UNITED STATES PATENT OFFICE.

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DEHYDRATION OF CAUSTIC ALKALI.

983,834. Specification of Letters Patent. Patented Feb. 7, 1911.

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To all whom it may concern:

Be it known that we, PAUL ERWIN OBERREIT and FRIEDRICH MORITZ JAHRMARKT, doctors of philosophy and chemists, subjects of the King of Saxony, residing, respectively, at Ludwigshafen-on-the-Rhine, in the Kingdom of Bavaria, and Mannheim, in the Grand Duchy of Baden, German Empire, have invented new and useful Improvements in the Dehydration of Caustic Alkali, of which the following is a specification.

When manufacturing caustic alkalis by treating the carbonates with caustic lime, the caustic alkali is first obtained in the form of a dilute solution. The old method of obtaining solid caustic alkali from this consisted first of all in concentrating the crude soda lye in a vacuum evaporator continuing the evaporation until the impurities in the lye began to separate out, that is to say, until the solution contained about 22% of caustic soda and more than 75% of water. This liquor was removed from the vacuum apparatus, separated from the impurities that crystallized out and then brought into the finishing pots and evaporated as far as was desired or as far as was possible. These finishing pots were rapidly destroyed by the action of the caustic alkali upon them, the material of which they were formed being in part dissolved and rendering the alkali impure. Various methods of avoiding the disadvantages of the old process have been suggested. Thus, in 1895, in the British Letters Patent No. 12,320, the process which consists in heating the alkali which contains water in an open vessel while passing an electric current through the alkali, using the vessel as one of the electrodes, was described. This also, however, has disadvantages, since a considerable quantity of alkali superoxid is formed and this renders the caustic alkali useless for some purposes, such for instance as in the preparation of indigo.

We have now discovered that the water can be removed from caustic alkalis which are solid at ordinary atmospheric temperatures and the said alkalis be obtained in a dehydrated condition by heating the said alkalis *in vacuo*. For instance, by heating caustic soda *in vacuo* at a temperature of about one hundred and eighty degrees centigrade, or by heating caustic potash *in vacuo* at a temperature of about two hundred and sixty degrees centigrade, that is, at temperatures below those at which the caustic soda and caustic potash, respectively, melt, they can be obtained in a dehydrated condition, while the vessels (which may be of iron) in which they are heated are practically unattacked and therefore last much longer, and dehydrated alkalis are obtained which contain only traces of foreign metals. Of course, temperatures higher than those aforesaid can be employed so long as the vessel remains unattacked, and temperatures lower than those aforesaid can be employed, but in the latter case, the dehydration proceeds more slowly and scarcely to the same extent. Since cautic alkali containing, say, 10% of water attacks iron at temperatures above about 400° C., we carry out the dehydration according to our invention at temperatures between 170° C. and 400° C.

It is known that dilute caustic alkaline solutions can be concentrated *in vacuo*, and this method of concentration has been practiced to obtain caustic alkali solutions containing up to about fifty per cent. of caustic alkali, and it has been proposed to concentrate heavy lyes by distributing the lye in a thin layer upon the heated inner surface of a vacuum vessel provided with a special apparatus and thus obtaining a product containing as little as fifteen per cent. of water, and no claim is made to these processes, but it has not hitherto been known that any advantageous effect could be obtained by heating in an ordinary vacuum apparatus caustic potash, or caustic soda, which are so far free from water that they are solid at ordinary atmospheric temperatures, and it is our discovery that such solid caustic potash and solid caustic soda will entirely part with their water *in vacuo* at such low temperature that it is possible to effect the complete dehydration without the vessels employed being attacked to any objectionable extent and with practically no contamination of the resulting product. When starting with either such solid caustic soda, or such solid caustic potash, containing but little water, it can, if desired, be completely dehydrated according to our invention, without the caustic alkali melting.

Instead of treating caustic potash, or caustic soda, alone, of course a mixture of the two may be treated according to our invention.

Although by the process according to our invention complete dehydration can be effected and it is the object of our invention to effect this, yet in the expressions "dehydrated" and "dehydration" we include such degree of dehydration beyond that hitherto obtained as will give, for practical purposes, the benefit of our invention, and we do not claim the production of caustic alkali containing more than ten (10 per cent. of water.

The following examples will serve to illustrate how our invention can be carried into practical effect, but the invention is not confined to these examples. The parts are by weight.

Example 1: Heat, *in vacuo*, at a temperature of one hundred and eighty (180) degrees centigrade, while stirring, caustic soda which, while solid at ordinary atmospheric temperatures (say fifteen (15) degrees centigrade), at a slightly higher temperature dissolve in the water contained in it. Such a caustic soda is for instance one containing sixty per cent. of NaOH and melting at 52° C. The mass, which is at first liquid, afterward becomes thiek and finally becomes solid, and when suitable stirring apparatus is employed, it can be obtained as a powder.

Example 2: Heat, *in vacuo*, at a temperature of two hundred and sixty (260) degrees centigrade, while stirring, one hundred (100) parts of caustic potash, which is solid at ordinary atmospheric temperatures (say fiftween (15) degrees centigrade), but which, on slightly heating, dissolves in the water contained in it. Such a caustic potash is for instance one containing fifty eight per cent. of KOH and melting at 32° C. Continue the heating so long as any water distils off. The reaction proceeds as described in Example 1. If in this example, a temperature of from one hundred and eighty (180) to two hundred (200) degrees centigrade be maintained till about thirty-seven parts of water have distilled off, a technically pure caustic potash, of about ninety (90) per cent. is obtained.

Example 3: Heat, *in vacuo*, at a temperature of two hundred and sixty (260) degrees centigrade, while stirring, a mixture of equimolecular proportions of caustic soda and caustic potash each solid at ordinary atmospheric temperatures (say fifteen (15) degrees centigrade), but containing so much water as to yield a solution upon slightly heating. Such a mixture is for instance one containing sixty-three per cent. of the mixed alkalis and thirty-seven parts of water and melting at about 60° C. In this case the mass, at the temperature of the melt, remains liquid after complete dehydration has taken place.

Example 4: Grind, to a fine powder, commercial caustic potash containing for instance seventy per cent. of pure caustic potash and melting at about 140–145° C. and then heat it, *in vacuo*, gradually raising the temperature to about two hundred and sixty (260) degrees centigrade and maintain this temperature until no more water distils off.

Now what we claim is:

1. The process of obtaining caustic alkali from solutions thereof which consists in concentrating the caustic alkali solution until it has reached the point at which it would act upon the material of the containing vessel, and then further concentrating *in vacuo* at a temperature sufficiently low to prevent action of the material of the vacuum drier but sufficiently high to drive off substantially all of the water.

2. The process of obtaining caustic alkali from solutions thereof which consists in concentrating the caustic alkali solution until it has reached the point at which it would act upon the material of the containing vessel and then further concentrating *in vacuo* at a temperature of about 180° to 260° C.

In testimony whereof we have hereunto set our hands in the presence of two subscribing witnesses.

PAUL ERWIN OBERREIT.
FRIEDRICH MORITZ JAHRMARKT.

Witnesses:
J. ALEC. LLOYD,
Jos. H. LEUTE.