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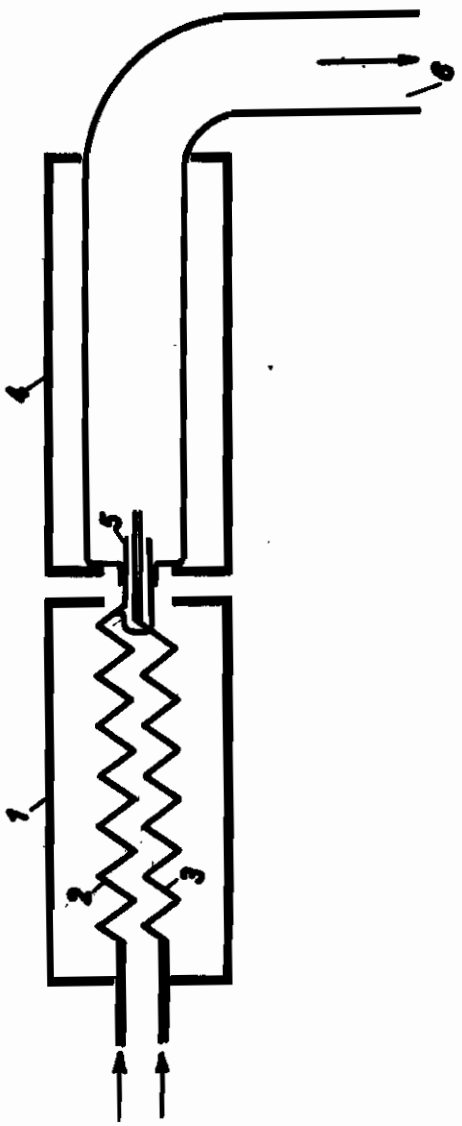

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ALIEN PROPERTY CUSTODIAN

MANUFACTURE OF TITANIUM DIOXIDE

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It is known that when titanium halogenides (that is to say binary compounds of titanium with a halogen) are caused to react with gases containing free oxygen, at temperatures above 1000° C, titanium dioxide and the halogen (chlorine for instance) are obtained. But crystallization effects often occur in the course of this manufacture of titanium dioxide and they are particularly disturbing when it is desired to obtain pigments.

According to a known method, these effects may be attenuated by causing the gaseous mixtude of the titanium halogenide and the oxygen containing gas to flow as quickly as possible through the heated reaction chamber. Also, care should be taken that the gases reach as quickly as possible the temperature of reaction of the titanium tetra-halogenide with oxygen.

The object of the present invention is to provide a method and apparatus for avoiding the injurious effects of crystallization in a different way.

I have found that it is possible to obtain titanium pigments of a particular value and to prevent the pigment particles from becoming too big by causing the gases to react in a vessel in a reaction space as small as possible and by providing, between the reaction zone and the wall of said vessel a difference of temperature such that the temperature close to said wall is lower than the temperature of reaction of the halogenide with the oxygen.

Therefore, according to the present invention, the two gases which are to react on each other as above stated are heated separately, to a temperature higher than the temperature of reaction, and then brought into the presence of each other, so as to react, by passing through a tube of suitable structure.

This tube is for instance mounted in a chamber maintained, from the outside, at a temperature lower than that at which the gases are initially heated. The reaction which takes place, and which (supposing that the halogen is chlorine) is the following:

$$TiCl_4+O_2=TiO_2+2Cl_2$$

is slightly exothermic. Therefore, after the mixture has been ignited, the combustion continues, even if the temperature of the wall of the reaction chamber is lower than the reaction temperature. The heat that is given off by the reaction prevents too great a cooling.

Owing to this arrangement, the transformation of the titanium product takes place in the central portion of the tube where the reaction gases are mixed to each other. The reaction product which reaches the colder wall of the chamber cannot, in this case, condensate into relatively big crystalline conglomerates, whereby the injurious crystallization effects are avoided. With a suitable ratio of the respective amounts of gases mixed together in the tube, I obtain a hundred per cent reaction inside the small space within which the gases are mixed together, generally with the formation of a yellow-green flame. When the gas with which the titanium halogenide is caused to react is pure oxygen, a hundred per cent reaction is very easily obtained, producing a bright light, with a proportion of tetrahalogenide to oxygen of 1 to 4. Of course, analogous results may be obtained with different proportions. According to the proportions of the elements caused to react on each other, various results may be obtained, for instance a hundred per cent reaction without formation of flame. Instead of employing pure oxygen, I may also employ air on any other gaseous mixture containing oxygen.

Other features of the present invention will result from the following detailed description of some specific embodiments thereof.

A preferred embodiment of the present invention will be hereinafter described with reference to the accompanying drawing, given merely by way of example, and in which:

The only figure is a diagrammatical view of an apparatus for carrying out the method according to the invention.

Oven 1 is for instance heated to a temperature of 1000–1100° C, preferably through electric means. The quartz coiled pipe 2 serves to the feed of oxygen, heated by flowing therethrough, while the coiled pipe 3 serves to the feed of titanium tetrachloride. Thus the two compounds which are intended to react together are heated separately to the necessary reaction temperature. At the inlet of oven 4, where the reaction is to take place, there is a tube 5 for mixing the gases and producing the combustion thereof. This oven 4 is kept at a temperature of 750° C. The amount of titanium tetrachloride fed for the reaction is, owing to the adjustable heating, such that the proportion of halogenide with respect to the amount of oxygen is 1 to 4. During the reaction, a yellow-green flame burns from the outlet of tube 5 and a pigment smoke issues from the oven which smoke is separated from the chlorine by depositing the titanium dioxide in vessels 6 conpected to the furnace.

The pigment that is obtained is very voluminous and has a coloring power which corresponds to those of the best pigments of titanium dioxide. The grain is very uniform and the shade a pure white.

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