ern Central Africa, whence its fruits are exported by way of Abyssinia and Massowah.

ROOT OF STATICE LATIFOLIA Sm.—Among the few drugs sent from Russia to the Exhibition, are specimens of the thick woody root of Statice latifolia Sm. (St. coriaria Pall.), which is remarkable for its compact substance and large size, so unlike what is usual in other species of Statice; this root I have recognized to be identical with that imported some years ago into Marseilles, and described by M. Guibourt in his 'Histoire des Drogues,' tome 2, p. 416. With respect to its production and uses, I have been favoured with the following remarks, by Mr. George Peterson, the Russian Commissioner of the Exhibition, who, as a member of the Scientific Committee for the Crown Lands of Russia, has the best opportunity for being correctly informed upon the subject. The "Statice coriaria of Pallas," says he, "grows wild in the whole prairie country of Southern Russia, known under the name of steppes, that is, land without forests. I have myself during two years travelled through this region, and seen roots of it of the length of more than thirty feet and with a top diameter of four and a half inches. The plant is also found in the north of the Crimea and in the southern part of Bessarabia, but in general it is more common in the eastern part of the prairie region near the Wolga, and to the east of this river. To dig out the roots is very difficult, because the subsoil is hard and cannot be worked with a pickaxe. The root penetrates the soil and subsoil in a nearly perpendicular line; the longest roots are discovered on steep river-banks, which are underwashed each springtime by the swollen rivulets and also after heavy rains, the latter occurring very seldom. A small number of tanners have begun to employ the roots of Statice, but as the supply is precarious and the leather tanned with them brittle, no progress is made in utilizing this tanning material, which in more skilful hands has proved one of the best, as, for instance, in Spain."

# ON THE MANUFACTURE OF CAUSTIC SODA.

#### BY A. NORMAN TATE, F.C.S.

## (Read at the Meeting of the Liverpool Chemists' Association held May 15th, 1862.)

The manufacture of Caustic Soda, although at present quite in its infancy, will, I believe, soon become one of very great importance. To a great extent the use of caustic soda will supersede that of soda-ash, especially for soap making, as it will not only lessen the amount of labour, but will also materially reduce the amount of apparatus required, thereby considerably lessening the cost of plant, and also economizing space—two very important considerations to the manufacturer.

I am well aware that, as was the case with soda-ash, there are some difficulties to be surmounted before caustic soda becomes so generally used for soapmaking and other purposes as soda-ash now is, and not the least of these is the prejudice of manufacturers. In the case of soda-ash the prejudice against its use was at first so great, that Mr. J. Muspratt had to give away tons of it, before he could get the soap-makers to employ it instead of the barilla, or kelp, which they were in the habit of using before the introduction of artificial soda.

I do not however anticipate quite so great an opposition to caustic soda, for, thanks to the diffusion of scientific knowledge, the manufacturers of the present day are to a great extent a different class of men to those of only a few years since, and we can now point to many of them who are eminent for their scientific attainments; and these scientific manufacturers—men who

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combine a knowledge of science with practical skill—cannot fail to see the great advantage caustic soda has over soda-ash, and it will therefore come rapidly into use; indeed the consumption of this article increases daily.

rapidly into use; indeed the consumption of this article increases daily. *Processes for Manufacturing Caustic Soda.*—There are at present two or three processes employed for the production of caustic soda, and I shall now proceed to describe them.

In the manufacture of soda-ash, or carbonate of soda, by Leblanc's process, there is found in the liquors produced by lixiviating the black ash a certain amount of caustic soda, or hydrate of soda, mixed principally with carbonate of soda. In evaporating these liquors the carbonate of soda separates, and there remains a liquor containing nearly all the hydrate of soda. This liquor is technically known as "red liquor," owing to its being of a deep red colour from the presence of a deep red compound of sulphide of sodium and sulphide of iron. It was formerly, and is indeed even now in some works, the practice to carbonate this caustic liquor and evaporate it with the other liquors to make soda-ash, and in some cases this answers well; but when the soda-ash is used for soap-making, the practice appears rather anomalous, inasmuch as the carbonate of soda of the ash must again be reconverted into the caustic state.

