

March 19, 1946.

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2,396,664

METHOD OF DEHYDRATING CAUSTIC SOLUTIONS

Filed April 10, 1942

2 Sheets-Sheet 1

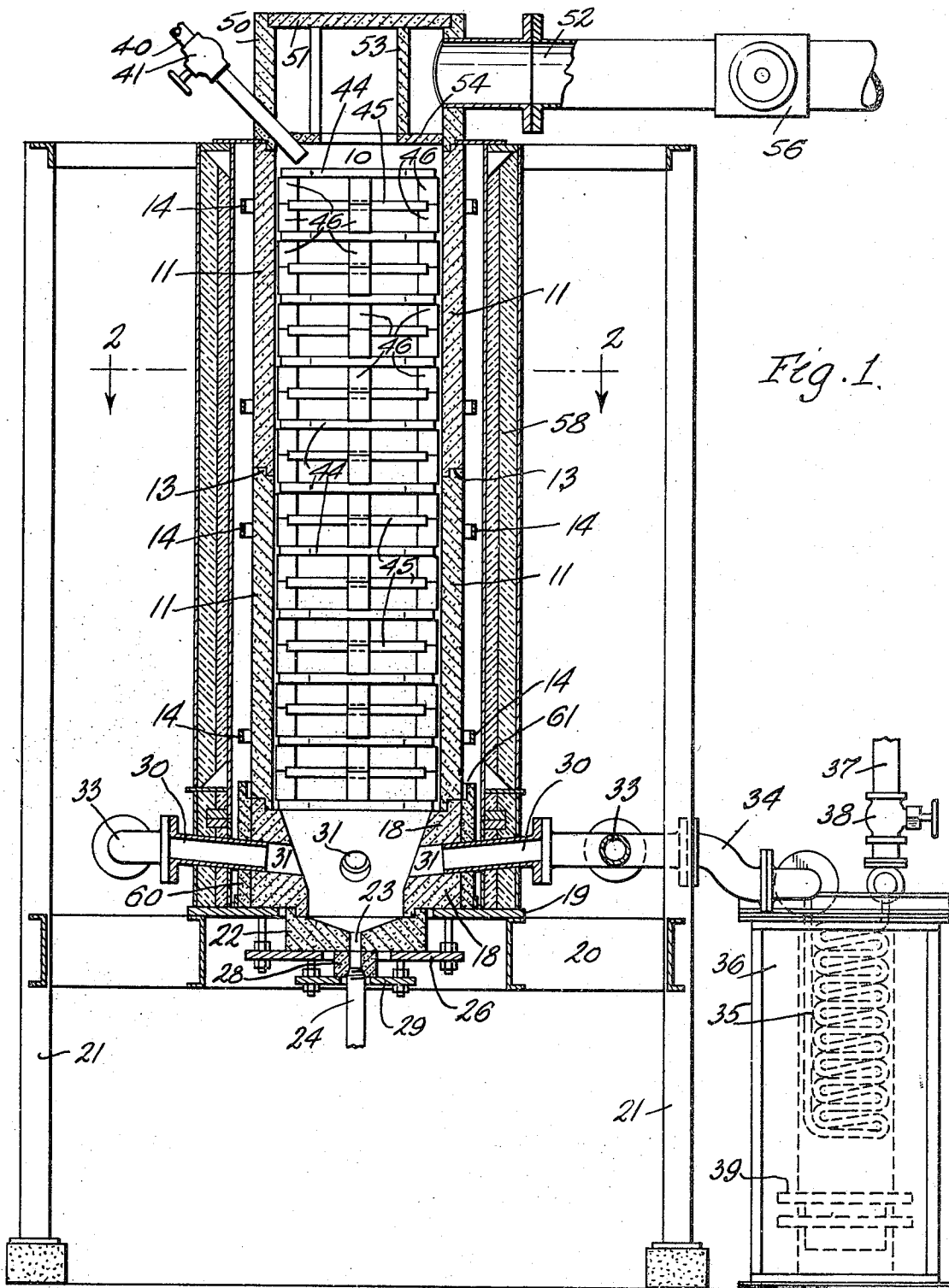


Fig. 1.

