This invention deals with the manufacture of anhydrous alkali metal hydroxides and more particularly involves the preparation and packaging of high purity sodium hydroxide.

Anhydrous alkali metal hydroxides of extreme purity are often required. Anhydrous sodium hydroxide (caustic soda) destined for particular uses such as in the manufacture of textiles, especially rayon, certain soaps and other fields is required to satisfy extremely rigid quality standards. One standard is a very low, almost negligible, iron content. Thus, anhydrous sodium hydroxide of superior purity contains less than 15 parts per million iron, and more usually less than 10 parts per million iron, by weight of the caustic soda.

Molten anhydrous sodium hydroxide is particularly prone to become contaminated with iron. Thus, when in contact with iron, steel or like iron containing surfaces, molten anhydrous sodium hydroxide is readily contaminated with concentrations of iron well above 15 parts per million by weight of the sodium hydroxide. Techniques for avoiding such iron contamination can be both costly and inconvenient.

One serious source of iron contamination is encountered in the packaging of molten anhydrous sodium hydroxide. Anhydrous sodium hydroxide is packaged by filling steel drums with the molten material at 640° F. to 750° F. The molten charge is then permitted to solidify, the drums closed and the covers welded in place. During this packaging, the molten anhydrous sodium hydroxide is in contact with the steel inner surface of the drum and thus becomes contaminated with iron in unduly high concentrations.

Of further consequence in the packaging of sodium hydroxide is the drum cost. Since these drums cannot be reused, their cost is an important consideration. Drums which are lined or fabricated of non-iron containing materials such as nickel are more expensive than steel drums, hence, except for their effect on iron contamination, steel drums would be employed.

According to this invention, an efficient, simple process has been devised for the manufacture and packaging of anhydrous alkali metal hydroxide whereby steel or like iron containing drums may be used without encountering serious iron contamination. By virtue of this invention, it is now possible to package anhydrous sodium hydroxide in steel drums and obtain a product of high purity with a minimum, tolerable iron content in contrast with other experiences. This permits a considerable savings in packaging costs.

It has now been discovered in accordance with this invention that no detrimental iron contamination occurs when essentially anhydrous molten alkali metal hydroxide, notably sodium hydroxide, containing less than 0.6 percent, and more suitably no more than 0.3 percent, water by weight of the alkali metal hydroxide is charged to drums of steel or like iron containing construction materials while at molten temperatures of 610° F. up to 635° F. Both the very low water content of less than 0.6 percent and charging temperatures between 610° F. and 635° F. of the alkali metal hydroxide coat in the elimination or retardation of iron contamination otherwise observed with the use of steel drums.

Essentially anhydrous alkali metal hydroxides are conventionally obtained by dehydrating aqueous solutions. That is, sodium hydroxide as it is manufactured by electrolysis of brine solutions is obtained as a cell liquor containing on the order of 10 to 20 percent sodium hydroxide by weight. Such solutions are concentrated by removal of water to obtain ultimately sodium hydroxide in substantially anhydrous state. The removal of water as the sodium hydroxide is more concentrated demands increasingly severe conditions such as higher temperatures and prolonged heating. One of the practical means for removing water from these more concentrated aqueous sodium hydroxide solutions involves their dehydration under vacuum conditions, using a high boiling heat exchange medium. In this manner, molten concentrated sodium hydroxide products containing upwards of 95 percent by weight sodium hydroxide are obtained.

Completely anhydrous sodium hydroxide, e.g. sodium hydroxide containing no water, is not conveniently obtained in this vacuum dehydration. Efficient vacuum dehydration precludes complete removal of water. Instead, sodium hydroxide containing from 0.8 to 2.0 percent water or possibly even a somewhat higher water content constitutes the practical product of vacuum dehydration. To operate otherwise requires excessively prolonged heating or, more than likely, a separate dehydration step, e.g. a second evaporator. Either case represents magnified costs.

It is in conjunction with packaging in steel drums of essentially anhydrous sodium hydroxide as produced by vacuum dehydration that this invention provides most pronounced benefits. When such molten sodium hydroxide is packaged in steel drums, it becomes contaminated intolerably with iron, notwithstanding that the dehydration is accomplished under conditions designed to avoid iron contamination, e.g. in nickel equipment, and that immediately prior to packaging the sodium hydroxide is not contaminated with iron. Performance of this invention is therefore particularly effective in packaging vacuum dehydrated, anhydrous, low iron content molten sodium hydroxide.