In 1853, Mr. William Gossage, of Widness Dock, near Warrington, whose numerous discoveries and improvements in the alkali manufacture have largely contributed to raise it to its present important position, took out a patent for using this red liquor for the production of *solid* caustic soda; and I believe I am right in stating that the greater portion of the caustic soda now used is made from red liquor.

Mr. Gossage's Patent.-The following is a brief outline of the patent of Mr. Gossage, which includes the caustic soda process. He first of all oxi-dizes the sulphide of sodium of the black-ash liquors by bringing them in contact with atmospheric air in a tower filled with pieces of coke; the liquors trickle slowly down through the coke, whilst a current of air is caused to pass up. By the action of the oxygen of the air, the sulphide of sodium of the liquors is oxidized, and the sulphide of iron before held in solution is thus set free and precipitated. He next adds a small portion of chloride of lime, to oxidize any hyposulphite. The liquor is then concentrated, to obtain salts or carbonate of soda; these salts are washed either with water or a solution of carbonate of soda, and are then drained and roasted in the ordinary manner, to form soda-ash. The mother-liquors from which the salts have separated are concentrated until they have a specific gravity of 1.45, and, by this time, nearly all the carbonate of soda and any ferrocyanide of sodium that may have been present have precipitated. After being allowed to settle, the liquor is drawn off and concentrated, to form solid caustic soda. The patent includes a process for the separation of the ferrocyanide of sodium from the carbonate of soda, and obtaining both salts free from admixture. I need not here describe the process for separating the ferrocyanide. There is also included an improved method for concentrating liquors by exposing them in thin films to the action of heated air.

I now proceed to describe the process as at present generally employed for the production of solid caustic soda without the use of lime, i.e. from the red liquor.

Manufacture of Caustic Soda from Red Liquor.—The liquors obtained by lixiviating black ash are concentrated by any of the methods usually employed, until the greater portion of the carbonate of soda has separated. The motherliquors thus obtained are the red liquors; they have a sp. gr. of about 1.30, and contain principally hydrate of soda; besides this, however, there exists in them some quantity of carbonate of soda, a deep red compound of sulphide

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of sodium and sulphide of iron, some sulphate of soda and chloride of sodium, a small amount of ferrocyanide of sodium, and a minute quantity of some other compounds. These red liquors are mixed with a small quantity of chloride of lime, and placed in a pan generally known by the name of a *fishing* or *boat pan* so called from its shape. In some works the bleaching powder is not added quite so early in the process.

This pan is made of quarter-inch wrought iron plates riveted together. It has a length of about 30 ft. and a width of about 8 ft.; the sides are sloping, so as to facilitate the collection of salts; but the greatest depth does not exceed 2 ft. 6 in. The pan is set in brickwork and heated by a fire underneath it, the flues from the fireplace passing directly under the entire length of the sloping sides of the pan. There are several methods of setting these boat-pans, and in the arrangement of the fireplace and flues, nearly every works having a distinct plan of its own.

The invention of this pan, which is largely adopted in alkali manufactories, is due to Mr. David Gamble, of St. Helen's. In this pan the liquor is concentrated by boiling until it has a specific gravity of 1.40, and a temperature of from 260° to 270° Fahr. Some nitrate of soda is added to the liquor whilst in this pan. During the concentration, salts separate which are fished out by means of a perforated spade. When the liquor has obtained the sp. gr. and temperature I have mentioned, it is run out into a vessel called the cooler or settler, and there allowed to remain some little time in order that salts may deposit; by cooling, the liquor deposits salts which at a high temperature are held in solution. Some manufacturers allow the liquors to settle some time in the boat-pan, but this I consider to be a bad plan, as the pan may then be said to be doing no work; but if the liquors are at once run off when sufficiently strong, the pan may again be filled up, and the boiling of other liquors commence.

