

(No Model.)

5 Sheets—Sheet 1.

A. KAYSER.

MANUFACTURE OF CAUSTIC ALKALI, &c.

No. 454,136.

Patented June 16, 1891.

Fig. 1



Fig. 8

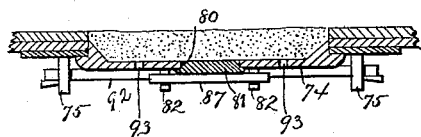


Fig. 9

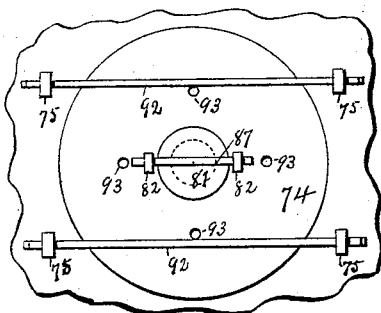
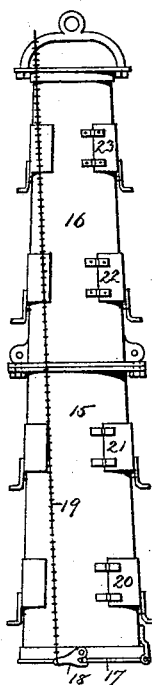


Fig. 7



Witnesses

