

PATENT SPECIFICATION

336,065

Application Date: Sept. 6, 1929. No. 27,108/29.

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PROVISIONAL SPECIFICATION.

Improvements in the Production of Phosphorus Oxychloride.

We, JOHN STANLEY DUNN and FRANK BRIERS, both British Subjects, both of Norton Hall, The Green, Norton-on-Tees, County Durham, 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 preparation of phosphorus oxychloride from phosphates of calcium such as rock phosphate or double superphosphate.

In our co-pending application No. 27109/29 we have described a method of obtaining phosphorus oxychloride from rock phosphate etc. in which the material is treated to remove calcium chloride formed initially by the action of phosgene upon calcium carbonate present in the raw material and the purified calcium phosphate is then submitted to the action of phosgene at a raised temperature.

According to the present invention the phosphatic material is treated with phosgene or with carbon monoxide and chlorine or chlorine alone in the presence of activated carbon, at a raised temperature and under such conditions that the calcium chloride formed is prevented from covering the particles of initial material so that new surfaces are constantly exposed to the action of phosgene.

Various methods are available according to our invention for accomplishing this end. For instance the raw phosphates may be treated with phosgene whilst being constantly subjected to grinding or stirring. An inert solid such as coke or the washed residue from a previous operation may be added to the phosphatic material and the mixture agitated continuously or from time to time in order to remove the deposited calcium chloride and expose new surfaces to the reaction.

The process may also be conducted in a rotary kiln in which the ground phosphatic material, if necessary with the addition of pebbles or similar bodies, is constantly agitated during the reaction. In each

case the mass may, if necessary, be washed with water at intervals and the residual solid after drying subjected to the renewed action of phosgene. It is important that the initial material whether consisting of phosphate alone or of phosphate mixed with coke or other solids should be dry. Instead of using phosgene in the above processes a mixture of carbon monoxide and chlorine or chlorine alone may be used if the phosphatic material be mixed with activated carbon. The use of chlorine alone necessitates a slightly higher temperature for the reaction. When coke is used, it may be activated, for example by preliminary treatment with chlorine at a high temperature, e.g. 1000° C. after which it is washed and dried before mixing with the raw material. A mixture of carbon monoxide and chlorine may then be used in place of phosgene.

EXAMPLE 1.

Dry ground rock phosphate mixed with an equal quantity of coke which has previously been activated is heated to 350° C. and a mixture of carbon monoxide and chlorine is passed through, the solid material being constantly agitated during the passage of the gas. Whilst the reaction is proceeding the temperature is gradually raised to 600° C. A yield of 90-100 per cent. of phosphorus oxychloride is obtained.

EXAMPLE 2.

75 parts of double superphosphate are mixed with 25 parts of activated coke and the mixture treated with agitation at 350°-600° with phosgene. A yield of 90 per cent. of phosphorus oxychloride is obtained.

Dated this 6th day of September, 1929.

W. P. THOMPSON & Co.

Chartered & Registered Patent Agents,

12, Church Street, Liverpool, and

50, Lincoln's Inn Fields, London.

W. C. 2.

Agents for the Applicants.

COMPLETE SPECIFICATION.

Improvements in the Production of Phosphorus Oxychloride.

We, JOHN STANLEY DUNN and FRANK BRIERS, both British Subjects, both of

Norton Hall, The Green, Norton-on-Tees, County Durham, 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 preparation of phosphorus oxychloride from phosphates of calcium such as rock phosphate or double superphosphate.

In our co-pending application No. 27109/29 we have described a method of obtaining phosphorus oxychloride from rock phosphate etc. in which the material is treated to remove calcium chloride formed initially by the action of phosgene upon calcium carbonate present in the raw material and the purified calcium phosphate is then submitted to the action of phosgene or its known equivalents at a raised temperature.

According to the present invention the phosphatic material is treated with phosgene or its equivalents at a raised temperature and under such conditions that the calcium chloride formed is prevented from covering the particles of initial material so that new surfaces are constantly exposed to the action of phosgene.

Various methods are available according to our invention for accomplishing this end. For instance the raw phosphates may be treated with phosgene whilst being constantly subjected to grinding or stirring. An inert solid such as coke or the washed residue from a previous operation may be added to the phosphatic material and the mixture agitated continuously or from time to time in order to remove the deposited calcium chloride and expose new surfaces to the reaction.

The process may also be conducted in a rotary kiln in which the ground phosphatic material, if necessary with the addition of pebbles or similar bodies, is constantly agitated during the reaction. In each case the mass may, if necessary, be washed with water at intervals and the residual solid after drying subjected to the renewed action of phosgene. It is important that the initial material whether consisting of phosphate alone or of phosphate mixed with coke or other solids should be dry. Instead of using phosgene in the above processes we may use its known equivalents. Thus a mixture of carbon monoxide and chlorine or

chlorine alone may be passed over the phosphatic material mixed with activated carbon. The use of chlorine alone necessitates a slightly higher temperature for the reaction. When coke is used, it may be activated, for example by preliminary treatment with chlorine at a high temperature, e.g. 1000° C. after which it is washed and dried before mixing with the raw material.

EXAMPLE 1.

Dry ground rock phosphate mixed with an equal quantity of coke which has previously been activated is heated to 350° C. and a mixture of carbon monoxide and chlorine is passed through, the solid material being constantly agitated during the passage of the gas. Whilst the reaction is proceeding the temperature is gradually raised to 600° C. A yield of 90–100 per cent. of phosphorus oxychloride is obtained.

EXAMPLE 2.

75 parts of double superphosphate are mixed with 25 parts of activated coke and the mixture treated with agitation at 350°–600° with phosgene. A yield of 90 per cent. of phosphorus oxychloride is obtained.

We do not broadly claim the reaction between phosphate and phosgene or equivalents but only under the condition above stated.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is:—

1. The production of phosphorus oxychloride from phosphate and phosgene or its equivalents, characterised by the feature that fresh surfaces of phosphate are constantly exposed to the gas.

2. Process as claimed in claim 1 in which the mixture is ground, stirred or agitated.

3. Process as claimed in claim 1 in which the treated mixture is washed with water dried and again subjected to reaction.

4. Process of manufacturing phosphorus oxychloride, substantially as described.

Dated this 5th day of June, 1930.

W. P. THOMPSON & Co.
12, Church Street, Liverpool,
Chartered & Registered Patent Agents,
Agents for the Applicants.