The boat-pans very frequently require repairs, which is a serious annoyance to the manufacturer. The principal reason for this is, that the salts sometimes adhere to the sides of the pan, notwithstanding the most careful attention on the part of the workman. Where the salts thus adhere, the pan becomes much hotter in that part, and presently when the liquor penetrates through this crust of salts the pan is suddenly cooled, and the unequal expansion and contraction of the plates injures it severely. This is not the only reason, for the caustic soda liquors appear to act upon the iron of the pan, and thus injure it, especially where the plates are riveted together. I shall allude to this action of the liquors upon iron again presently.

The salts which fall in the boat-pan and settler consist principally of carbonate of soda, mixed with some sulphate of soda, and also with a small quantity of chloride and ferrocyanide of sodium. They are sometimes converted into soda-ash, but more frequently are dissolved, the carbonate converted into caustic soda by means of lime, and then added to the red liquors. The liquor, when properly settled, is run out into another vessel. This vessel is a hemispherical cast-iron pot, which at the top has a diameter of about 5 ft.; to the top of the pot is fitted a cast-iron ring, which is about  $2\frac{1}{2}$  feet high; and the greatest depth of the vessel, including the ring, is from 5 ft. to 5 ft. 6 in. It is set in brickwork, and heated either by a fire underneath it which plays directly upon the rounded bottom of the pot, or it is heated by the waste heat from another pot of the same description which is used in the finishing process.

To the liquor in this pot more nitrate of soda is added, and the whole boiled together for some time (from twelve to twenty-four hours, according to circumstances); more salts separate which are fished out by the workmen; the liquor is also allowed to settle some time.

When salts cease to fall, the liquor is placed in another pot called the finisher

or finishing-pot, which is similar to the one last described, but is always heated by its own fire. In this pot the liquor is boiled until it contains upwards of 60° of soda. More nitre is thrown into this pot. During the boiling a thick scum appears upon the surface of the liquor, which is carefully skimmed off by the workman; and when the contents of the pot possess the desired percentage of soda, the fire is slackened or entirely withdrawn, and the liquor after being allowed to settle a short time is run off into iron casks, where, as it becomes cold it assumes the solid form, and is then ready for removal from the works.

The process for the manufacture of caustic soda from red liquor differs slightly in minor details in different works, but in all principal points it is conducted in the manner I have just described.

I have not in this short sketch alluded to the changes which the liquid undergoes, for I think it better to mention them after describing another process for the manufacture of caustic soda, which necessitates the use of lime.

Preparations of Caustic Soda from Black Ash and Soda-Ash.—In this process either black ash or soda-ash is used as the source of the soda. The liquors obtained by lixiviating black ash are diluted with water until they have a sp. gr. of from 1.09 to 1.12, or if soda-ash is used, it is dissolved in water until the solution has the specific gravity. The liquor is then agitated with lime, in order to convert the carbonate of soda into hydrate of soda :

# $NaO CO_2 + CaO HO = NaO HO + CaO CO_2$ .

If the specific gravity is higher than 1.10, the causticizing is not complete, as some carbonate of soda remains in the liquor. Even on a small scale in the laboratory this is the case; and it is decidedly preferable not to have the specific gravity of the liquor higher than 1.09.

The vessels in which the liquors and lime are agitated together are usually made of a portion of an ordinary cylindrical boiler. The boiler is generally cut in half lengthways. Fitted in this vessel is an agitator made of iron, and somewhat similar in form to the agitator of an ordinary butter-churn. When at work, it is kept revolving by means of steam power. The vessel is filled to a certain height with liquor, and milk of lime or caustic lime in lumps is then mixed with it, and the mixture agitated until the decomposition is complete. It is decidedly the best plan to use milk of lime rather than lump lime, as the process of agitating the mixture requires much less time. The milk of lime should be made in a separate vessel, and from it run into the causticizing pan. With milk of lime the decomposition may be completed in about half an hour; and the liquor is then run into large iron settlers, and there allowed to remain until all the lime-mud has deposited.

Treatment of Lime-Mud.—Before describing the process for concentrating the liquors, I will first dispose of this lime-mud. After the liquor has been run off, the mud is washed, in order to free it as much as possible from any soda which may be mixed with it.