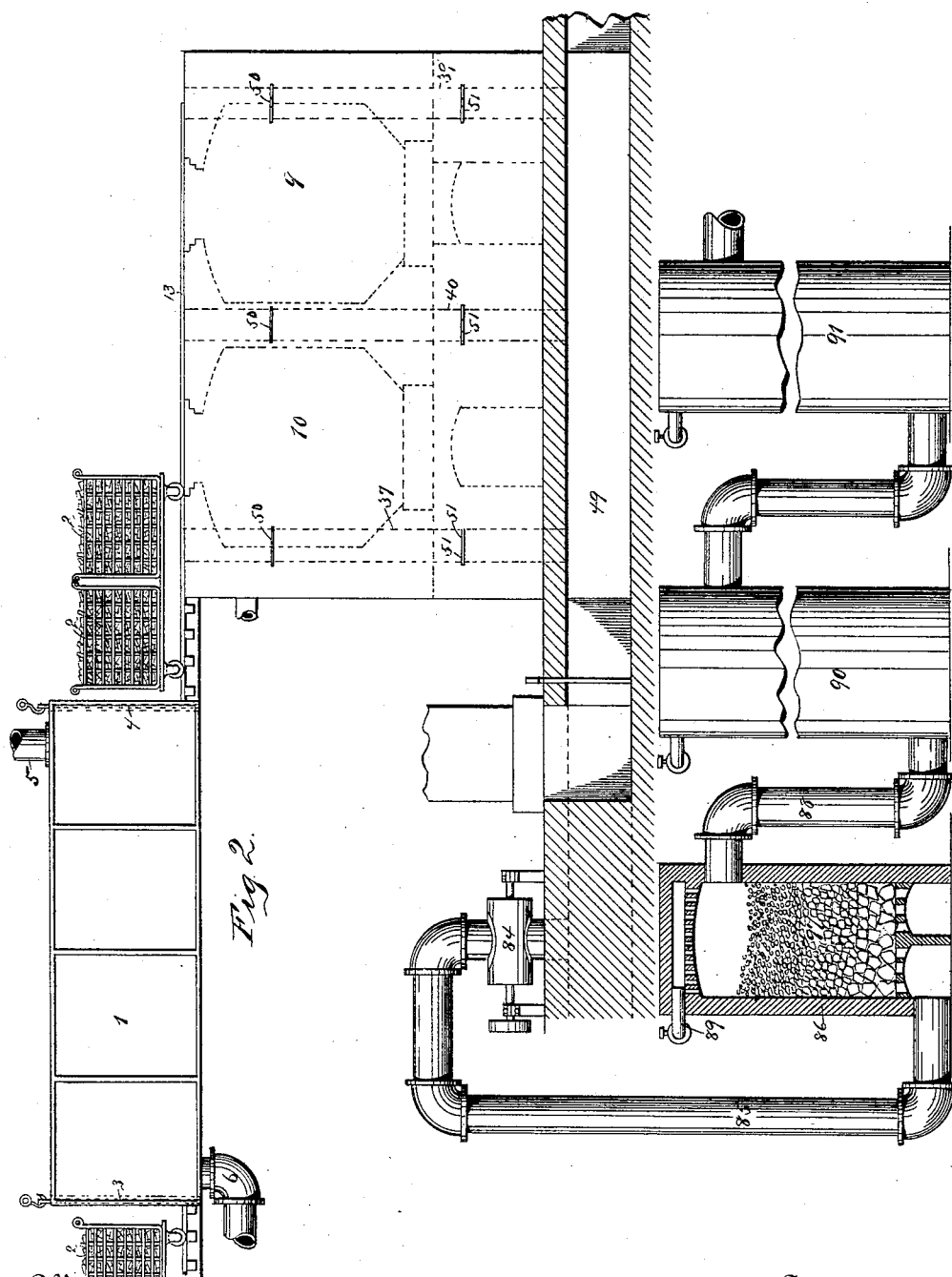
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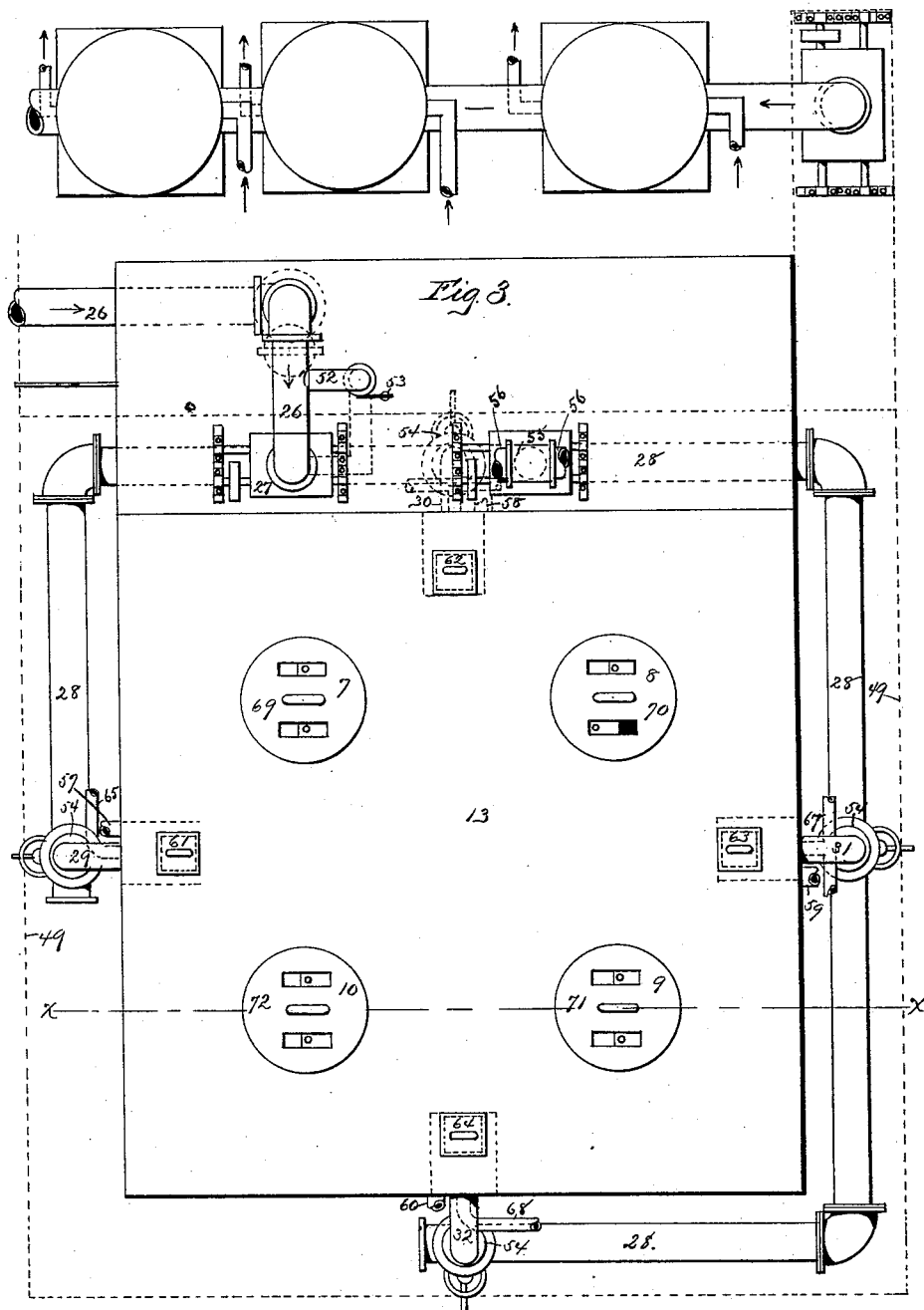
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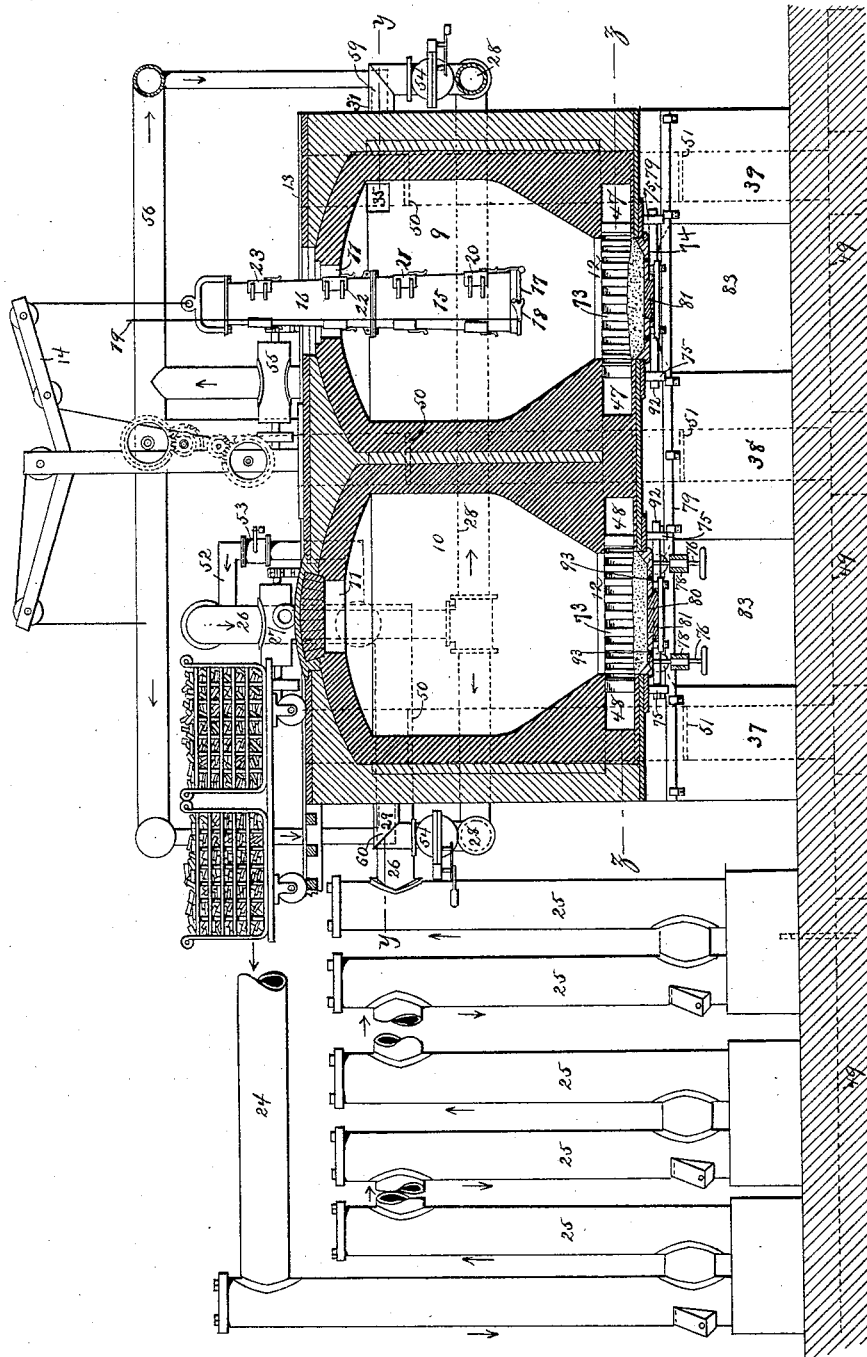
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Fig. 4.



