$

REAGENTS

Ammonium Thiocyanate, 0.05 N, Standardized
Silver Nitrate, 0.05 N, Standardized
Ferric Indicator
Nitric Acid, 1:1 Solution

DETERMINATION OF SODIUM

PURPOSE AND THEORY

Sodium content in potassium carbonate is determined with an atomic absorption (AA) spectrophotometer. The sample is diluted to maintain total dissolved solids at an acceptable level for proper operation of the burner-aspirator system. An air-acetylene flame is utilized.

Sodium in parts per million (Na, ppm) is determined through a standard additions methodology. The diluted sample is analyzed along with additional sample aliquots which are 'spiked' with known amounts of sodium. Most atomic absorption instrumentation includes built-in programs to calculate standard addition results. If appropriate software is not available, raw sample results may be plotted manually and the content of the original sample may be extrapolated from the curve. If manual calculation is necessary, curves should be prepared daily. If a large number of samples is to be analyzed, it may be sufficient to prepare only one or two sets of standard additions. The slope of the resulting curve(s) will be suitable for analysis of additional samples providing all sample matrices are similar.

ANALYSIS

1. Weigh 10 grams of potassium carbonate into a 100 ml volumetric flask. Dilute the flask to volume with deionized water and assure that the entire sample is dissolved.
2. Into each of three 100 ml volumetric flasks, pipet 0.5 ml of the diluted sample.
3. For the first flask, merely dilute to volume with deionized water. Mark this flask ADD0.
4. To the second flask, pipet 1 ml of a 100 ppm sodium standard. Mark this flask ADD1. To the third flask, pipet 2 ml of the 100 ppm sodium standard. Mark this flask ADD2. Dilute both of these flasks to volume with deionized water.
5. Set up and optimize your AA according to the manufacturer's specifications. Analyze all three samples using the standard additions mode on your AA.
   a) ADD 0 is obviously the zero addition.
   b) ADD1 is an addition of 1 ppm or, if you prefer, an addition of 2000 ppm if the sample dilution is taken into account.
   c) ADD2 is an addition of 2 ppm or 4000 ppm if the sample dilution is taken into account.
6. If your instrument does not automatically calculate results for standard additions, record the instrument readout and plot the results manually. If information is needed concerning manual calculation of standard additions, you may wish to consult an analytical chemistry textbook.
7. If additions were considered as 2000 and 4000 ppm, results obtained are the ppm sodium in the original sample. If additions were considered as 1 and 2 ppm, multiply the result by 2000 to obtain the ppm sodium in the original sample.

REAGENTS

Standard Sodium Solution, 100 ppm

NOTE:
Other techniques, including ICP-AES may be utilized. Direct analysis, rather than standard additions, is possible if matrix matched standards are utilized. Contact Technical Service for further information concerning these other options.

DETERMINATION OF IRON

PURPOSE AND THEORY

The thiocyanate method is used for the determination of small amounts of ferric iron. Ferric iron and potassium thiocyanate in an acidic solution form a red-colored species. The intensity is proportional to the quantity of iron present.

$Fe^{+3} + 3CNS \rightarrow Fe(CNS)_3$

A visual comparison may be made between a prepared sample and a standard. The color intensity of the standard is adjusted to match that of the sample by adding a solution containing a known amount of iron. The volume of standard iron solution required is used to calculate the quantity of iron in the sample.

More commonly, especially if iron is determined routinely, a spectrophotometer is utilized. A calibration curve is constructed for the instrument using solutions of known iron concentration.

PROCEDURE

1. Weigh 10 grams of anhydrous potassium carbonate into a 100 ml volumetric flask. For 47% K_2CO_3 solution, use a 20 gram sample.
2. Add 25 ml of distilled water and neutralize with 6.5 N HCl using litmus paper as indicator and add 6 ml HCl in excess. Caution: Neutralize carefully - the sample will effervesce during neutralization.
3. Cool in water bath.
4. Add one drop hydrogen peroxide (H_2O_2) and mix well.
5. Add 10 ml of 1.5 N potassium thiocyanate solution and dilute to volume with distilled water. Mix well.
6. Measure absorbance on a Beckman "B" spectrophotometer (or equivalent instrument) using a wavelength of 480 millimicrons and a sensitivity setting of "2". Zero instrument with a reagent blank using 50 ml cells. Read within 15 minutes.
7. A calibration curve is prepared by using iron standards in an equivalent potassium chloride concentrated solution. Then, starting with Step 4, the same procedure is followed.

NOTE: Other techniques, including ICP-AES may be utilized. Contact Technical Service for further information.