This washing is effected in two or three ways. One method is to agitate it with water in a vessel similar to the causticizing pan, or else to agitate it by manual labour with a wooden pole or iron plunger. When the lime and water have been sufficiently agitated together, the mixture is allowed to stand, so that the lime-mud may settle to the bottom of the vessel. This settling may either take place in the washing vessel or in a settler set apart for the purpose. The clear liquor or washing, which contains any soda that may have been dissolved, is run off and used either for dissolving more soda ash or for diluting fresh black-ash liquor, as the case may be. The lime-mud is run out of the settler into a heap to dry.

Another method employed is to run the mixture of lime-mud and washings on to a filter. The filter is a large iron vessel fitted with a perforated false

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bottom. Upon this false bottom is placed first of all a layer of coke, above this a layer of pebbles, and on this again a layer of sand.

The washings are either allowed to drain slowly through the filter, or the process is hastened by producing a vacuum by connecting the lower part of the filter with a large air-pump, and then exhausting the air, or by injecting steam into the space at the bottom.

By producing a vacuum, the liquid is caused to pass through immediately. When all the liquid has passed through, fresh water is run on to the lime, and percolating through it washes out more of the soda; and this process is repeated until the mud is sufficiently free from soda. The lime is then thrown into a heap to dry; a tolerably warm place (such as a slip of ground immediately over a flue) being selected for the drying place.

The lime-mud, when sufficiently dry, is used instead of limestone in the manufacture of black ash.

Composition of the Lime Deposit.—By the most careful washing it has not, I believe, been found practicable to entirely free the lime from soda. Although the last washings passing away from it may not contain any soda whatever, yet, if the washed lime is subjected to a dull red heat sufficient to expel the carbonic acid, and the mud then washed with water, more soda may be extracted. Mr. Kynaston, formerly a pupil of Dr. Muspratt, and afterwards chemist at the works of Messrs. Muspratt, Brothers, and Huntley, of Flint, was I think the first to notice this, and he supposes that the soda is chemically combined with the lime. This is, I know, the opinion of several chemists who have had to deal with the subject, and I certainly believe it to be so myself. Mr. Kynaston states that from five to six per cent. of soda remains after washing the undried mud. I have not found the amount quite so great. When the washing has been carefully performed on a small scale in the laboratory, I have never found more than 3 per cent. or less than  $2\frac{1}{2}$  per cent.

This soda is of course found in the lime-mud produced by the soap-makers, and many of them send it away as refuse, or sell it at a very low price to persons who use it for mortar. By so doing they are losing a large quantity of soda. A year or two ago I examined some of this refuse from a soap-works, and found it to contain  $5\frac{1}{2}$  per cent. of soda; but this had been carelessly washed.

Dr. Muspratt, in his 'Dictionary of Chemistry,' suggests that the soapmanufacturer should first dry this lime deposit by exposure to the air, and then heat it, to expel carbonic acid. The lime, being thus converted into the caustic state, may be used to prepare fresh lye, and the soda which it contains would be thus saved. This process may be repeated until the lime becomes too highly charged with the impurities of the soda-ash.

This suggestion is worthy the attention of the caustic soda makers, who may perhaps find it an advantage to use the same lime for causticizing several quantities of liquor.

Concentration of Causticized Liquors.—Imust now go back to the causticized liquor which I left in the settlers. This liquor is now to be concentrated. In some works (indeed, as far as I know, the practice is general) it is first placed in an iron boiler (an ordinary steam boiler), which may be closed in and fitted with safety-valves, pipes, etc., or an opening left for the escape of steam. In the former case the steam disengaged may be used as a motive power for working the machinery required in the works.

A patent has been taken out by Messrs. Roberts, Dale, and Co., of Warrington and Manchester, for the concentration of liquors in this manner and the employment of the steam disengaged.