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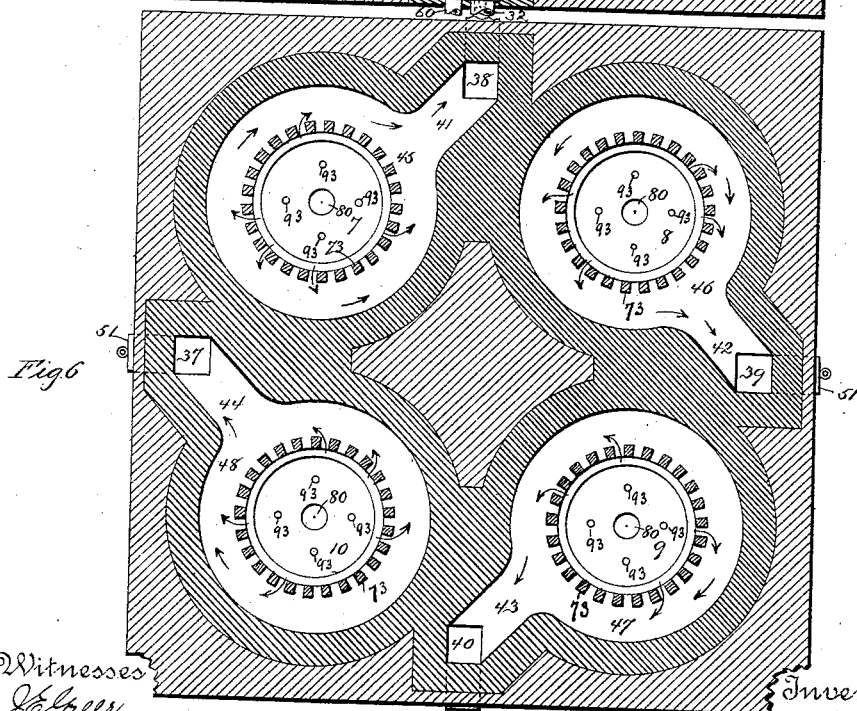
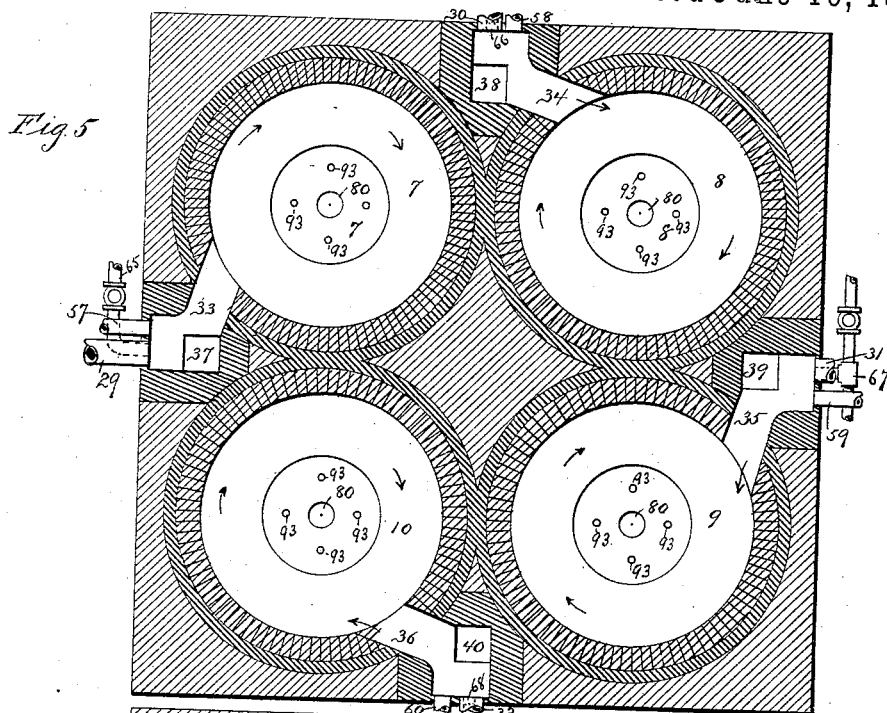
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# UNITED STATES PATENT OFFICE.

