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DECOLORIZING TITANIUM TETRACHLORIDE

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13 Claims. (Cl. 23—87)

This invention relates to the decolorizing of titanium tetrachloride. While pure titanium tetrachloride is a water white liquid, the product which is obtained by chlorination of titanium compounds and the commercial products generally available on the market have a distinct yellowish cast. Prior to this invention, decolorizing this liquid has been attempted by treatment with certain metals, such as reduced iron or sodium amalgam. Some decolorizing may be secured in this manner, but in general, it is found that the treated liquid does not remain colorless permanently and that it is contaminated with metallic impurities. This is highly undesirable when titanium tetrachloride is being decomposed into titanium dioxide pigment, since the metallic impurities tend to reduce the whiteness of the pigment produced.

In accordance with my invention, I have found that titanium tetrachloride may be decolorized to form a water white liquid by treatment with lower valent titanium compounds. I have found that especially effective results may be obtained by treatment with chlorides of titanium, such as titanium trichloride, or titanium dichloride. other titanium compounds which may be found suitable are titanium tribromide, titanium triiodide, titanous sulfate, etc. However, it is preferred to use the lower valent chlorides of titanium in order to avoid contamination of the material.

The treatment may be secured by any convenient method which will insure the required contact. I have found that very satisfactory results may be obtained by vigorously agitating a mixture of titanium tetrachloride and titanium trichloride while heating under a reflux condenser and subsequently distilling and condensing the titanium tetrachloride.

Since the tetrachloride is readily hydrolyzed in a moist atmosphere, it is preferred to carry this process out in the presence of a dry inert gas. Dry carbon dioxide or nitrogen are suitable for this purpose although other gases may be resorted to.

The concentration of the lower valent titanium salt required depends to a great extent upon the color of the product being treated. In general, it is preferred to maintain an excess of this salt, and it has been determined that concentrations in excess of about 2.0% titanium trichloride yield satisfactory products in most cases.

The time for which the mixture is refluxed likewise depends upon the degree of coloration of the product being treated. In general, refluxing at a temperature of 125–135° C. for several hours, followed by subsequent distillation is suitable. