

# UNITED STATES PATENT OFFICE.

ERNST RUDOLF CASPARI AND OTTO NYDEGGER, OF UERDINGEN, AND ANTON GOLDSCHMIDT, OF DÜSSELDORF, GERMANY, ASSIGNORS TO R. WEDEKIND & CO., M. B. H., OF UERDINGEN, GERMANY, A FIRM.

## PRODUCTION OF AMMONIUM NITRATE.

No. 864,513.

Specification of Letters Patent.

Patented Aug. 27, 1907.

Application filed October 2, 1906; Serial No. 337,121.

To all whom it may concern:

Be it known that we, ERNST RUDOLF CASPARI, a subject of the King of Prussia, residing at Uerdingen-on-the-Rhine, Germany, OTTO NYDEGGER, a citizen of the Republic of Switzerland, residing at Uerdingen-on-the-Rhine, Germany, and ANTON GOLDSCHMIDT, a subject of the King of Prussia, residing at Düsseldorf, Germany, have invented certain new and useful Improvements in and Relating to the Production of Nitrate of Ammonium; and we do hereby declare the following to be a full, clear, and exact description of the invention.

Previous attempts to replace the usual process of formation of nitrate of ammonium from nitric acid and ammonia by a cheaper process of double decomposition from nitrate of soda with sulfate of ammonium have proved unsuccessful because the nitrate of ammonium produced has not possessed the desired purity.

Under a recent German patent (now expired) No. 149026 of February 3, 1903, the troublesome double salt formation of non-decomposed sulfate of ammonium with sulfate of sodium and nitrate of sodium is lessened by the application of a surplus of nitrate of sodium, which, however, for the purpose of economically carrying out the process must amount to at least one hundred per cent (100%) of the quantity theoretically necessary. But the Letters Patent makes no statement as to the production of pure nitrate of ammonium from this mixture of nitrate of ammonium, sulfate of sodium, and nitrate of sodium, and thus the object of the present process is not attained.

Another German Patent No. 166746 was granted on September 8, 1903 (the corresponding U. S. Patent No. 764251 on July 5, 1904) for a process which is impracticable as involving a costly artificial cooling step and as being subject to the hazard of the slight accident of jarring which injuriously affects the result. See page 3 line 54 et seq. of U. S. Patent 764251. The specification of this patent also discloses that in this process an unusual procedure is necessary to successfully separate the salts of sodium from the nitrate of ammonium.

In contrast to the older processes above described, our process makes it practically possible to carry out the double decomposition smoothly, to separate sulfate of sodium and nitrate of sodium as such, and to gain pure nitrate of ammonium directly.

The relations of solubility of the two salts of sodium were examined in the presence of nitrate of ammonium and the following results were obtained:

A mixed solution of nitrate of sodium and sulfate of ammonium, which contains at least twenty per cent (20%) more nitrate of sodium than is theoretically necessary for double decomposition, yields sulfate of sodium plen-

tifully on evaporation to a temperature of about 114°. If the evaporation is continued until the solution, at about 35° or at a higher temperature, is saturated with nitrate of ammonium, most of the sulfate of sodium formed by the double decomposition is crystallized out. This is due to the presence of nitrate of sodium nearly to saturation, in the solution, which not only causes the double decomposition to proceed smoothly but effects the crystallization of the sulfate of sodium. Now, this solution at a temperature near the point of crystallization of nitrate of ammonium—which takes place, according to the extent of evaporation, at about 35° or even at a higher temperature—crystallizes out quantities of nitrate of sodium sufficient for the success of the process, and a further portion of sulfate of sodium. For, in proportion to the amount of the surplus of nitrate of sodium, as well as in proportion to the hereinbefore mentioned degree of evaporation and the concentration of nitrate of ammonium in connection therewith, when the hot solution is cooled off, at about 50° or even at a higher temperature, complete saturation of the solution with nitrate of sodium ensues, which, besides the cooling, tends to separate the sulfate of sodium still in solution. So, also, the complete saturation of the solution with sulfate of sodium, which always exists during the cooling, tends to crystallize out the nitrate of sodium. This action is so noticeable that considerably more nitrate of sodium is crystallized than would be expected from its solubility in water. The portion of the sulfate of sodium and of the nitrate of sodium which has been crystallized in the manner described by mutual salting out from hot solutions supersaturated with both soda salts, are then removed from the solution. The portion of the soda salts still in solution therein has, as has been found furthermore, the desirable effect that far more nitrate of ammonium remains dissolved in the hot solution than would be expected from its solubility in water. On the other hand, when the temperature falls below +30°, less nitrate of ammonium remains in solution than corresponds to its solubility in water. From these relations of solubility there appeared a means of effecting a large yield of nitrate of ammonium, by applying temperatures above +35°, as above indicated, for the separation of the salts of soda from the solution almost saturated with nitrate of ammonium, and using temperatures below +35° for the separation of the nitrate of ammonium.

The solution almost saturated with nitrate of ammonium at 35° or at a higher temperature would, during the cooling, permit solid soda salts to be crystallized out as well as much nitrate of ammonium; but as the industry of explosives demands a very pure nitrate of ammonium, this invention aims at procuring pure nitrate of ammonium directly from the hot



solution. Such a pure product is secured in this process by adding so much water to the warm solution before cooling that the soda salts at the temperature to which they are cooled off—for example, at say 5 +20°—remain in solution. By experiments on a small scale it can be determined how much water is necessary to keep the nitrate of ammonium free from the crystallization of the soda salts. It generally suffices to add from one-fifth to one-seventh of the weight 10 of the solution.

The same success is not attained when impure nitrate of ammonium is re-crystallized, as is intended, for example, in the German Patent 166,746, inasmuch as in the resultant impure solution a large portion of 15 the nitrate of ammonium remains dissolved.

The process of this invention is illustrated in the following example: 645 parts of nitrate of sodium (of which 25% is surplus) and 400 parts of sulfate of ammonium are heated with 800 parts of water, so that 20 a complete double decomposition can take place. The mixture must then be evaporated until a sample of the solution, filtered off from the separated sulfate of sodium, on cooling to about 70° begins to separate nitrate of ammonium. The technical separation of 25 sulfate of sodium from the solution is facilitated, however, if the solution is filtered even before this concentration is reached, for instance, when it shows a boiling point of about 118°.

When the desired concentration has been attained, 30 the solution is cooled off to nearly 70°. In this case it is already saturated with nitrate of sodium at about 80°. After the crystallized nitrate of sodium and sulfate of sodium have been removed from the solu-

tion, the latter is mixed with so much water that, on further cooling off, to room-temperature, +15° to 20°, 35 the soda salts remain in solution. In the present example, about 16 parts of water are added to 100 parts of the solution obtained, in order to attain this end. The solution thus diluted is then cooled off to the desired temperature during which about 40% of the 40 nitrate of ammonium in the solution is separated free from soda salts. By "covering" with a solution of nitrate of ammonium, the same is liberated from the solution. The nitrate of ammonium thus produced yields 25 to 30% of the theoretical amount of this salt 45 and the purity of the product is 99 to 100%.

I claim as my invention

The process of producing pure nitrate of ammonium from nitrate of sodium and sulfate of ammonium, which consists in adding nitrate of sodium to a solution of sulfate of ammonium until the mixture is supersaturated 50 with the former at a temperature over about 49° C., cooling this solution to the temperature of about 35° C. just above the saturation point of nitrate of ammonium, thereby crystallizing out most of the sodium sulfate and sodium 55 nitrate drawing off the solution from the crystallized sodium salts, adding water equal to about one-fifth to one-seventh the weight of the solution, to keep the soda salts dissolved during the remainder of the process, cooling this solution to about 15° to 20° C. thereby crystallizing out 60 the ammonium nitrate and removing the nitrate of ammonium which has crystallized out.

In testimony whereof we have signed our names to this specification, in the presence of two subscribing witnesses.

ERNST RUDOLF CASPARI.

OTTO NYDEGGER.

ANTON GOLDSCHMIDT.

Witnesses:

W. BRUCE WALLACE.

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