

PATENT SPECIFICATION



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PROVISIONAL SPECIFICATION.

Improvements in or relating to the Manufacture of Alkali Metal Chlorates.

We, CHARLES CARTER, a British subject, of Meadowside, Hunts Cross, Liverpool, and IMPERIAL CHEMICAL INDUSTRIES LIMITED, a British Company, of Imperial Chemical House, Millbank, London, S.W. 1, do hereby declare the nature of this invention to be as follows:—

This invention relates to the manufacture of alkali metal chlorates or mixtures containing alkali metal chlorates by the action of chlorine on alkali metal carbonates.

It has previously been proposed to prepare solutions containing sodium chlorate by passing chlorine through solutions or slurries containing sodium carbonate. It has also been proposed to treat finely divided dry anhydrous sodium carbonate with chlorine preferably in a mechanical apparatus, in order to obtain a mixture of chloride and chlorate. In this process it was further stated that aqueous vapour which might be present in the chlorine was a disadvantage and that improved results were obtained by using a supply of chlorine dried by means of sulphuric acid. In the operation of this process, however, the action of the chlorine on the sodium carbonate is too slow for an economically working process, and we have discovered that it is essential for a certain amount of water to be present.

According to the invention, therefore, we treat a solid alkali metal carbonate with chlorine in the presence of an amount of water corresponding to from about 3% to about 20% by weight of the carbonate. The water may be supplied to the reaction in any convenient manner, for instance, it may be introduced partly as vapour carried by the chlorine and partly as water of crystallization combined with alkali metal carbonate or, if desired, it may be introduced as liquid water, e.g. in the form of a spray. Bicarbonate may also be present in addition to the carbonate.

The chlorine may be introduced as a substantially pure gas or may be diluted

with large quantities of an inert gas such as air. Preferably the reaction is conducted at a temperature of approximately 60—70° C.

The process, which may be continuous or discontinuous, may be carried out in any convenient form of apparatus constructed of or lined with resistant material and arrangements may be made for control of temperature.

Thus in a discontinuous process sodium carbonate which contains about 13% by weight of water and which is in the form of powder is spread in layers and exposed to the action of chlorine until the reaction is complete. If desired, to accelerate the reaction the process may be interrupted from time to time so that the layers of material may be raked in order to expose a fresh surface.

The continuous process may be operated conveniently in a rotating tube which may be slightly inclined to the horizontal. Thus powdered sodium carbonate containing about 5% by weight of water is fed to the upper end of the tube and passes down while the tube is rotating, in counter current to a stream of chlorine introduced at the lower end. A dry powder consisting of a mixture of sodium chloride and sodium chlorate in a molecular ratio of about 5½ to 1 is obtained.

For certain purposes, e.g. as a weed killer, the mixture of sodium chlorate and sodium chloride may be used in the dry state as it is produced but if desired the powder may be leached with water and a strong solution obtained from which sodium chlorate can be recovered by evaporation and crystallisation.

Dated the 4th day of February, 1932.

E. C. G. CLARKE,
Imperial Chemical House, Millbank,
London, S.W. 1,
Solicitor for the Applicants.

COMPLETE SPECIFICATION.

Improvements in or relating to the Manufacture of Alkali Metal Chlorates.

We, CHARLES CARTER, a British subject, of Meadowside, Hunts Cross, Liverpool, and IMPERIAL CHEMICAL INDUSTRIES LIMITED, a British Company, of Imperial Chemical House, Millbank, London, S.W. 1, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

This invention relates to the manufacture of alkali metal chlorates or of solid products containing alkali metal chlorates by the action of chlorine on alkali metal carbonates.

It has previously been proposed to prepare solutions containing sodium chlorate by passing chlorine through solutions or slurries containing sodium carbonate.

Solutions of sodium chlorate have also been prepared by the passage of chlorine through an absorbing tower packed with hydrated sodium carbonate which may have been brought into a granular or a pasty condition by the addition of water.

Weak solutions of the chlorate are allowed to trickle over the solid carbonate during this process in which the amount of water added to soda ash was of the same order as that of the soda ash itself.

It has also been proposed to treat finely divided dry anhydrous sodium carbonate with chlorine preferably in a mechanical apparatus, in order to obtain a solid product consisting of a mixture of chloride and chlorate. In this process it was further stated that aqueous vapour which might be present in the chlorine was a disadvantage and that improved results were obtained by using a supply of chlorine dried by means of sulphuric acid. In the operation of this process, however, the action of the chlorine on the sodium carbonate is too slow for an economically working process, and we have discovered that it is essential for a certain amount of water to be present. In addition, the quantity of water which is present must not be so great that the reaction mass or the final product assumes a pasty condition. This careful regulation of the water content we have found to be of particular importance to ensure a smoothly working manufacture, especially one which is operating continuously.