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## MANUFACTURE OF CAUSTIC ALKALI, &c.

SPECIFICATION forming part of Letters Patent No. 454,136, dated June 16, 1891.

Application filed October 20, 1890. Serial No. 368,644. (No model.)

*To all whom it may concern:*

Be it known that I, ADOLF KAYSER, a citizen of the United States, residing at Buffalo, county of Erie, and State of New York, have  
5 invented a new and useful Improvement in the Manufacture of Caustic Alkali, Carbonates of the Alkaline Metals, and Muriatic Acid, of which the following is a specification.

My present invention is the result of developing and improving the process and apparatus described in United States Letters  
10 Patent granted to me January 10, 1888, and numbered 376,409.

In the drawings, Figure 1 represents a cake  
15 of material with which my process starts. Fig. 2 is an elevation of the drying apparatus and the converting-furnace and apparatus for disposing of the muriatic-acid gas. Fig. 3 is a plan view of the converting-furnace  
20 and various passages connected therewith. Fig. 4 is a vertical section through the line *x x* of Fig. 3. Fig. 5 is a horizontal section through the line *yy* of Fig. 4. Fig. 6 is a horizontal section through the line *z z*, Fig. 4.  
25 Figs. 7, 8, and 9 are details.

The material with which the process starts consists of cakes, preferably of the form shown in Fig. 1, being cylindrical, with a central hole or channel. These cakes are  
30 composed of a pasty compound of salt, alumina, and silica, preferably in proportions of about one and a half pound of silica to one pound of alumina, with enough salt to produce upon conversion from thirty-one to thirty-three  
35 per cent. of  $\text{Na}_2\text{O}$ . I obtain the required silica and alumina by making use of clay; but as it is difficult, if not impossible, to obtain a clay which in the natural state contains the silica and alumina in the desired proportions I ordinarily procure a strong aluminous clay and a sandy or silicious clay and mix the two in such proportions as will give the desired composition. For a more detailed  
40 description of these cakes reference is made to an application filed of even date herewith, serially numbered 368,643. These cakes are first dried at a temperature gradually raised to from  $220^\circ$  to  $250^\circ$  Fahrenheit, which is accomplished by the drier shown in Fig. 2.

50 1 is a furnace, into which trucks carrying the baskets 2 are run at one end and out at

the other end. Doors 3 4 are provided at each end, so that the furnace can be closed, and the hot air is introduced by the flue 5 and taken away by the flue 6. A series of  
55 trucks is employed, so that a batch of the cakes can be introduced at the receiving end 3 as fast as a previous batch is finished and removed at the delivery end 4, which will occur about every hour. The temperature of  
60 this drier shall at the hot end be not less than  $220^\circ$  Fahrenheit, but may be increased to  $250^\circ$ . The hot air travels from the delivery end to the receiving end, and thereby the cakes first introduced are gradually heated.  
65 It is important that the cakes should be gradually heated to avoid cracking. The effect of this drying is to remove the mechanically-held water from the cakes and harden them sufficiently to withstand the subsequent handling.  
70