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2 Sheets-Sheet 2

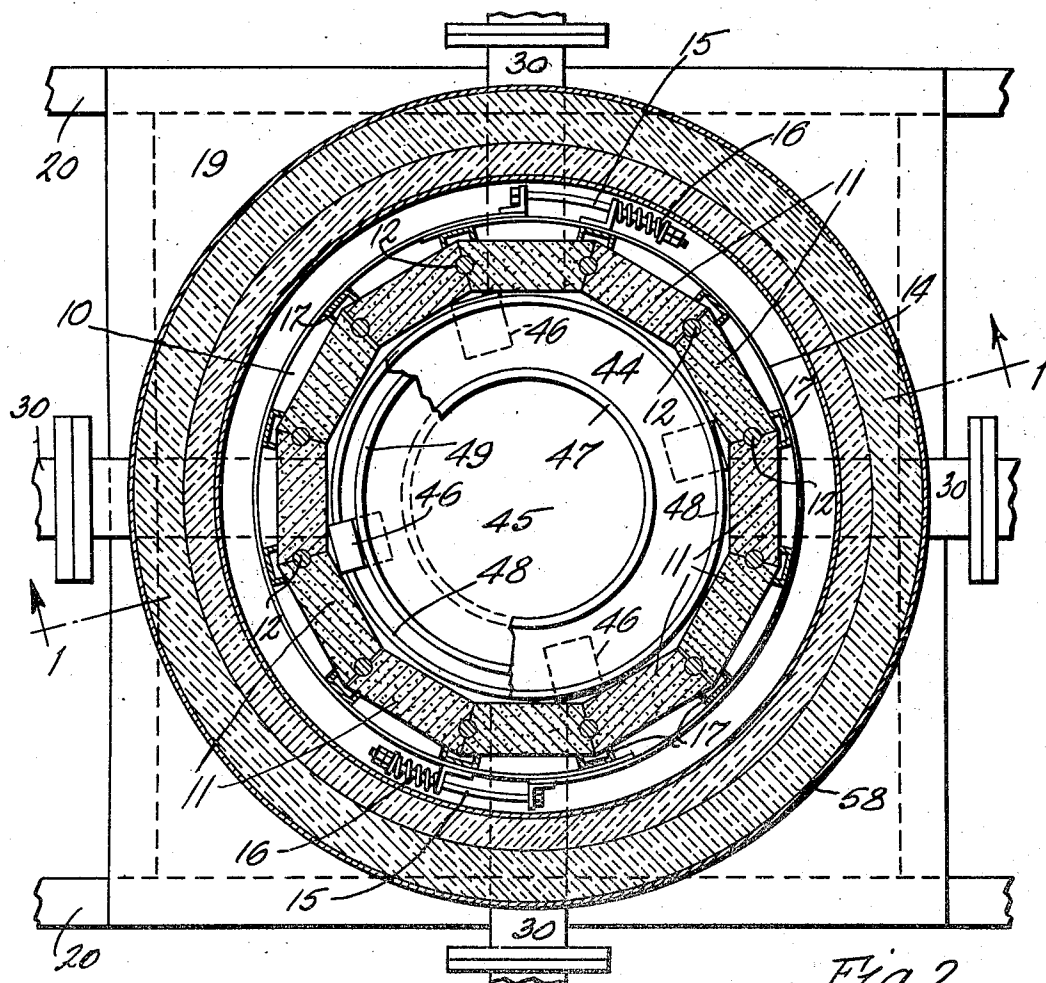


Fig. 2.

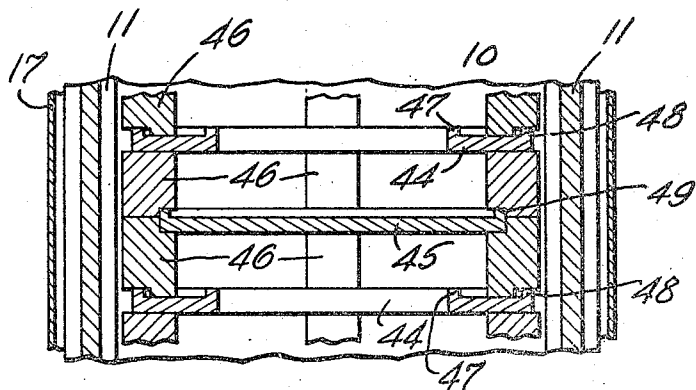


Fig. 3.