CALCULATION

$$\text{Weight of Fe (grams)} = \frac{\text{Microgram Fe from Curve}}{10^6}$$

$$\%\ Fe = \frac{\text{(Weight Fe)(100)}}{\text{Weight of Sample}}$$

REAGENTS

Hydrogen Peroxide, 30% solution
Potassium Thiocyanate,
1.5N solution (146 g/L)
Hydrochloric Acid, 6.5 N

DETERMINATION OF SULFATE

PURPOSE AND THEORY

The addition of a barium chloride solution to a slightly acidic solution containing sulfate ion results in the precipitation of barium sulfate. Under specified conditions, the precipitation may be regarded as quantitative. The barium sulfate is recovered and weighed. The sulfate content of the sample is then computed and reported as percent K_2SO_4.

$$K_2SO_4 + BaCl_2 \rightarrow BaSO_4 + 2KCl$$

PROCEDURE

1. Weigh a 50.0 gram sample in a 600 ml beaker, or 100.0 grams of 47% solution. Add 300 ml of distilled water and 3-4 drops of methyl orange indicator.
2. Add concentrated hydrochloric acid; 1 ml in excess of that required to change the solution color from yellow to red. Caution: Neutralize carefully - the sample will effervesce during neutralization.
3. Filter through a No. 42 Whatman filter paper and wash filter paper with two washes of hot distilled water, collecting the filtrate in a 600 ml beaker.
4. Heat the filtrate to boiling, add 10 ml of barium chloride solution slowly with constant stirring. Continue to boil gently for 3-5 minutes. Allow precipitate to stand in a warm place for three hours or at room temperature overnight.
5. Filter off the precipitate (cold) on a Whatman 42 filter paper, use a rubber policeman to free any solids sticking to the beaker.
6. Wash the precipitate with successive small portions of cold distilled water until a small test portion of the wash water does not become cloudy with the addition of 1-2 drops of silver nitrate.
7. Transfer precipitate and filter paper to a tared 15 ml crucible (heated 15 minutes at 1000°F and cooled in a desiccator). Cautiously char filter paper by tilting the crucible on a triangle over a controlled Fisher burner, then heat to about 1000°F for one-half hour.
8. Transfer the crucible to a desiccator, cool to room temperature then weigh. Record weight to nearest 0.0001 gram.

CALCULATIONS

Report the results as K_2SO_4 to the nearest 0.0001%.

Let: W_1 = Sample Weight (grams)
W_2 = Tared Crucible Weight (grams)
W_3 = Tared Crucible Weight plus precipitate (grams)

$$\% K_2SO_4 = \frac{(W_3 - W_2)(74.65)}{W_1}$$

REAGENTS

Barium Chloride, 10% Solution
Hydrochloric Acid, Concentrated
Silver Nitrate Solution, 5%

DETERMINATION OF HEAVY METALS (as Pb)

PURPOSE AND THEORY

Metals, such as lead, zinc, tin and copper, in low concentrations are precipitated as colloidal sulfides in weakly acidic solution. The turbidity of the precipitated sample is compared with that of a standard lead solution treated in an identical manner.

PROCEDURE

1. Weigh a 20.0 ± 0.1 gram sample in a small beaker and transfer to a 100 ml volumetric flask with water. For a 47% solution, weight 40.0 ± 0.1 grams of solution into a 100 ml volumetric flask.
2. Transfer a 10.0 ml aliquot to a 100 ml beaker. Neutralize with 1.0 N acetic acid to a pH of 3.0 to 4.0. Transfer quantitatively to a 50 ml Nessler tube.
3. To a second 50 ml Nessler tube pipet 2 ml of the standard lead solution prepared as given below. Dilute to 25 ml with water and adjust to a pH of 3.0 - 4.0 as in Step 2.
4. Dilute both tubes to about 35 ml with water and add 10 ml of freshly prepared hydrogen sulfide solution then fill to 50 ml volume with water. Mix well and allow to stand for five minutes.
5. Compare the sample tube with the standard by viewing downward over a white surface. The sample should be no darker than the standard. The standard solution contains 10 ppm heavy metals as lead. For other lead limits, change the size of aliquot taken from lead standard.

NOTES: Hydrogen sulfide is a poisonous, flammable gas. Commercially available saturated hydrogen sulfide solution may be purchased to avoid the hazards of handling hydrogen sulfide gas.

REAGENTS

Acetic Acid, 1 N Solution
Heavy Metals Standard, 10 Microgram of Lead per Milliliter
Hydrochloric Acid, Concentrated
Hydrogen Sulfide, Saturated Solution

DETERMINATION OF MOISTURE

PURPOSE AND THEORY

Two methods may be used to determine moisture in potassium carbonate. If a relatively complete analysis of the sample is made, then water can be calculated by difference. If the moisture content is desired quickly, then the oven loss method can be used. In this method, care must be taken in sample handling since potassium carbonate is deliquescent. When potassium bicarbonate is present, a correction for its volatile decomposition products equivalent to 0.3 of the determined potassium bicarbonate should be subtracted from the oven loss to give free moisture.

$$2KHCO_3 \rightarrow K_2CO_3 + H_2O + CO_2$$

PROCEDURE

1. Weigh a 5.000 gram sample in an aluminum dish.
2 Dry for two hours at 250°C.
3. Sample should be placed in a desiccator for no more than 10 minutes. Weigh sample warm, but not at the elevated drying temperature. Potassium carbonate is deliquescent and will pick up moisture even in a desiccator.