The next step in the process is to introduce them into one of the converters 7, 8, 9, or 10, each of which converters is cylindrical near the top and sufficiently conical toward the  
75 bottom to allow the whole charge to roll out and at the same time direct the gases throughout all the lower part of the mass. Each converter has a charge-opening 11 at the top and a discharge-opening 12 at the bottom. The  
80 cakes should not be dumped roughly into the converter, but should be placed therein gently to avoid breaking and pulverizing, since an important feature of the process is the maintaining of the individual cakes forming the  
85 contents of the converter intact, which, owing to their size and shape, will maintain passages for the entrance of the gases to all parts of the mass, providing these passages are not filled up by dust or fine particles resulting  
90 from the crumbling of the cakes. I therefore, in charging the converter, proceed as follows: The cakes are dumped upon the floor 13 above the converters, and the contrivance shown in Fig. 7 is utilized. This contrivance  
95 consists of two cylindrical or slightly-conical sections 15 and 16. The section 15 is provided with a trap-door 17, closing the bottom opening and held in place by a latch 18, operated from above by the chain 19. The section is also provided with the side doors 20  
100 and 21 at different elevations, and at the top

is provided with means of attachment to section 16, which latter section is also provided with two side doors 22 and 23 at different elevations. The means of attachment or connection between the sections may be of any suitable construction. As shown, they consist, simply, of bolts 16<sup>a</sup>, hinged to the top section and arranged to swing into notches in the top flange of the bottom section, their ends being provided with nuts 16<sup>b</sup>. Detached from the section 16, the section 15 is first suspended in the charge-opening of the converter, as 9, by the yoke 16<sup>c</sup>, or otherwise, from the crane 14, so that the door 20 is at convenient height for the introduction of cakes, and the cakes are introduced through the door 20 to as high a level as possible. Then the section 15 is lowered until the door 21 is at a convenient height above the floor 13, and as many more cakes are introduced as possible. Then the section 15 is again lowered until its top portion is at a convenient height and completely filled with cakes. The section 15 is then sustained temporarily by bars laid beneath its top flange and resting on top of the retort, and yoke 16<sup>c</sup> is attached to the top section 16. Then the section 16 is placed on top of and secured to the section 15, and the like operation is repeated through the doors 22 23 of section 16, thus filling the two sections nearly to the top, by which time the bottom of the section 15 will have descended to near the bottom of the converter, and thereupon the door 17 is released from the latch 18 and the cakes allowed to slide out at the bottom of the converter. Then the contrivance is raised, and as each successive door comes at a convenient height above the floor 13 it is again filled with cakes up to that level. When the contrivance has been lifted entirely out of the converter, the converter will be so near full that the cakes necessary to complete the charge may be introduced directly from above. When the charging is complete, the top level of the charge will be below the level of the entrance of the heating or converting gases. It will be convenient to next describe the provision for heating the converters and removing the products therefrom. Fuel-gas is brought from a generator through the pipe 24 and the cooler 25 and pipe 26 to the gas-forcing apparatus 27, whence it is forced into the pipe 28, running nearly all around the furnace. The cooling is simply to protect the gas-forcing apparatus from undue heat. The branches 29, 30, 31, and 32 lead from this pipe, respectively, to passages 33, 34, 35, and 36, which communicate with the top of the respective converters at angles nearly tangential to the peripheries thereof. The introduction of the converting-gases into the vacant space above the top of the charge and in a tangential direction is advantageous, in that the gases are given a circulatory motion, thoroughly mixed, and distributed around the circumference of the converter-chamber, so that in sinking they permeate all portions of

the charge, and thus effect a thorough and uniform heating of the same. In view of the fact that the converter-chamber is usually of a diameter of ten feet and upward and that the entire charge must be maintained at a white heat to secure a thorough conversion and prevent the choking of the gas-passages by the deposition of salt therein, the importance of effecting the distribution of all gases above the charge will be readily appreciated. From the passages 33, 34, 35, and 36, respectively, vertical passages 37, 38, 39, and 40 extend downward through the walls of the furnace until they communicate, respectively, by the passages 41, 42, 43, and 44 with the circular passages 45, 46, 47, and 48, surrounding the bottom of the several converters and receiving the gas discharged therefrom. Then the passages 37, 38, 39, and 40 extend still farther down until they all connect with an underground passage 49. They are each provided with a damper 50 between the passage, as 33, connected with the top of one converter, and the passage, as 44, connected with the bottom of the next converter. Each is also provided with a damper 51 between the last-named passage and the underground passage.

52 is a by-pass pipe extending around the gas-forcing apparatus 27 and provided with a weight-controlled valve 53, by the adjustment of which the pressure of gas supplied to the converters can be controlled. A valve is placed in each of the pipes 29, 30, 31, and 32 at 54, so that the supply of fuel can be cut off from either of the converters, as desired.

55 is a fan or air-forcing apparatus which forces air into the pipe 56, (shown broken off in Fig. 3,) extending around above the pipe 28 and provided with branch pipes leading to the pipes 57, 58, 59, and 60, which discharge the air into the passages 33, 34, 35, and 36, respectively, where it mingles with the gas discharged therein, as already described.

61, 62, 63, and 64 are covers for holes leading from the floor above the converters down into the passages 37, 38, 39, and 40, respectively.

65, 66, 67, and 68 are steam-pipes discharging steam into the passages 33, 34, 35, and 36 to mingle with the air and gas therein. The converters are all lined with fire-bricks, as are also the covers of the charge-openings 69, 70, 71, and 72. It will be observed that each of the vertical passages 37, 38, 39, and 40 connects at its bottom with one adjacent converter and at its top with the other, so that by the proper arrangement of the dampers the bottom of each converter may be connected with the top of its neighbor throughout the whole series; or by another arrangement of dampers this connection may be broken and the bottom of any one of the converters may be connected directly with the underground passage. The passage around the lower end of each converter, as the passage 45, is communicated with from the interior of the converter through a series of

radial openings between the separated fire-bricks 73, thus constituting a perforated ring between the interior of the converter at the bottom and the passage, as 45, surrounding the same.

74 is a plate closing the discharge-opening of the converter, being held in place by the hangers 75 and the cross-bars 92, and also being supported on the points of screws 76, (shown beneath converter 10, Fig. 4,) which are mounted in the cross-bars 78, adapted to slide, as a carriage, on the guideways 79, and thus form a ready means for the removal of the heavy plate to one side for the discharge of the converter. The plate 74 is provided with tap-holes 93 and with a central opening 80, covered by a supplemental plate 81, held in position by suitable hangers, as 82, and the cross-piece 87. 83 are archways below the converters, into which their contents are discharged and from which they are removed for subsequent operations.

The manner in which the converters are operated may be described as follows: The product of the combustion of the fuel-gas employed must be slightly oxidizing, in order to avoid any loss of sodium by reduction—an action which becomes more likely the more basic the cinder. This is within the control of the operator by the devices already described, enabling him to regulate the relative quantity of gas and air employed. It is very important to have a fuel-gas of the highest pyrometrical effect. Common generator (producer) gas, containing a large percentage of nitrogen, is necessarily of a limited pyrometrical effect. Heating the air used for the combustion will help a great deal in elevating the temperature; but it is better yet to use natural gas, where it can be had at reasonable figures, or to use water-gas alone or mixed with generator-gases. Four converters are combined in a set. The arrangement of the flues, as described, is such that each of them may be worked independently or with the following in order. The converter 7 having been charged, the proper proportions of gas, air, and steam are introduced into the passage 33 by opening the suitable valves and the gas therein ignited through an opening in the floor above. The flame and products of this combustion enter the vacant space in the top of the converter-chamber in a tangential direction and with considerable velocity, this tangential admission of the gases being followed by the uniform heating of the entire charge to a white heat, for the reasons already pointed out. During the treatment of the charge in the converter the opening in the bottom plate enables the operator to clear away any obstructions which may block the gasways at the bottom, and tap-holes two inches in diameter are provided to drain away any salt previously condensed which may descend as a liquid to the bottom of the converter, and which, if allowed to remain, would solidify there and in the flues, and

thus choke up the passages for the escape of the gas and create a solid mass in the bottom of the converter, which upon cooling would entirely prevent the discharge of the contents of the converter and render the process almost inoperative. These tap-holes are used successively, and the melted liquid salt is therefore discharged several times during the preparation of each charge in the converter. While in use the bottom plate is covered with three or four inches of dust, and the central opening, as well as the tap-holes, is closed. About four days will be required to complete the conversion. When the operation in converter 7 is complete, the bottom plate is removed, and the finished incandescent salt falls through the opening and is removed. As soon as the temperature permits the bottom plate is replaced again, and the converter is ready to be recharged.

Pending the conversion in converter 7 the other converters will be charged successively, one each day, and when in continuous operation they will be worked in series as follows: Suppose the contents of converter 8 to be at a perfect white heat and the operation therein to be almost complete. The material under treatment now produces but little muriatic acid. The highly-incandescent gas from converter 8 therefore is not conducted to the underground flue, but by regulating the dampers 50 and 51 is conducted into the top of converter 9, also at a white heat at the top, but still at a red heat at the bottom, where it mingles with new fuel-gases in order to establish the required temperature, and where the vaporized salt which it contains is utilized and converted, and from this converter 9 the gases of both 8 and 9 descend into the underground flue to be removed by the exhaust. In the meantime converter 10, having been charged only the day before, is worked independently from the others. Though red heat may have been established at the top of its contents, it is comparatively cold below and is not yet fit to receive the gases from converter 9, since the lower and colder strata would condense the salt contained in those gases, and this would obstruct the gasways. On the next day, however, the converter 7 has been charged and set to work, converter 8 has been finished and discharged, converter 9 is at a white heat and sends its gases (hot, but weak in acid) to converter 10, and converter 7 is run independently of the others. Thus each day one converter is charged and one discharged, and the operation is practically continuous, providing no interruption happens in any one of the converters. The principal causes of interruptions which this construction of apparatus and process obviate would arise from a faulty composition of the clay. If the clay contains an excess of silica, the cakes or bricks will, under the high temperature employed, become glazed or fused. If, on the other hand, they contain an excess of alumina, they will disintegrate and