The liquor is concentrated in the boiler until it has a specific gravity of **1**·24 or 1·25 and a temperature of 230° Fahr. It is then run into the boat-pan, and from that time the process is conducted in the same manner I have described when speaking of the red-liquor caustic, except that in some instances the second concentrating vessel is not used, but the liquor is run from the boat-pan into the cooler, or settler, and from that direct into the finishing-pot. When however soda-ash is employed, it is not necessary to give so large an amount of settling, or to add any nitrate of soda, except in occasional instances.

Chemistry of the Process.—I will now describe the chemical changes which take place during the concentration. As I before mentioned, the red liquor contains hydrate of soda, carbonate of soda, sulphate of soda, chloride of sodium, sulphides of sodium and iron, together with some ferrocyanide of sodium and a small quantity of other compounds. The causticized black-ash liquor also contains the same compounds. Both the red liquor and the causticized black-ash liquor, as they become stronger by boiling in the boat-pan, deposit salts, which consist principally of carbonate of soda, some sulphate of soda, and a little ferrocyanide of sodium. When nitre is added to the liquor some of the sulphide of sodium is oxidized, and the sulphide of iron being at the same time converted into peroxide is precipitated with the salts, giving them a deep red colour. It is not well to add the nitre too soon in the boatpan, as when it is added early, before the temperature is sufficiently high, some of it instead of being decomposed is deposited with the salts.

The oxidation of the sulphide proceeds slowly in the boat-pan, the sulphide being converted into sulphate and the nitrate into nitrite of soda. This is when the temperature is under 280° Fahr., and as a rule the liquor in the boat-pan never reaches a higher temperature than 270° Fahr.; but when, as is the case in the next vessel, the temperature is above 280° Fahr. or 290° Fahr., the decomposition proceeds much more rapidly, and ammonia is disengaged to a very large extent. Although some of this ammonia is without doubt derived from the decomposition of the cyanides, yet the quantity is so very great that the amount of cyanides present could not produce it all. It proceeds from the decomposition of the nitrate of soda; and, according to Dr. Pauli, of St. Helen's, who has given much attention to the subject, the change may be thus represented :--

## $2 \text{ NaS+NaO NO}_5 + 4 \text{ HO} \equiv 2 \text{ NaO SO}_3 + \text{NaO HO} + \text{NH}_3$

When the temperature rises above  $310^{\circ}$  Fahr., as it does in the finishing-pot, free nitrogen is also given off. This decomposition is explained by Dr. Pauli thus:—

#### $5 \text{ NaS} + 4 \text{ NaO NO}_5 + 4 \text{ HO} \equiv 5 \text{ NaO SO}_3 + 4 \text{ N}.$

This I believe to be a true explanation of the principal decompositions which take place; but it must be remembered that there are other compounds present, such as sulphites, hyposulphites, etc., and these also are decomposed. Dr. Muspratt represents the decomposition :—

 $2 \operatorname{NaS} + \operatorname{NaO} S_2 O_2 + \operatorname{NaO} \operatorname{NO}_5 + 4 \operatorname{HO} = 4 \operatorname{NaO} \operatorname{SO}_2 + \operatorname{NH}_4 O.$ or  $\operatorname{NaS} + \operatorname{NaO} S_2 O_2 + \operatorname{NaO} \operatorname{SO}_2 + \operatorname{NaO} \operatorname{NO}_5 + 4 \operatorname{HO} = 4 \operatorname{NaO} \operatorname{SO}_3 + \operatorname{NH}_4 O.$ 

The quantity of sulphide of sodium present in the liquor is, however, in greater proportion to the sulphites and hyposulphites than is represented in the equation; and there can be no doubt that hydrate of soda is also formed during the decomposition. It is rather a difficult matter to say with certainty what decomposition really does take place when there are so many compounds present. Probably the changes as represented both by Dr. Muspratt and Dr. Pauli take place, and others most likely also occur; but the explanation given by Dr. Pauli appears to me to represent the principal decomposition.

