

# UNITED STATES PATENT OFFICE.

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## METHOD OF PRODUCING AMMONIUM PERCHLORATE.

985,724.

Specification of Letters Patent.

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No Drawing.

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*To all whom it may concern:*

Be it known that I, OSCAR BIRGER CARLSON, a subject of the King of Sweden, and resident of Månsbo, Avesta, in the Kingdom of Sweden, have invented certain new and useful Improvements in the Method of Producing Ammonium Perchlorate, of which the following is a specification.

In practice it has been found impossible to produce ammonium perchlorate directly from ammonium chlorid by electrolysis on account of the instability of the intermediate product, viz. the ammonium chlorate. One has, therefore, been left to the conversion, by means of ammonium chlorid, of sodium perchlorate, which can be produced by electrolysis. A method of converting sodium perchlorate by means of ammonium nitrate has also been patented, but this method is, from a technical and economical point of view, inferior to the first mentioned one.

Experience has, however, proved that it is possible in practice to use for the said purpose ammonium sulfate, the cheapest ammonium salt in the market, and the present invention has for its object a method of producing ammonium perchlorate by converting sodium perchlorate by means of ammonium sulfate.

If one tries to separate ammonium perchlorate by means of crystallization from the lye obtained by adding ammonium sulfate to sodium perchlorate after the said lye has been sufficiently concentrated in any desired manner, ammonium perchlorate will settle on cooling the said lye, but at a certain point the by-product, viz. sodium sulfate, will also crystallize, containing 10 molecules of water of crystallization. On account of this a fresh quantity of ammonium perchlorate inclosed within the sodium sulfate will also be deposited. It is therefore very difficult and expensive to separate ammonium perchlorate from the by-product in this way. This fact which is based upon the known condition of crystallization of sodium sulfate, is evidently the reason why ammonium sulfate has not hitherto come into use in producing ammonium perchlorate. Further investigations have proved, that the greatest part of the ammonium perchlorate has, contrary to expectation, crystallized before the crystallization of the sodium sulfate takes place, and more-

over that in the presence of ammonium perchlorate this crystallization begins, on account of phenomena of supersaturation, at about 22° Cent. instead of at 33° Cent. Thus, there is nothing to prevent the use of ammonium sulfate for the conversion, provided that the mother liquor is separated from the deposited ammonium perchlorate at a temperature somewhat above 22° centigrade. In order to get out the ammonium perchlorate, which is still in the mother liquor, the latter is concentrated by boiling, whereby waterfree sodium sulfate deposits while the liquor is hot and is separated before the same has become cold. In this manner a fresh portion of ammonium perchlorate is obtained, and so on. The ammonium perchlorate obtained in this way contains but a small quantity of sodium sulfate, as the solubility of the waterfree sodium sulfate increases when temperature decreases. In the above manner the ammonium perchlorate can be quantitatively utilized at the same time as the sodium sulfate is obtained free from water and ammonia. If one should not wish to use evaporation, the mother liquors may be employed to dissolve fresh quantities of sodium perchlorate and ammonium sulfate. In these operations waterfree sodium sulfate also deposits from the solutions and is then removed.

It has been found that, on account of the conditions of solubility, it is possible to proceed in two ways in practically making the conversion. According to the first way hot solutions of ammonium sulfate and sodium perchlorate are mixed together—one of these solutions may also be replaced by the corresponding salt in solid state—whereupon the ammonium perchlorate deposits, when the solution is cooled. According to the second way cold solutions are used; one of these may also be replaced by solid salt. In this the temperature will rise from 35°-40° because heat is developed in the conversion. The greatest part of the salt deposits during the mixing operation. Temperature is then allowed to fall to about +22°, and ammonium perchlorate is separated from the mother liquor before the sodium sulfate with 10 molecules of water of crystallization begins to crystallize.

The above mentioned method of producing ammonium perchlorate also offers other



advantages than those, which are due to the lower price of ammonium sulfate.

In the foregoing it is stated, that cold solutions may be used in the conversion, whereby steam is economized and; furthermore, the cost of installation will be very low as no crystallization cisterns are needed for the raw salt, which deposits directly during the mixing operation. Furthermore, the use of metallic vessels is objectionable when ammonium chlorid or ammonium nitrate are used as raw materials, as in this case the cisterns are acted upon by the liquid. If, on the contrary, ammonium sulfate be used, cisterns of lead may for instance be employed. The lead is in this case evidently protected against the action of the liquid by a covering of lead sulfate. When ammonium chlorid or ammonium nitrate is used, nitrous gases are easily evolved, which evidently depends on an intermediate formation of nitrogen-chlorid ( $\text{NCl}_3$ ). This is not only dangerous for the health of the men, but also causes a loss of valuable ammonia-nitrogen. This inconvenience is avoided when ammonium sulfate is used.

Sodium nitrate and sodium chlorid will be less soluble at lower temperature, while sodium sulfate as stated above becomes more easily soluble. When using the old methods a portion of the by-product deposits together with the ammonium perchlorate, while in the present method the by-product is deposited separately.

Having now described my invention, what I claim as new and desire to secure by Letters Patent is:

The herein described method of producing ammonium perchlorate which consists in re-acting upon sodium perchlorate with ammonium sulfate and crystallizing out the ammonium perchlorate while the temperature is not lower than  $22^\circ \text{C}$ ., substantially as described.

In witness whereof, I have hereunto signed my name in the presence of two subscribing witnesses:

OSCAR BIRGER CARLSON.

Witnesses:

AXEL EHRLICH,  
OSCAR GRAHN.