obstruct the passage of the gases. In either case the converting action will be stopped or seriously impeded. Care should be taken to increase the temperature slowly of a newly-filled converter, so as to avoid the volatilization of salt while the cakes at the bottom are comparatively cold. As soon as a red heat is established at the bottom of the converter, however, no further obstruction from this cause will be had, and the temperature is now increased as rapidly as possible to beginning white heat, so as to produce the acid reaction of the silica and alumina. A certain amount of salt sublimed in the bottom of the converter during the earlier part of the process will then melt and will be tapped consecutively through the holes in the bottom plate. I use the series of tapping-holes extending downward through the bottom for the reason that when a hole is tapped it becomes filled with the salt so that it cannot be reopened, thus necessitating the tapping of another hole when it is again required to draw away the melted salt.

It will be observed that my present process differs from that described in my original patent, No. 376,409, in that I now employ in the primary step a compound containing a much higher percentage of alumina, and that instead of employing a red heat I subject the material to a high white heat. The advantages attending the use of the higher temperature are, first, a much more rapid conversion; second, a much richer acid salt; third, the elimination of nearly all the sodium chloride, which was frequently present in the former product to the extent of one and one-half per cent.; fourth, the possibility of using a highly-heated aluminous compound, which allows a considerable increase in the percentage of sodium acid in the acid salt; fifth, the production of a greater and a stronger acid; sixth, a more efficient and economical employment of the fuel; seventh, the uniform reduction of the entire charge in the retort; eighth, the use of a cleaner and more efficient operation, as the higher temperature prevents the precipitation of the sodium chloride in the lower part of the apparatus. The proportion of silica and alumina described in the prior patent are not adapted to withstand the high temperatures now employed. It is by increasing the proportion of alumina that the necessary refractory property is developed without, on the other hand, rendering the charge liable to be fused or disintegrated.

I have at this point of the process two products:

First. Acid salt (sodium silico-aluminate) containing from thirty-one to thirty-three per cent. or more of sodium oxide. It is in cakes or blocks, which crumble but little in handling and stand the crushing action of their own weight. It is refractory at a beginning white heat and requires no silica when smelted into a basic salt.

Second. Muriatic-acid gas containing a percentage of salt-crystals as impurity.

It will be necessary to pursue the subsequent operations on these two products separately, and I will first describe the treatment of the acid salt. The acid salt (sodium silico-aluminate) is converted into the basic salt by the addition of alkali by melting either with raw lye or its equivalent of soda-ash. If raw lye is used, the carbonic acid from the combustion of fuel-gas first transforms the caustic alkali into carbonate until a temperature is reached where carbonic acid is displaced again by the acids silica and alumina and escapes. In my said patent I advised the production of a basic salt so rich in soda as to contain fifty per cent.; but further practical experience has shown that, especially where the object is to make caustic soda, the production of so high a percentage of soda is unwise. The elimination of carbonic acid can only be accomplished when the temperature of the furnace and the percentage of sodium oxide are balanced. To reach this end more readily, I prefer that the basic salt should contain from forty to forty-two per cent. of sodium oxide, because with this percentage a lower temperature answers the purpose, and the operation then acts less destructively on the lining of the furnace than the more highly alkaline cinder. The basic salt resulting from the melting operation is next crushed to a chestnut size and is leached systematically until the lye shows about 1° Baumé. The soluble matter remaining is hydrous sodium silico-aluminate with from seventeen to twenty per cent. of sodium oxide. This is then ground and floated and decomposed by quicklime or carbonic acid. If quicklime is employed for this purpose, the operation is conducted as follows: The ground and floated hydrous sodium silico-aluminate is treated with a slight excess in equivalent of lime, avoiding too large a volume of liquid in relation to the solid matter, which by interfering with contact would prevent speedy action. (I have found a creamy consistency of the material to be the most convenient.) This reaction can be accomplished cold; but I prefer to accelerate it by boiling, which assists at the same time in mixing the ingredients perfectly and also in causing the hydrous calcium silico-aluminate to subside and separate clear from the caustic lye. The speed of the operation depends on the fineness of the grain. The digestion should be carried on until all the soda is displaced by lime. The lye so produced will be found perfectly pure, of a gravity from 4° to 5° Baumé. It is conducted back to the leaching-tanks and there used for leaching a fresh supply of basic salt. This lye is preferably separated from the residue (hydrous calcium silico-aluminate) in a filter-press, which is provided with a washing arrangement to perfectly exhaust it. Employing a pressure of seventy-five pounds, cakes are produced which are found

to contain about forty per cent. of water. The same material leached in filter-tanks would contain on an average eighty per cent. of water and is very difficult to handle or dry.

5 This residue upon exposure to the air is decomposed by the carbonic acid of the air, producing a body composed of calcium, carbonate of silica, and alumina in the finest division. This body is, I am confident, an entirely new  
10 article and is capable of many practical applications. Being extremely fine, it can be used with advantage as a base for a glaze, as a flux with kaolin for stoneware, and again for hydraulic cement. It is eminently fitted as a  
15 substitute for infusorial earth. It is remarkably pulverulent, and is furthermore distinguished by containing a very large percentage of chemically-bound water when air-dried—say thirty per cent.