The iron also present in different forms is oxidized into peroxide, and unless

separated would give the soda a very objectionable colour. Its separation is principally effected by settling in the pans and settlers. As the liquor becomes more dense the iron does not settle down readily, and is found to rise to the top of the liquid in the finishing-pot as a scum, which is skimmed off by the workman; and the caustic soda, when properly made, is almost white and comparatively free from iron. It however still contains some, which is sure to be the case, owing to the fact that peroxide of iron is soluble in caustic soda to a slight extent; although it is generally stated in chemical books that soda *completely* precipitates persalts of iron. By digesting precipitated peroxide of iron with solution of caustic soda, I have found the peroxide to be taken up in greater or less proportion according as the solution was stronger or weaker. When however the solutions were diluted with water, some of the peroxide was again precipitated, but there still remained dissolved permanently, even when the solution was extremely dilute, a small quantity of iron, equal to one part of peroxide to 7000 parts of soda.

There is more than this in much of the caustic soda of commerce, which is owing to the fact just mentioned, that a concentrated solution of caustic soda takes up a larger quantity in proportion of peroxide of iron than is taken up by more dilute solutions.

I am inclined to believe that some of this is merely mechanically held, owing to the oxide being in a very finely divided state, and therefore very light; and the soda solutions being very dense, the oxide does not readily precipitate. As I mentioned just now, if the strong solution of soda is diluted with water some of the peroxide is precipitated, and a similar result takes place when some of the caustic soda of commerce is dissolved in water. It may be that a strong solution of soda *dissolves* more peroxide of iron in proportion than a weaker solution, and that upon dilution the peroxide is precipitated, but I doubt it; for I have found that when concentrated caustic soda liquor, to all appearances clear and bright, is placed in a stoppered vessel and allowed to stand for several days, the quantity of iron in it gradually decreases, and the bottom of the vessel becomes covered with a deposit of peroxide of iron. This appears to show that the iron is merely mechanically held in a very finely divided state, and by prolonged settling was deposited.

It has been suggested that the peroxide of iron during the boiling of the caustic liquor with nitre at a high temperature is by oxidation converted into ferric acid, and this uniting with soda forms ferrate of soda. At one time I was much disposed to doubt this, but lately I have made some experiments upon this point, and the results I have obtained lead me to believe that ferrate of soda is formed and does exist in solid caustic soda, but is decomposed upon dissolving it in water.

Some of the caustic soda sold has a very delicate blue or greenish tinge, which is, I believe, due to the presence of a trace of manganese.\*

The quantity of nitre used varies considerably. For red-liquor caustic, from 3 quarters to  $1\frac{1}{2}$  cwts. is used per ton of caustic; for caustic soda made from black-ash liquor, about  $\frac{1}{2}$  cwt. per ton is employed; and when soda-ash is used it is seldom necessary to add any.

The consumers of solid caustic soda have a great objection to the presence of iron in it, and it is very desirable that it should be separated. Now,

<sup>\*</sup> Mr. Martin Murphy, assistant to Dr. Muspratt, states, that he has proved this colour to be due to manganese, and supposes the manganese to be derived from the limestone used in making the black ash. He has found manganese in several samples of limestone which he has examined.

without doubt, this should be done as early as possible, and before the liquors become strong; and if the iron can be separated, not in the state of oxide, so much the better. I am not aware that this is done. It may, however, be effected by the employment of Mr. Gossage's oxidizing tower. If the blackash liquors, or even the red liquors, are subjected to the action of air in Mr. Gossage's tower, the sulphide of sodium is converted into sulphate or sulphite, and the sulphide of iron is precipitated. It therefore follows, that if the liquors are properly settled, all the iron which existed as sulphide may thus be separated at the very commencement of the process (a great saving of nitrate of soda would also be effected).\* There still, however, remains some iron in the form of ferrocyanide of sodium, and all this does not separate as salts, but the iron of the ferro-cyanide is converted into peroxide of iron, which either remains dissolved by the soda or separates as a precipitate. There is also the oxide of iron (in the shape of rust) which the soda takes up from the vessels used in the manufacture, and, unless I am mistaken, the concentrated soda liquors act upon metallic iron. By exposing a piece of good wrought iron to the action of the liquor in the finishing pot for two or three days, I found it to materially lessen in weight. I had not time to carry out the experiment in a complete manner, so as to satisfy myself upon the point. There are so many compounds present in the liquors that the action upon iron may not be due to hydrate of soda, but probably to other compounds. The iron is, however, only acted upon but very slowly. I have no doubt means will soon be adopted to free the caustic soda from the small quantity of iron it now contains, for several persons are working at the subject, and I am informed that already some samples have been produced which are very nearly chemically pure.