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METHOD OF DEHYDRATING CAUSTIC SOLUTIONS

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11 Claims. (Cl. 159—47)

This invention relates to methods of dehydrating caustic solutions, and more particularly to methods of this kind which may be carried on without adding impurities to the caustic liquids.

It has always been a serious problem to produce solid caustic potash and caustic soda or fairly concentrated solutions thereof free from contamination from the equipment in which this material has been concentrated or dehydrated, since these caustic solution have a tendency to attack nearly all materials which are ordinarily available for use in the building of such equipment. For many purposes, caustic potash or soda free from metallic impurities and carbonates is essential, and it is also desirable to have these pure caustics available in solid form. In my Patent No. 2,270,376, dated January 20, 1942, I have disclosed a process by means of which metallic impurities, such as compounds of iron, aluminum, and other metals, can be removed from caustic solutions. If, however, such pure solutions are then dehydrated in the ordinary manner now commonly used, which involves heating the solutions in a large iron container, then during the dehydration, the solutions pick up iron to such an extent as to greatly reduce their commercial value and to render them unsuitable for certain uses.

One of the objects of this invention is to provide a process whereby a caustic solution can be dehydrated without the addition thereto of impurities. Another object is to provide a process of this kind by means of which a caustic solution free from metallic impurities can be dehydrated without adding such impurities thereto. It is also an object of this invention to provide an efficient and reliable process of making solid caustic potash or soda in which no metallic impurities are added to this material during the dehydration thereof. A further object is to provide a continuous process for dehydrating caustic solutions without adding impurities thereto.

Other objects of this invention will appear from the following description and claims.

I have found that under certain conditions, caustic solutions can be dehydrated without contamination while in contact only with carbon surfaces. The use of carbon for lining containers for the caustic solutions during dehydration has heretofore not been considered practical for the reason that the temperatures employed for dehydration are so high as to cause fairly rapid oxidation of the carbon, and furthermore, carbonates would be formed as impurities in the caustic solutions. I have found, however, that oxidation of

carbon and the formation of carbonates of potassium or sodium can be prevented by the exclusion of an oxidizing atmosphere, such as air, from contact with the carbon surfaces which are subjected to the action of the caustic solutions. Under these conditions, it is also possible to expose the carbon to temperatures materially higher than those to which carbon can ordinarily be subjected, without oxidizing the carbon.

While any form of carbon may be employed on the surfaces contacting with the caustic liquids, I have found that graphitic carbon, particularly when in dense or compacted form is preferable and capable of withstanding higher temperatures than other forms of carbon. Ordinarily in the commercial production of solid caustics, the pots or containers in which water is evaporated from the caustic solutions are of large size, sufficient to contain at least several tons of caustic solution, and heat is applied to the outer wall of the pot by means of oil burners, coal fires or the like. No pots or containers of this size could at present be made of carbon and the heat could, of course, not be applied to the exterior surfaces of such pots, since this would destroy them. Consequently, it is desirable for large production to provide an apparatus having a heating chamber through which a relatively small stream of caustic solution passes, and in which the heat necessary for dehydration is applied to the interior of the apparatus. In the accompanying drawings, I have, consequently, shown a preferred form of apparatus of the latter type, it being understood, however, that it is not intended to limit this invention to use in connection with the particular apparatus disclosed, since obviously my process may be carried on in any type of evaporating apparatus having a chamber so constructed that caustic liquid contacts therein with carbon surfaces only, and in which heat is applied directly within the chamber, and in which air is to a large extent excluded from the interior of that part of the chamber in which the caustic liquid is contained.

In the accompanying drawings:

Fig. 1 is a central sectional elevation of one form of dehydrating apparatus, by means of which my improved process may be carried out, the section being taken approximately on line 1—1, Fig. 2.

Fig. 2 is a sectional plan view thereof, on an enlarged scale on line 2—2, Fig. 1.

Fig. 3 is a fragmentary sectional elevation thereof, showing in section some of the trays for the liquid which is being dehydrated.

In the apparatus shown in the drawings, there

is provided a heating chamber or tower through which liquid to be dehydrated passes from the top to the bottom in contact with carbon surfaces only, and means are provided for introducing heat into the chamber, for example, by admitting a heated non-oxidizing gas or vapor near the bottom of the tower, from which heat is transferred to the liquid to cause water to be evaporated therefrom. For example, superheated steam may be employed for removing water from the solution, and preferably a pressure slightly in excess of atmospheric is maintained within the chamber so that air is excluded.