20 As a modification, it may be found convenient to reduce the basic salt without previous leaching and treat it with the required amount of lime, &c. By so doing the separated lye will be found pure and free from silica. The  
25 tankage for the leaching of the crushed basic salt is done away with, while more power is required for the grinding. If carbonic acid is employed for decomposing the hydrous sodium silico-aluminate, it (in aqueous solution)  
30 acts instantaneously upon this substance, if finely divided, forming a solution of sodium carbonate. The residual regenerated clay has all the qualities mentioned in my said Letters Patent, and may be used as therein  
35 indicated.

I will next describe the treatment of the muriatic-acid gas after its discharge from the converters into the underground passage 49, as already described. Great difficulties were  
40 encountered in eliminating the salt from the acid gases produced in the converters, the presence of which salt is due to the elevated temperature employed; but I have discovered that such gas can be purified by passing  
45 it through small channels, where it coats the walls with salt-like frost, and to utilize this discovery the gas which is exhausted by the fan 84 is forced through the pipe 85 into a small tower 86, the area of which is dependent upon the volume of gas to be sent through.  
50 This tower is filled with a substance 97, presenting condensing-surfaces—such as coke—through which the gas disseminates and upon which it deposits and leaves the salt, passing  
55 out into the passage 88 in a purified condition. 89 is a water-pipe which is employed when the tower is not in use to wash out the salt which has been deposited. From the pipe 88 the gas is passed through a series of coke-towers 90 91, wherein it is caused to be condensed in a well-known manner.

In lieu of sodium chloride, with which my process starts, I may employ as an equivalent any of the chlorides of the alkaline metals.

65 The treatment of the silico-aluminates dissociated from the process of their production is made the subject of a separate application

for Letters Patent, filed December 5, 1890, Serial No. 373,703, and is therefore not separately claimed herein.

I claim—

1. The herein-described method of producing an alkaline oxide, which consists in mixing the chloride with clay containing silica and alumina in approximately the proportion of one and one-half pound of silica to one pound of alumina and heating the mixture to a white heat in a converter by passing highly-heated gases containing steam through the converter, substantially as described, whereby the conversion is accomplished without materially fusing or pulverizing the charge.

2. The herein-described method of treating an alkaline chloride, which consists in heating a mixture of the chloride with clay by passing highly-heated gases of combustion and steam through the same, then combining with the resulting gases additional highly-heated gases, and applying this combination directly to heat a second mixture of the chloride with clay, whereby economy of heat is secured and the vaporized salt resulting from the first operation carried over and decomposed by the second operation.

3. The herein-described method of treating an alkaline chloride, which consists in heating a mixture of the chloride with clay by the direct action of highly-heated gases, thereby producing an acid silico-aluminate of the alkali, then melting the same with an alkali, and decomposing the basic salt thus produced, substantially as described.

4. The herein-described method of treating an alkaline chloride, which consists in heating a mixture of the chloride with clay by the direct action of highly-heated gases, then melting the product with an alkali, then floating the same in a finely-divided state, and then decomposing, substantially as described.

5. The herein-described method of treating an alkaline chloride, which consists in heating a mixture of the chloride with clay by the direct action of highly-heated gases, then melting with an alkali, then decomposing, and then filtering the product of the decomposition, substantially as described.

6. The herein-described method of treating an alkaline chloride, which consists in heating the mixture of the chloride with clay by the direct action of highly-heated gases, then melting with an alkali, then leaching, then grinding the residue, and then decomposing, so as to release the alkali, substantially as described.

7. In an apparatus for treating alkaline chloride, a converter provided with a gas-inlet at the top and with a series of circumferential gas-outlets at the base, whereby the distribution of the gas and the uniform heating of the charge are secured.

8. In an apparatus for treating alkaline chloride, a converter provided with a tangential gas-inlet at the top and a series of cir-

cumferential gas-outlets at the base, substantially as and for the purpose described.

9. In an apparatus for treating alkaline chloride, a converter having at the base circumferential gas-outlets and tap-holes extending down through the bottom.

10. In an apparatus for treating alkaline chloride, a converter having a removable bottom plate covering the entire lower end and a supplemental cover closing an opening in the bottom plate, whereby the entire contents may be discharged or the main contents retained and the removal of small obstructions permitted in order to open a passage for the gases.

11. In an apparatus for treating alkaline chloride, a converter provided at the top with an inlet-passage, in combination with a steam-pipe, air-pipe, and fuel-gas pipe, all discharg-

ing into said passage in advance of the re-tort, whereby the gases are mingled and ignited before entering the converter.

12. In an apparatus for treating alkaline chloride, in combination, a series of converters, a passage to receive the generated gases from all, an exit-passage provided with a damper from each converter leading thereto, and a passage provided with a damper leading from the last-named passage to the adjacent converter, whereby the generated gases from each converter may be discharged either into the first-named passage or into the adjacent converter, substantially as described.

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