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MANUFACTURE OF PHOSPHORUS AND COMPOUNDS THEREOF

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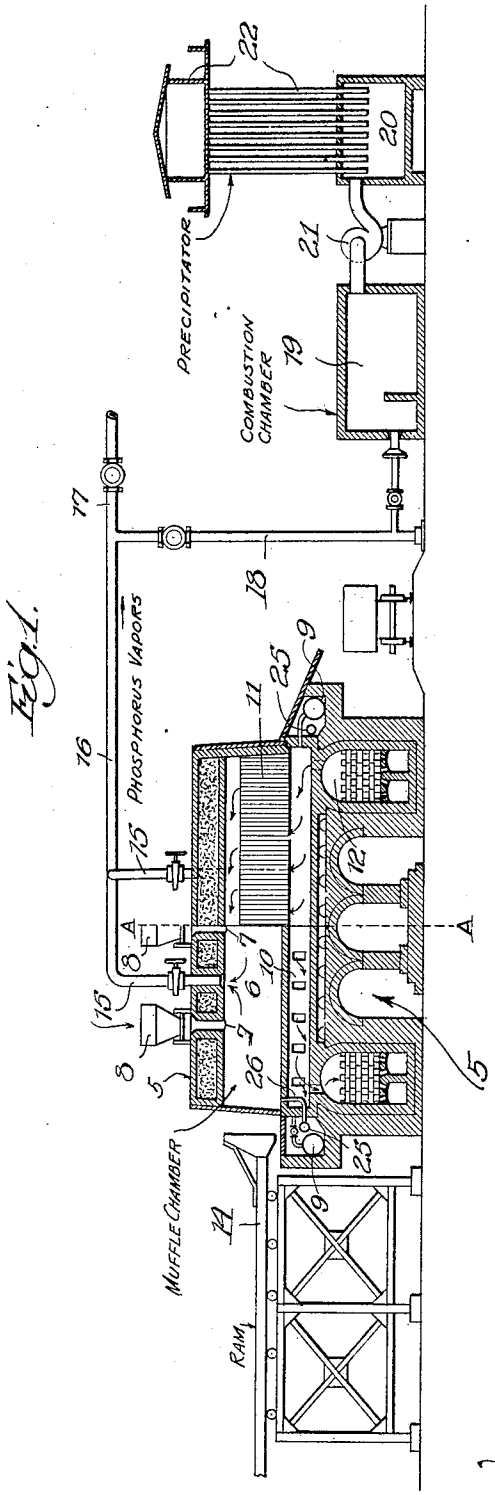


Fig. 1.

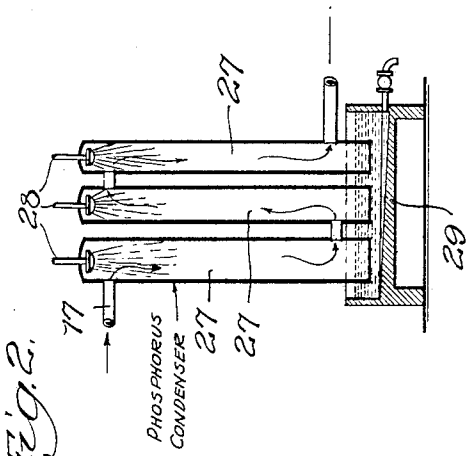


Fig. 2.

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MANUFACTURE OF PHOSPHORUS AND COMPOUNDS THEREOF

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The present invention relates to improvements in the manufacture of phosphorus or of compounds thereof, and will be fully understood from the following description, illustrated by the accompanying drawings, in which:

Figure 1 is a diagrammatic view, partially in section, showing apparatus suitable for use in carrying out the present invention; and

Fig. 2 is a diagrammatic sectional view of condensing apparatus suitable for use in connection with the present invention, as hereinafter set forth.

In accordance with the present invention, the production of phosphorus and compounds thereof from phosphatic materials is facilitated and the effective recovery thereof substantially increased. In the preferred method of carrying out the invention, conditions may be so maintained that an effective recovery of phosphorus is secured without the disadvantage of bringing the reaction materials to the fusion point of the mixture.

Referring more particularly to the drawings, the numeral 5 indicates an oven or furnace of the by-product coke oven type, that portion on the left of the vertical line A—A being shown in section through the retort or muffle chamber of a bench, and that portion of the right of the line A—A being a sectional view through the heating flues interposed between muffles or retorts in the bench. The retort shown may be regarded as one in a bench of retorts, certain of which are employed in the manufacture of by-product coke in the usual manner, that illustrated being employed in association therewith for the manufacture of phosphorus or phosphorus compounds as hereinafter more fully set forth.

The retort or muffle chamber 6 is charged with the phosphatic reaction mixture by the method usual in charging coke ovens with coke; that is, the reaction mixture is charged thereinto through the ports 7 from suitable hopper cars 8. The individual retorts or ovens of the bench, which are relatively narrow, are heated in the customary manner, by means of a suitable fuel gas supplied through the mains 9 to the flues 10 below the ovens and communicating in turn with the flues 11. Preheated air for combustion is supplied to the flues from the regenerators 12, the operation of which is not here described, as it is that conventional in coke oven practice. The retorts 6 are provded with discharge pipes 15, through which the phosphorus vapors given off by the charge pass out to be handled as hereinafter more fully described. A suitable ram 14 is provided for forcing the spent charge from the retort after completion of the reaction.