The apparatus shown in the drawings includes an upright column or tower 10, the walls of which are made of staves or blocks 11 of carbon or graphite, the staves shown being of greater length than width and having their side faces provided with longitudinally extending recesses in which suitable caulking material 12 may be inserted so as to produce a liquid tight joint between adjacent staves 11. Since the tower 10 is higher than the staves, two or more series of these staves 11 are preferably employed, one on top of the other, and the joints between the two tiers of staves are preferably of the stepped or overlapping type as shown at 13, so that liquid would have to flow upwardly in order to pass out of the tower at these joints.

The staves 11 of the tower may be held in correct relation to each other in any suitable manner, for example, by means of bands or hoops 14, those shown being made of two halves connected by means of bolts 15 passing through suitable brackets secured near adjacent ends of the half hoops. Springs 16 acting on the bolts tend to contract the hoops to clamp the staves 11 together. The bolts may have suitable nuts, by means of which the tension of the springs may be varied as desired. The hoops or bands 14 may engage the blocks or staves 11 directly, or if desired, channel bars 17 may be provided, as shown in Fig. 2, which have their flanges engage portions of adjacent blocks. The hoops, consequently, engage the channels 17 and press them toward the center of the tower and into engagement with adjacent staves. Any other means for constructing the tower may, of course, be employed, if desired.

The lowest tier of staves 11 rests upon a base block 18 suitably supported on a steel plate 19, which in turn is suitably mounted by means of frame members 20 on upright columns or pillars 21. The base block 18 has a central recess in which the liquid will collect after passing down through the interior of the tower 10, and this recess terminates in a pan-shaped lower block 22, which has a discharge opening 23 therein which may connect with a discharge pipe 24 through which the liquid end product may be discharged from the apparatus. The block 22 may be supported from a plate 26 suitably suspended from the plate 19, and a smaller block 28 having an aperture in registration with the aperture 23 and to which the pipe connection 24 is secured, may be supported in correct relation to the block 22 by means of a plate 29 suspended from the plate 26. Any other means for withdrawing the finished material from the apparatus may be provided, if desired.

The block 18 preferably is also provided with a series of inlet passages for the heating medium, and in the construction shown, four pipes 30 extend into downwardly inclined apertures 31

formed in the block 18 and terminating in the hollow middle portion thereof. In the construction shown, the gaseous heating medium is conducted to the radially extending pipes 30 by means of a manifold pipe 33 which connects by means of a pipe 34 with a suitable heating coil 35 arranged within a heater 36. A supply of the gaseous heating medium passes into the coils 35 of the heater through an inlet pipe 37 controlled by a valve 38.

Any suitable or desired heating medium may, of course, be used in the column 10. The heating medium may be any non-oxidizing gas or vapor which does not react with the caustic solutions or the carbon material, of which the column is constructed, and preferably I employ superheated steam. Steam or other vapor or gas passes into the heater 36 from the pipe 37 and is raised therein to a high temperature by means of any suitable source of heat. For example, an oil burner indicated diagrammatically at 39 may be used. In order to provide for efficient operation of the apparatus, it is desirable to heat the steam or other medium to a fairly high temperature, and this temperature should be high enough to maintain anhydrous caustic in molten or liquid condition, so that it can readily be discharged through the pipe 24. For example, temperatures approximating from 500° to 800° F. are preferably employed.

Liquid to be dehydrated is conducted into the tower or chamber 10 in any suitable or desired manner, an inlet pipe or duct 40 being shown more or less diagrammatically in Fig. 1, and a suitable valve 41 is preferably employed for controlling the flow of liquid to the apparatus.