In the practice of this invention, an inert gas such as air is passed through essentially anhydrous, low iron content molten sodium hydroxide containing in excess of 0.6 percent water by weight, usually from 0.8 to 2.0 percent by weight water, such as provided by vacuum dehydration, until the water content of the resulting aerated molten product is less than 0.6 percent, and preferably less than 0.3 percent. This molten product is thereafter loaded into drums of steel or like iron construction material at a regulated temperature of 610° F. to 635° F., more ideally 615° F. to 625° F. Drummed in this manner, the sodium hydroxide satisfies the rigid requirements of high purity, particularly with respect to iron content.

 Expedients for bubbling, injecting or otherwise passing the inert gas through molten anhydrous sodium hydroxide are varied. Direct injection of the inert gas into a molten body of sodium hydroxide suffices. One convenient technique involves placing an enclosed storage vessel containing a pool of molten sodium hydroxide under
vacuum and sucking the inert gas, notably air, through the pool. Slightly subatmospheric pressures are adequate and suit this end.

Normal atmospheric air, e.g. air at prevailing atmospheric pressure and moisture content, functions effectively and is recommended because of availability. Other inert gases including neon and nitrogen by way of example are appropriate. By inert gases, those gases which are not reactive under the prevailing conditions with the liquid being treated are intended.

The molten sodium hydroxide in this treatment need be maintained only at temperatures which preclude solidification, e.g. temperatures above the melting point of the sodium hydroxide composition. Most used are temperatures of 610° F. to 700° F.

Surprisingly, by passing air or like inert gas through the molten sodium hydroxide, a substantial portion of residual water content of vacuum dehydrated sodium hydroxide is removed with ease and at low cost. Molten sodium hydroxide containing no more than 0.3 percent water by weight is obtained from compositions originally having water contents of 0.8 to 1.5 percent, or more, by weight. The ease and economy by which such low water content sodium hydroxide is realized contrasts strikingly with the difficulty and practical impossibility of accomplishing the same by vacuum dehydraton.

Molten sodium hydroxide which has been heated in this manner may be packaged in steel drums at from 610° F. to 635° F. Tests clearly indicate the iron contamination of these packaged products attributable to contact with the steel drum is below the maximum tolerable for high purity sodium hydroxide. On the other hand, sodium hydroxide not treated and packaged by the prescribed procedure develops an iron concentration which fails to meet high purity requirements.

This process is, of course, dependent upon the treatment and packaging of sodium hydroxide which after it has been dehydrated to substantially anhydrous state does not already contain an intolerable iron content. The usual techniques recognized to avoid iron contamination are therefore advisable employed during vacuum dehydration and the ensuing treatment with inert gas. Nickel lined equipment constitutes one precaution. Vacuum dehydrated anhydrous molten sodium hydroxide may be produced which contains less than 10 parts per million, and usually 2 to 8 parts per million, iron by weight. This quality material is well suited to packaging according to this invention.

The following example illustrates the manner in which this invention may be practiced:

**Example**

Essentially anhydrous sodium hydroxide containing 1.1 percent water and 6 parts per million iron by weight of the sodium hydroxide was obtained by vacuum dehydration in a nickel lined evaporator of aqueous sodium hydroxide. Typically, this vacuum dehydration involved feeding aqueous sodium hydroxide containing 77 percent by weight sodium hydroxide to an evaporator maintained at 635° F. and operated under 190 millimeters mercury pressure absolute.

This sodium hydroxide was then fed to a nickel-lined storage vessel to provide a liquid body of 50 tons of molten sodium hydroxide at a temperature of 610° F. to 660° F. Through two nickel nozzles disposed beneath the liquid level of the caustic soda, atmospheric air at 77° F., having a dew point of 25° F., was injected into the pool. Approximately 1,440 cubic feet of air (basis standard pressure and temperature) per ton of sodium hydroxide was employed. This treated anhydrous sodium hydroxide contained 0.3 percent water by weight. It was then loaded at 612° F. into steel drums wherein it was allowed to solidify while the drums were exposed to normal temperatures, e.g. 75-80° F. The iron content of these solidified drummed products were obtained as follows:

<table>
<thead>
<tr>
<th>Drum No.</th>
<th>Before Drumming</th>
<th>After Solidification in Drum</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>6</td>
<td>0</td>
</tr>
<tr>
<td>2</td>
<td>8</td>
<td>5</td>
</tr>
<tr>
<td>3</td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td>4</td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td>5</td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td>6</td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td>7</td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td>8</td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td>9</td>
<td>10</td>
<td>10</td>
</tr>
</tbody>
</table>

When molten sodium hydroxide was drummed while at 625° F., the solidified product in the steel drum contained 13 parts per million iron by weight of sodium hydroxide.