CALCULATIONS

Let: W = Sample Weight (grams)
A = Oven Loss Weight (grams)
B = Potassium Bicarbonate Concentration (%)
0.3B = Concentration of Potassium Bicarbonate Volatiles (%); when $KHCO_3$ is small or missing, this term is very small or zero.

$$\% \ H_2O = \frac{(A)(100) - (0.3B)}{W}$$

PREPARATION OF SPECIAL AND STANDARD SOLUTIONS

Acetic Acid, 1N: Dilute 60 ml of glacial acetic acid to one liter with water.

Ammonium Thiocyanate, Standardized, 0.05N: Dissolve 3.8062 grams of NH_4CNS in water and dilute to a liter. It is standardized against a standard silver nitrate solution, using a ferric alum indicator.

Barium Chloride, 10%: Dissolve 120 grams of reagent-grade $BaCl_2 \cdot 2H_2O$ in 880 ml of distilled water.

Ferric Indicator: A saturated solution of reagent-grade $FeNH_4(SO_4)$ in distilled water.

Hydrochloric Acid, Standardized, 1N: Add 83 ml of concentrated HCl to a one liter volumetric flask containing distilled water, dilute to mark and mix well. (This solution is also available commercially.) Standardize with 5.0 grams of freshly prepared anhydrous sodium carbonate. Use primary standard analytical reagent grade Na_2CO_3. Heat 10 to 20 grams of sodium carbonate in a weighing bottle at 250°C for one hour.

Hydrochloric Acid, Standardized, 0.1N: Dilute exactly 100 ml of 1N standardized hydrochloric acid solution at 20°C to one liter with distilled water in a volumetric flask and mix thoroughly.

Hydrochloric Acid, 6.5N: Add 540 ml of concentrated hydrochloric acid to a one liter volumetric flask containing distilled water and dilute to the 1000 ml mark.

Hydrogen Sulfide Saturated Solution: Prepare fresh solution daily. Bubble hydrogen sulfide through 250 ml of distilled water for 15 minutes in a hood. Hydrogen sulfide is very toxic. (A saturated solution is available commercially.)

Iron Standard Solution: Dissolve 0.7022 grams of reagent-grade ferrous ammonium sulfate crystals, $FeSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$, in 50 ml of 5N nitric acid. Dilute to 500 ml in a volumetric flask with distilled water and mix well. Dilute 10 ml of this solution to 1 liter in a volumetric flask and mix thoroughly. One ml equals 20 micrograms of Fe.

Lead Standard Solution: Dissolve 0.1598 grams of lead nitrate, $Pb(NO_3)_2$, in 100 ml of water containing one ml of concentrated nitric acid. Solution A: dilute to one liter for 100 micrograms per ml. Solution B: dilute 10 ml of solution A to 100 ml for 10 micrograms per ml. Prepare solutions daily.

Modified Methyl Orange Indicator: Dissolve 0.14 grams of methyl orange and 0.12 grams of xylene cyanolle FF in deionized water and dilute to 100 ml.

Phenolphthalein Indicator: Dissolve 1 gram of phenolphthalein in 100 ml of methanol.

Potassium Chloride, 20%: Dissolve 200 grams of reagent-grade potassium chloride in 800 ml of distilled water.

Potassium Thiocyanate, (~ 1.5N solution): Dissolve 146 grams of reagent-grade potassium thiocyanate in one liter of distilled water.

Silver Nitrate, Standardized, 0.05N: Weigh out about 8.55 grams of silver nitrate and dissolve in a liter of distilled water. Standardize against a standard sodium chloride solution by the method of Mohr, Volhard or Fajous.

Silver Nitrate, 5%: Dissolve 5 grams of reagent grade silver nitrate in 95 ml of distilled water.

Sodium Hydroxide, Standardized, 0.1N: Dissolve about 6 grams of caustic soda in a half-liter of distilled water and add enough $BaCl_2$ to precipitate any carbonate that may be present. Allow $BaCO_3$ to settle, then filter into a liter flask and dilute to mark with CO_2-free water. Standardize with hydrochloric acid using phenolphthalein as indicator. To prevent CO_2 from entering the bottle, the incoming air is filtered through a guard tube containing ascarite.

Sodium Standard Solution: ASTM certified standard atomic absorption sodium solution.

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