The 'Chemical News,' of June 28, 1862, contains an article by Dr. P. Pauli, of the Union Alkali Works, St. Helen's, upon "The Preparation of Pure Caustic Soda on a Large Scale," and the process there described will, I believe, meet the difficulty which is at present experienced in preparing caustic soda free from iron, etc. The following is Dr. Pauli's description of the process :— "Three tons of commercial caustic soda, containing excess of water, alumina, and all the impurities which commonly occur in this substance, are fused in a cast-iron pet. During the evaporation, nearly all the carbonate and by far the larger quantity of the other salts separate out on the surface as a scum, and can be easily removed. The liquid mass is then heated to dull redness, and kept at that temperature during the night. In the morning the mass appears perfectly transparent, the sides and bottom of the vessel being coated with cauliflower-shaped masses of crystals, consisting of silicate of alumina, with chloride and sulphate of sodium, and a little lime. The clear fused liquor is ladled off from these crystals, and when cooled is ready for use."

Dr. Pauli remarks, that the soda thus obtained contains only a trace of carbonate of sodium, is perfectly free from alumina, and that oxide of iron is completely separated from it; also that it is hard and brittle, and may be easily obtained as a fine powder by attrition in an iron mortar.

A few experiments I have made since reading Dr. Pauli's paper fully corroborate the statements therein contained.

Use of Chloride of Lime.—Before going any further, I would make a few observations respecting the use of chloride of lime. Its use should be avoided, for, although it effects the object desired,—the oxidation of some of the sulphur compounds,—yet the advantage is more than counterbalanced by the loss of alkali it occasions by converting it into chloride of sodium. It is decidedly preferable to use nitrate of soda instead of chloride of lime, although

\* I am informed that this process has lately been adopted in an extensive Lancashire works.

the price of the former is much higher than that of the latter; but I have found by experiment that nitrate of soda is in the end the cheapest, as by its decomposition hydrate of soda is formed, whilst by the employment of chloride of lime some alkali is rendered useless. The results of some practical experiments which I have just completed, show that 100 parts of chloride of lime render useless about 50 parts of alkali.

Other Processes for Manufacturing Caustic Soda.—There are two or three other processes which I will just mention before concluding.

One, formerly employed, consisted in intimately mixing together one part of common salt and from four to six parts of litharge and a little water. Chloride of lead and hydrate of soda were formed. The chloride of lead afterwards combined with some of the undecomposed litharge to produce oxychloride of lead, which, being insoluble, was allowed to deposit, and the clear liquor containing the hydrate of soda was run off and evaporated. The lead compound, when dried and fused, formed the colour known as patent yellow. This process has, I believe, been abandoned on the score of expense. With the large production of caustic soda of the present time there would also probably be a difficulty in making use of the yellow pigment. Another process which has been proposed, was to mix together solutions of hydrate of baryta and sulphate of soda. The insoluble sulphate of baryta is formed and precipitates, and hydrate of soda remains in solution. Caustic baryta cannot, however, be procured in anything like the quantity required for the carrying out of this process on a large scale.

Mr. Hunt proposed to decompose sulphate of soda by roasting it with coal or coke, and then lixiviate the mass with water. The solution contains sulphide and bisulphide of sodium, and carbonate and sulphate of soda, and is boiled with oxide of zinc or black oxide of copper. By the action of the oxide upon the sulphides of sodium, sulphide of zinc or copper is produced, which precipitates, whilst hydrate of soda remains in solution.