In contrast, molten sodium hydroxide drummed directly into steel drums (without the inert gas treatment) became unduly contaminated with iron. Vacuum dehydrated sodium hydroxide of the quality drummed in the foregoing example (e.g., containing 1.1 percent water and 6 parts per million iron) packaged in the same type of steel drums as used in the example was found to have the following iron contents:

<table>
<thead>
<tr>
<th>Temperature of Molten Sodium Hydroxide as Fed to Drum, ° F.</th>
<th>Iron Content in Drummed Solidified Product, Parts Per Million by Weight of Sodium Hydroxide</th>
</tr>
</thead>
<tbody>
<tr>
<td>610</td>
<td>17</td>
</tr>
<tr>
<td>615</td>
<td>31</td>
</tr>
<tr>
<td>620</td>
<td>30</td>
</tr>
<tr>
<td>625</td>
<td>35</td>
</tr>
<tr>
<td>630</td>
<td>50</td>
</tr>
<tr>
<td>635</td>
<td>55</td>
</tr>
</tbody>
</table>

Comparison of the iron contents of products packaged in steel drums in accordance with this invention and those not so-packaged, as evidenced by the foregoing data, clearly shows the advantages afforded by this invention.

It will be understood that this invention is applicable to the manufacture of high quality, essentially anhydrous sodium hydroxide, e.g. sodium hydroxide containing upwards of 95 percent, and usually about 99 percent, by weight sodium hydroxide on an anhydrous basis.

While this invention has been described by reference to specific details of certain embodiments, it is not intended that the invention be construed as limited to such details except insofar as they are included in the appended claims.

**Claim:**

1. The method which comprises vacuum dehydrating aqueous sodium hydroxide to essentially anhydrous molten sodium hydroxide, passing an inert gas through the molten sodium hydroxide until its water content is reduced to below 0.6 percent by weight and packaging the molten sodium hydroxide while at 610° F. to 635° F. in steel drums.

2. The method which comprises vacuum dehydrating aqueous sodium hydroxide to essentially anhydrous molten sodium hydroxide containing 0.8 to 2.0 percent water by weight, passing an inert gas through the molten sodium hydroxide whereby to reduce its water content to below 0.6 percent by weight and packaging at 610° F. to 635° F. this sodium hydroxide in steel drums.

3. The method which comprises vacuum dehydrating aqueous sodium hydroxide to essentially anhydrous mol-
ten sodium hydroxide containing by weight 0.8 to 2.0 percent water and between 2 and 8 parts per million iron, passing an inert gas through the molten sodium hydroxide until it contains less than 0.6 percent water by weight and packaging at 610° F. to 635° F. this molten sodium hydroxide in steel drums.

4. The method of claim 3 wherein the inert gas is air.

5. The method of claim 3 wherein the water content of the molten sodium hydroxide is reduced to no more than 0.3 percent by weight.

6. The method which comprises vacuum dehydrating aqueous sodium hydroxide to essentially anhydrous molten sodium hydroxide, passing an inert gas through this vacuum dehydrated molten sodium hydroxide while the molten sodium hydroxide is at 610° F. to 700° F. until its water content is reduced to below 0.6 percent by weight and packaging the molten sodium hydroxide while at 610° F. to 635° F. in steel drums.

7. The method of packaging essentially anhydrous sodium hydroxide which comprises vacuum dehydrating aqueous sodium hydroxide to provide molten sodium hydroxide, establishing a water content in the molten sodium hydroxide below 0.6 weight percent and packaging at 610° F. to 635° F. this molten sodium hydroxide in steel drums.

References Cited in the file of this patent

UNITED STATES PATENTS

1,883,211 Wilson .................. Oct. 18, 1932
1,907,988 Lynn et al. ................ May 9, 1933
1,956,138 Staub .................... Apr. 24, 1934
2,023,271 Hooker ................... Dec. 3, 1935
2,556,185 Josceline .................. June 12, 1951

FOREIGN PATENTS

134,902 Australia ................ Oct. 26, 1949