

PATENT SPECIFICATION

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COMPLETE SPECIFICATION.

Improved Manufacture of Phosphorus Chlorides.

- I, EDOUARD URBAIN, a Citizen of the French Republic, of 6, Rue Eyautey, Paris, France, 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:—
- It is known that the preparation of ferro-phosphorus from natural phosphates is easier and more economical than the preparation of phosphorus itself. The present invention relates to a process of manufacturing phosphorus chlorides from ferro-phosphorus.
- When ferro-phosphorus is treated with a current of dry chlorine, the reaction of the chlorine on the ferro-phosphorus takes place at once and produces penta-chloride of phosphorus and ferric chloride; but the separation of these substances cannot be effected by distillation owing to the existence of a molecular compound PCl_5 , FeCl_3 , which passes without decomposition. Nevertheless, the separation of the phosphorus and iron becomes possible by taking into account a new discovery of great technical value, connected with the action of the molecular compound obtained on a fresh quantity of ferro-phosphorus.
- The process according to the invention consists in treating ferro-phosphorus with dry chlorine to the maximum chlorination, in adding, before a red heat is reached, a fresh quantity of ferro-phosphorus, and in raising the temperature until the phosphorus tri-chloride distils.
- The action of the fresh quantity of ferro-phosphorus results in the formation of ferrous chloride and phosphorus trichloride according to the equation: $3\text{FeCl}_2 \cdot \text{PCl}_5 + (\text{Fe} + \text{P}) = 2\text{Fe}_2\text{Cl}_6 + 4\text{PCl}_3$. These are then easy to separate by distillation. It has been found moreover that ferrous chloride itself reacts in turn on the phosphorus penta-chloride, with the formation of ferric chloride and phosphorus trichloride. These reactions render possible a quantitative separation of the iron and phosphorus, the latter being in the form of phosphorus tri-chloride, which may be eventually converted into penta-chloride by the action of chlorine.
- The process may also be carried out by
- using a limited quantity of dry chlorine and by producing, by a suitable elevation of temperature, the reaction between the penta-chloride formed and the excess of unattacked ferro-phosphorus, as well as the distillation of the tri-chloride thus obtained.
- The following manner of carrying the invention into effect is given by way of example: Ferro-phosphorus is treated with chlorine at any convenient temperature (the reaction takes place either at the ordinary temperature or at a high temperature without modification of the products thus obtained). The heat produced results in the temperature being always high enough to effect the distillation of FeCl_3 and $\text{PCl}_5 \cdot \text{FeCl}_3$.
- Consequently two methods may be used. According to one method the distillation is allowed to occur and the products of the reaction are condensed. According to the other method a reflux apparatus is used in order to keep the products of the reaction in contact with the unattacked ferro-phosphorus.
- In the first case, ferro-phosphorus is added to the products of the distillation and the mixture is heated in a reflux apparatus without allowing the temperature of the escaping gases to fall below 80° to 100° C. The mixture may be heated below 200° C. in order to volatilise only a small part of the ferric chloride or to 300° C. in order to volatilise all the ferric chloride. In both cases, the ferric chloride is condensed in the reflux apparatus and returned over the ferro-phosphorus, while the phosphorus trichloride formed is not condensed in the reflux apparatus, but is recovered by condensation outside the above apparatus.
- If the reaction itself has taken place in the reflux apparatus, the current of chlorine is stopped when the ferro-phosphorus has been converted into ferrous chloride and phosphorus trichloride. In reality the result of the reaction is not FeCl_3 , and PCl_5 , but FeCl_2 , $\text{FeCl}_3 \cdot \text{PCl}_5$ and some unattacked ferro-phosphorus. A further heating of the mixture will dissolve the remaining ferro-phosphorus, and phosphorus trichloride will be recovered as above stated.

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

1. A process for the industrial manufacture of phosphorus chlorides, consisting in treating ferro-phosphorus with dry chlorine to the maximum chlorination, in adding, before a red heat is reached, a fresh quantity of ferro-phosphorus, and in raising the temperature until the phosphorus tri-chloride distils.

2. A process for the industrial manufacture of phosphorus chlorides, consisting in treating ferro-phosphorus with a limited quantity of dry chlorine, and in

producing, by an increase of temperature, the reaction of the penta-chloride of phosphorus formed, on the excess of ferro-phosphorus not attacked by the chlorine, until the phosphorus tri-chloride thus obtained is distilled.

3. The process of manufacturing phosphorus chlorides substantially as described.

4. Phosphorus chlorides when manufactured by the process claimed in any of the preceding claims.

Dated this 3rd day of May, 1929.

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