Any suitable means may be provided for causing the liquid to flow slowly through the apparatus so that ample opportunity is provided for removal of moisture therefrom. Any suitable or desired checker work of carbon or graphite may be employed for this purpose, and by way of example, I have illustrated in the drawings a series of pans 44 and 45 which are alternately arranged so that the liquid to be acted upon cascades from one pan to another until it reaches the bottom of the tower. The pans are also made of carbon and may be supported in any suitable or desired manner, this being preferably done by means of rows or tiers of blocks 46, four such rows being shown in the construction illustrated, see Fig. 2. The pans 44 are annular in shape and of larger outside diameter than the pans 45 and preferably have inner flanges 47 and outer flanges 48, and if desired, the outer flanges may be slightly greater in height so that liquid will tend to overflow the inner flanges 47 of the pans 44 and thus discharge into the next lower pans 45, having outer flanges 49. By means of the blocks 46 as shown, the pans are so supported that when a pan 48 overflows, the liquid will drop upon the next lower annular pan 44. It will also be noted that the arrangement of these pans is such that the circular pans 45 prevent the passage of steam or other gaseous heating medium directly upwardly through the middle portion of the tower, and the annular pans 44 of larger diameter approach so closely to the inner wall of the column 10 as to permit only a small part of the heating medium to flow between these annular pans and the inner wall of the column 10. This arrangement of the pans, consequently, cause the greater portion of the gases to flow through the middle opening of an annular pan 44 and then outwardly around the outer edge of the next higher circular pan 45 and

back through the central opening of the next higher annular pan. In this way, a greater portion of the heating medium will be forced to flow crosswise of the cascading liquid and also most of the heating medium will contact with all of the pans so as to impart a high degree of heat to the same.

The apparatus is arranged on the counterflow principle so that the hottest heating medium or superheated steam acts on the most concentrated solution near the bottom of the tower, while the gaseous heating medium at the top of the tower acts on the relatively cool liquid which has just entered the tower. For most efficient operation of the apparatus, it is, of course, desirable to supply liquid to the apparatus through the pipe 40 at a rate just enough to enable the desired amount of water to be removed from the caustic liquid while passing through the tower. If an anhydrous caustic is desired, this can readily be obtained by the proper regulation of the flow of liquid and gaseous heating medium passing to the tower. Any degree of concentration of the caustic liquid can, of course, be obtained by the proper regulation of the quantity of caustic liquid and heating fluid admitted to the apparatus.

The top of the tower 10 may be closed in any suitable or desired manner. In the construction shown for this purpose, I provide a dome or top including an annular outer wall 50 and a top wall or lid 51. A discharge passage 52, for the spent heating medium and steam resulting from dehydration of the caustic solution is provided in the annular wall 50, and an annular baffle wall 53 is preferably provided within the dome and which rests upon a supporting wall 54. The baffle wall 53 has an opening through which steam from the top of the tower may enter the annular space between the walls 50 and 53 and finally pass out through the pipe 52.

In order to exclude air or other oxidizing medium from the dehydrating chamber or tower 10, a discharge valve 56 may be provided in the discharge passage 52 and the opening in the discharge valve may be readily adjusted so as to maintain a slight pressure within the chamber 10.

Preferably the tower is also insulated to retard the escape of heat from the same, and consequently, the tower may be surrounded by any suitable insulating wall 58, which is arranged about the column 10 in spaced relation thereto.

60 represents an annular member or band of carbon which extends about the block 18 and which has its upper end shaped to form a trough 61 in which any caustic liquid may be collected which has seeped through the wall of the tower or chamber 10. From this trough, which may obviously be formed as a part of the block 18, liquid may flow back into the tower through the lap joint which connects the block 18 with the lowest set of staves 11.

The finished material withdrawn from the discharge pipe may, of course, be led into suitable containers in which it may cool and crystallize.

In operating in accordance with my process, any type of carbon surface may be employed with which the caustic liquid may contact. In order to provide for the most efficient operation and for utilizing the highest possible temperatures, it is preferable to employ graphite blocks of a compact and dense nature and relatively impervious to caustic solution. In using such graphite blocks, temperatures as high as 800° F. and even somewhat higher may be employed in connection with my dehydration process, providing that air

or other oxidizing medium is substantially excluded. It is well known that a temperature of about 800° F., carbon oxidizes rapidly and the oxidation of carbon in contact with caustic solutions would, consequently, result in the production of carbonates of potassium or sodium. If air is entirely excluded from the process, both the oxidation and deterioration of the carbon at the high temperatures is prevented, as well as the production of carbonates in the caustic material.