A process mentioned in 'Silliman's Journal' for November, 1858, consists in first causticizing black-ash liquors with lime. The caustic solution is then evaporated, to separate salts, and fine sesquioxide of iron (in the form of Venetian red or pure earthy hæmatite) added to it in such a quantity, that the sesquioxide shall somewhat exceed in weight the amount of dry hydrate of soda contained in the liquor. This mixture is dried down with constant stirring, and roasted at a dull red heat. The roasted mass is then treated with water so as to get a solution of sp. gr. 1.40. The clear solution is poured off and evaporated. I am not aware that this process has been adopted anywhere in this country.

In the 'Chemical News,' of May 10, 1862, is a notice of a paper by W. Woehler, in the 'Annalen der Chemie der Pharmacie,' on "The Preparation of Caustic Soda." This process consists simply in calcining nitrate of soda with peroxide of manganese. No chameleon is formed as might be supposed, since the nitrate decomposes long before the mixture can reach the temperature necessary for the production of manganic acid. This process appears to me to be too expensive to be used for the preparation of caustic soda on a large scale.

A word or two respecting the mode of using the black-ash liquors for the production of solid caustic soda. The black-ash liquors contain both hydrate of soda and carbonate of soda, the quantity of soda as hydrate existing generally in the proportion of about 25 parts to every 75 parts of soda as carbonate. Now, if the carbonate of soda is at once converted into caustic soda by the addition of lime, it is necessary, as the black-ash liquor has on the average a specific gravity of 1.25 or 1.26, to add water to the whole of the liquor to reduce it to a sp. gr. of 1.10, and by so doing the caustic portion is diluted as

well as the carbonate. In order to produce solid caustic soda, this water must again be expelled. It appears to me to be a better plan to first concentrate the black-ash liquors, so as to separate all the carbonate as salts. This may then be redissolved in water, causticized, and then evaporated with the other caustic liquor (the red liquor).

I am of opinion that the process for the manufacture of caustic soda, as the consumption of that article increases, must be conducted something after the manner of the process described in the patent of Mr. Gossage, which I mentioned at the commencement of this paper; but, instead of using the salts obtained for the production of soda-ash, they will be redissolved, causticized, added to the red liquors, and the mixed liquors concentrated to produce solid caustic soda.

In a few words I will give an outline of what the process would then consist of.

Oxidize black-ash liquor in the oxidizing tower, and allow the sulphide of iron to deposit; concentrate the liquors until all carbonate has separated; redissolve this carbonate and causticize; add the causticized liquor to the red liquor and evaporate, adding as much nitrate of soda as is required.

I have now described the manufacture of solid caustic soda. I might perhaps have given a more minute description of the various processes and of the apparatus employed, but such details are only interesting to those actually engaged in the manufacture, and therefore I consider it unnecessary to enter into them here. What I have described embraces all the principal points in the manufacture.

In conclusion, I would remark that the production of solid caustic soda is one of the many advantages which have been derived from bringing science to bear upon the practical working of our manufacturing operations.

Of all the sciences, chemistry has without doubt contributed the most to promote the growth and development of the arts and manufactures; and one of the greatest examples of the importance of chemistry in its practical applications is the rise and development of the soda manufacture.

As Liebig justly remarks,\* "The manufacture of soda from common culinary salt may be regarded as the foundation of the extraordinary impulse given to improvement in every department of modern industry; and we may take it as affording an excellent illustration of the dependence of the various branches of human industry and commerce upon each other, and their relation to chemistry."

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# ON THE WEIGHTS, MEASURES, ETC., OF THE NEW PHARMACOPCEIA.

#### TO THE EDITOR OF THE PHARMACEUTICAL JOURNAL.

Dear Sir,—I am glad to see the subject of weights to be used in the New Pharmacopœia is again engaging attention. Not having expressed an opinion in this important matter, I am induced to make a few observations; if you think them worthy of place in the Journal, they are at your service.

think them worthy of place in the Journal, they are at your service. I may premise that in the compiling of the New Pharmacopœia, the application of old names to new preparations was a subject frequently adverted to, and the evils of so doing carefully pointed out. If this be true with respect to articles to be weighed, it is no less true as regards weights to be used,