

# UNITED STATES PATENT OFFICE.

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## PROCESS OF MAKING ALKALINE PHOSPHATES.

SPECIFICATION forming part of Letters Patent No. 446,815, dated February 17, 1891.

Application filed November 15, 1890. Serial No. 371,595. (No specimens.)

*To all whom it may concern:*

Be it known that I, CHARLES GLASER, a citizen of the United States, residing at Baltimore, in the State of Maryland, have invented certain new and useful Improvements in the Manufacture of Phosphates of the Alkalies; and I do hereby declare the following to be a full, clear, and exact description of the invention, such as will enable others skilled in the art to which it appertains to make and use the same.

This invention relates to a new and useful process for manufacturing phosphates of the alkalies by the use of crude commercial phosphoric acid.

The use of crude commercial phosphoric acid in the manufacture of pure phosphates has heretofore presented considerable difficulties when it was desired to obtain these salts in a very pure state without resorting to expensive means of purifying either the acid used or the salts after formation. It is considered particularly difficult to get rid of the last traces of organic impurities and of sulphuric acid, which are always found in considerable quantities in crude phosphoric acid; but by the process hereinafter described such difficulties are easily and completely overcome, and it becomes possible to manufacture from crude phosphoric acid phosphates of the alkalies particularly free from the above-named impurities, without other purification than is accomplished by the process itself in its several stages.

The basis of my process lies in the two following reactions: First reaction: When the salt of an alkali and an acid volatile at higher temperature is fused with an amount of phosphoric acid more than sufficient to form the acid-pyrophosphate of that alkali until complete decomposition of the salt has been effected, a product results consisting of a mixture of either acid pyrophosphates or metaphosphates of the alkali, or both, and free metaphosphoric acid. Second reaction: When this product is dissolved in water and boiled for a sufficient time, the total phosphoric acid present is changed to orthophosphoric acid, (free and in combination,) and if when this is

completed I add enough of the carbonate of the same alkali to produce a slight alkaline reaction in the liquid the latter will contain the normal phosphate of that alkali, ready to be separated by crystallization or otherwise.

It is evident that by partial substitution of the salts of other alkalies in the material of this process I may obtain as product mixtures of their normal phosphates.

During the fusion described above in one all the organic impurities and any sulphuric acid may be expelled, while most impurities of an earthy character will remain insoluble and be separated upon dissolving, as described, in two.

To enable others skilled in the art to put my process in practice, I describe here its application in the best way known to me, taking, for example, nitrate of soda as the salt to be acted upon, remarking that the proportions given below may be considerably varied without essential difference in the result.

Take one part of nitrate of soda and about two and a half parts, by weight, of crude commercial phosphoric acid of 50° Baumé density, or the approximate equivalent of acid of other density. Heat this together in a suitable vessel to a temperature sufficient to expel all nitric and sulphuric acid, (the fumes of which may be condensed or used directly for other purposes,) but avoiding a temperature at which phosphoric acid would volatilize. The destruction of organic matter under these circumstances is complete, and if the total expulsion of sulphuric acid is not desired the process can be finished at a lower temperature by suspending the application of heat when the nitric acid has been expelled. The result will be a yellowish mass—a mixture of metaphosphoric acid, pyrophosphate of sodium, and earthy impurities. This after cooling is immersed in water, where it will be readily dissolved, excepting the earthy matter. This solution is now boiled until all the meta and pyrophosphoric acids are converted into orthophosphoric acid, which process will be rapidly completed, provided there is a considerable excess of free metaphosphoric acid in the product of fusion. Then add carbonate



of sodium, either solid or in solution, until the liquid gives a distinctly alkaline reaction, at which stage some more earthy impurities will be separated by precipitation.

5 Instead of carbonate of sodium caustic soda may be used, but will require more care to apply in the right amount, as an excess of the latter will cause the formation of trisodic phosphate, while an excess of the carbonate  
10 will not produce that result.

Separating the solution from the residuum by decantation, filtration, or otherwise, the now clean and colorless solution of phosphate of sodium is sufficiently evaporated and al-  
15 lowed to crystallize.

The salt thus obtained will be pure enough for most purposes, and can be easily freed from the last traces of foreign matter by re-crystallization.

20 What I claim as new is—

The process for the manufacture of pure phosphates of the alkalies from crude com-

mercial phosphoric acid by the following successive stages, viz: first, decomposing the salt of an alkali and an acid volatile at higher 25 temperature by fusing same with crude commercial phosphoric acid in excess of an amount required to form a pyrophosphate; secondly, dissolving the fused mass in water and boiling till conversion of meta and pyro phos- 30 phoric acid is effected; thirdly, treating with the carbonate of an alkali (or free alkali) till alkaline reaction is obtained, and finally separating the solution from insoluble impurities and obtaining from the former the pure 35 salts by crystallization or otherwise, all substantially as described above.

In testimony whereof I affix my signature in presence of two witnesses.

CHARLES GLASER.

Witnesses:

L. K. SPARROW,  
A. SEEL.