In carrying out the invention, the phosphatic charge is prepared for the retorts by admixing previously ground phosphate rock and bituminous coal, both being preferably ground so that 90% thereof will pass through a 40 mesh screen. The mixture is so made (with the addition of suitable quantities of sand, if required) that the resulting charge has approximately the following constitution.

Silica (SiO_2) 18 to 20%.
(CaO) 34 to 37%.
Phosphoric acid (P_2O_5) 22 to 25%.
Bituminous coal 18 to 25%.

This mixture is then employed as the charge in the retorts 6. Before charging this mixture into the retorts, it is preferred that a small quantity of bituminous coal be charged thereinto to form a layer extending over the base or bottom of the oven, the phosphatic charge being then filled in over this layer of bituminous coal.

The ovens having been charged, they are brought to a temperature between 1100° and 1450° C., at which temperature the reaction takes place and phosphorus is evolved. The charge of the proportions hereinbefore set forth is not reduced to a molten condition, nor does it do more than sinter at temperatures as high as 1450° C. The phosphorus is therefore evolved under these conditions without fusion of the reaction mixture. The relatively narrow width of the retorts or ovens permits of a very even distribution of heat through the charge.

In accordance with this invention, bituminous coal is employed as the carbonaceous

constituents of the charge, and as a result of its use there is a shrinkage of the charge during reaction which substantially facilitates the removal of the spent charge from the furnaces. The employment of a bottom layer of bituminous coal in the retorts, as above described, aids substantially in preventing adhesion of the charge thereto. Although it is preferred to use this bottom layer of bituminous coal, it may be omitted if desired, the proportion of bituminous coal in the charge and the resulting shrinkage reducing substantially the adherence of the charge to the walls of the oven. During reaction, a suitable non-oxidizing gas, such as nitrogen, carbon monoxide or coke-oven gas may be caused to pass through the retorts, thus aiding in sweeping out the phosphorus vapors. Thus, in the apparatus herein described, coke-oven gas derived from other ovens of the bench may be supplied through the main 25 and the pipe 26 to the interior of the retorts 6 in which the phosphatic charge undergoes treatment. A current of the non-oxidizing gas thus supplied is preferably passed through the reaction mixture during the entire period of evolution of phosphorus therefrom.

The phosphorus vapors, together with the non-oxidizing gas when the latter is employed, pass out through the pipes 15 into the discharge main 16. If phosphorus is desired as the final product, the vapors are caused to pass from the main 16 through the valve controlled line 17 to the condensing apparatus illustrated diagrammatically in Fig. 2. If it is desired to oxidize the phosphorus vapors to form phosphorus pentoxid and phosphoric acid, the line 17 is closed off and the phosphorus vapors are caused to pass through the line 18 into the combustion chamber 19, in which sufficient air is also supplied to effect the desired oxidization. The resulting gases, including phosphorus pentoxid and phosphoric acid, pass into a collecting chamber 20, being suitably forced thereinto by a pump 21. Loss of phosphorus pentoxid is prevented by the precipitator 22, suitably of the Cottrell type.

The operation of the invention in connection with a coke-oven bench permits of the ready application of the ammoniacal liquors from the coke ovens to the neutralization of the phosphates. The ammoniacal liquors may be purified to any desired extent before their employment in the formation of the ammonium phosphates.

As indicated in Fig. 2, the phosphorus vapors from the retorts may be caused to pass through a series of suitable condensing towers 27, each provided with a condensing water spray 28 and opening at its bottom below the water level in a collector 29. The latter has a properly drained bottom, so that the collected phosphorus may be readily withdrawn therefrom.

Although the present invention has been described in connection with particular details of apparatus and of a method for carrying it into effect, it is not intended that these details shall be regarded as limitations upon the scope of the invention, except insofar as included in the accompanying claims.

We claim:

1. The method of producing phosphorus from a phosphatic material which comprises preliminarily applying to an interior wall of an externally heated chamber a layer of bituminous coal, subsequently charging the chamber with a phosphatic material admixed with silica and a carbonaceous material, and applying heat to remove the phosphorus from the charge, adherence of the charge to the wall of the chamber being thereby prevented.

2. The method of producing phosphorus which comprises heating in an externally fired chamber to a temperature between 1100° and 1450° a mixture of natural phosphate, silicious material and bituminous coal, the proportion of bituminous coal being in excess of 18% and that of silica not being above 20%.

3. The method of producing phosphorus from a phosphatic material which comprises preliminarily applying to an interior wall of an externally heated chamber a layer of bituminous coal, subsequently charging the chamber with a phosphatic material admixed with silica and a carbonaceous material, and applying sufficient heat to remove the phosphorus from the charge without fusing said charge, adherence of the charge to the wall of the chamber being thereby prevented.

4. The method of producing phosphorus from a phosphatic material which comprises preliminarily applying to an interior wall of an externally heated chamber a layer of bituminous coal, subsequently charging the chamber with a phosphatic material admixed with silica and bituminous coal, and applying sufficient heat to remove the phosphorus from the charge without fusing said charge, adherence of the charge to the wall of the chamber being thereby prevented.

5. The method of producing phosphorus which comprises heating a phosphatic reaction mixture comprising a natural phosphate, silica and carbonaceous material in a chamber, separately and simultaneously heating a coking coal to a coking temperature, thereby evolving coal gas therefrom, and drawing said coal gas through the heated phosphatic reaction mixture.

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