One of the advantages of my process is that by means of the same, caustic solutions can be dehydrated without the introduction into the same of any metallic impurities, and this can be done without increased cost in producing the dehydrated materials. While it is preferable to operate my process with air entirely excluded from the chamber, yet the presence of small quantities of air will, of course, have only a very slight deteriorating effect upon the material of which the column is constructed and the amount of carbonates thus produced in the product will be practically negligible. However, even this slight degree of contamination of the caustic and oxidation of carbon can readily be avoided by maintaining a slight pressure of steam or heating medium in the tower 10. The process also has the advantage that once it is started, it requires very little attention, except to maintain an adequate supply of caustic solution and of steam or other heating medium.

I claim as my invention:

1. A process of producing caustic solutions of high concentration and substantially free from metallic impurities, which process includes passing a stream of the solution to be evaporated and which is substantially free from metallic impurities through a substantially closed container from which air is substantially excluded and in contact only with carbon surfaces, supplying heat to said solution to evaporate moisture therefrom and to maintain said solution in liquid form, and withdrawing the remaining solution from said container while the same is in liquid form.

2. A process of dehydrating caustic solutions, without adding metallic impurities thereto, which includes admitting a caustic solution to be dehydrated into one end of a chamber into contact only with graphitized carbon surfaces, admitting to said chamber a non-oxidizing gaseous heating medium elevated to a temperature in excess of the fusing point of the dehydrated caustic and into contact with said caustic solution to remove moisture therefrom by evaporation, and discharging the heating medium together with vaporized water from said chamber near that end thereof at which said solution is admitted.

3. A process of dehydrating caustic solutions, without adding metallic impurities thereto, which process includes passing a stream of said solution into a substantially closed chamber into contact only with graphitized carbon surfaces, passing through said chamber in contact with said solution a current of a gaseous, non-oxidizing heating medium raised to a temperature of approximately from 500° F. to 800° F., and excluding air from said chamber.

4. A process of dehydrating caustic solutions, without adding metallic impurities thereto, which process includes passing a stream of said solution into the one end of a substantially closed chamber into contact only with graphitized carbon surfaces, admitting to the other end of said chamber into contact with said solution steam

superheated to temperatures from approximately 500° F. to 800° F., and discharging steam from the first mentioned end of said chamber.

5. A process of dehydrating caustic solutions, without adding metallic impurities thereto, which includes continuously passing a stream of caustic solution to be dehydrated into the upper portion of a closed chamber lined with graphitized carbon, cascading the solution through said chamber and while in contact only with graphitized carbon surfaces and non-oxidizing gas, heating the solution in said chamber above the fusing point of the caustic solutions to vaporize water therefrom and to maintain said solution liquid, and discharging vapor from said chamber.

6. A continuous process of dehydrating caustic solutions, without adding metallic impurities thereto, which includes continuously passing a stream of caustic solution to be dehydrated into the upper portion of a closed chamber lined with graphitized carbon, cascading the solution through said chamber and while in contact only with graphitized carbon surfaces, excluding air from said chamber, heating the liquid to a temperature above the boiling point of water while passing through said chamber to vaporize water contained in said solution, and discharging the vaporized water from one end of said chamber and dehydrated caustic in molten form from the other end thereof.

7. A process of dehydrating caustic solutions, without adding metallic impurities thereto, which

includes continuously passing a stream of caustic solution to be dehydrated into the upper portion of a closed chamber lined with graphitized carbon, cascading the solution through said chamber and while in contact only with graphitized carbon surfaces and non-oxidizing gas, heating the liquid in said chamber to a temperature above the boiling point of water to vaporize water therefrom, maintaining in said chamber a non-oxidizing gas at a pressure in excess of that of the atmosphere to exclude air from said chamber, discharging water vapor from the upper end of said chamber, and withdrawing dehydrated caustic in liquid form from the lower end of said chamber.

8. A process of dehydrating caustic solutions without adding metallic impurities thereto, which includes passing a stream of caustic solution to be dehydrated into a closed chamber into contact only with carbon surfaces, and bringing a current of heated non-oxidizing gas which is inert with reference to carbon and said caustic solution into contact with said solution to evaporate water therefrom.

9. A process according to claim 8 and including the step of excluding air from said chamber.

10. A process according to claim 8 and including the step of maintaining said gas under pressure to exclude air from said chamber.

11. A process according to claim 8 in which the gas is superheated steam.

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