According to the invention, therefore, we treat a solid alkali metal carbonate with chlorine in the presence of an

amount of water corresponding to from 3% to 20% by weight of the carbonate. The water may be supplied to the reaction in any convenient manner, for instance, it may be introduced partly as vapour carried by the chlorine and partly as water of crystallization combined with alkali metal carbonate or, if desired, it may be introduced as steam or as liquid water, e.g. in the form of a spray. Bicarbonate may also be present in addition to the carbonate.

The chlorine may be introduced as a substantially pure gas or may be diluted with large quantities of an inert gas such as air. Preferably the reaction is conducted at a temperature of approximately 60—80° C.

The process, which may be continuous or discontinuous, may be carried out in any convenient form of apparatus constructed of or lined with resistant material and arrangements may be made for control of temperature.

Thus in a discontinuous process sodium carbonate which contains about 13% by weight of water and which is in the form of powder is spread in layers and exposed to the action of chlorine until the reaction is complete. If desired, to accelerate the reaction the process may be interrupted from time to time so that the layers of material may be raked in order to expose a fresh surface. In a continuous process suitable amounts of water are 3—15% by weight of sodium carbonate.

The following example illustrates one form of a continuous process.

EXAMPLE.

Soda ash containing an excess of water, which for experimental reasons varied from 3% to 12% on the weight of the carbonate, was fed continuously at the rate of 96 lbs. per hour to the upper end of a rotating iron tube lined with earthenware tiles, and mounted at a slight angle to the horizontal, while 1100 cubic feet per hour of gas previously heated to about 110° C. and containing 31% Cl₂, were blown in at the lower end of the tube, passing countercurrent to the descending solids and issuing at the upper end of the tube at a temperature of about 30—40° C. After a short period of operating it was found desirable to maintain the water content of the soda ash at 3—5% under the conditions described. The

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soda ash travelling down the tube was converted to a mixture of chlorate and chloride principally in a main reaction zone at about the middle of the tube where the solids had a temperature about 70—80° C. The solid product was produced at the rate of 119 lbs. per hour and on issuing from the tube was very slightly moist to the touch. An average of analyses made over a period of 24 hours showed its composition to be:—

21.47% NaClO₃
 71.72% NaCl
 0.46% Na₂CO₃
 2.80% NaHCO₃
 3.55% H₂O (by difference)

The exit gas from the tube contained a slight amount of residual chlorine and some hypochlorous acid in addition to the carbon dioxide produced in the reaction. It was passed through a small scrubbing tower before being allowed to escape to the atmosphere. As an alternative to this procedure the gas may be recirculated, a suitable purge for the excess gas being arranged for and the requisite chlorine being introduced to maintain the strength of the reacting gas. If desired, the necessary water could have been introduced as a spray of liquid water or as a jet of steam rather than as an addition to the soda ash as is described.

For certain purposes, e.g. as a weed killer, the mixture of sodium chlorate and sodium chloride may be used in the dry state as it is produced but if desired the powder may be leached with water and a strong solution obtained from which sodium chlorate can be recovered by evaporation and crystallisation.

Having now particularly described and ascertained the nature of our said inven-

tion, and in what manner the same is to be performed, we declare that what we claim is:—

1. A process for the production of a solid product containing alkali metal chlorate which comprises reacting chlorine with alkali metal carbonate in the presence of an amount of water which is equal to 3% to 20% of the weight of the alkali metal carbonate.

2. A continuous process for the production of a solid product containing sodium chlorate which comprises passing chlorine counter current to sodium carbonate in the presence of water equal in amount to from 3% to 15% of the weight of sodium carbonate.

3. A process as claimed in Claim 1 or 2, in which the water is introduced into the reacting system in association with the carbonate, or as vapour or as liquid or by a combination of two or more of these methods.

4. A process as claimed in Claim 1, 2 or 3 in which the temperature of the reaction zone is maintained at 60° to 80° C.

5. A process as claimed in Claim 4 in which the chlorine is heated prior to its introduction to the reacting system.

6. A process for the production of a solid product containing alkali metal chlorate, substantially as hereinbefore described, with reference to the example.

7. Alkali metal chlorates, or products containing the same, whenever prepared by the process claimed in any of Claims 1—6.

Dated the 31st day of October, 1932.

E. C. G. CLARKE,
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 Solicitor for the Applicants.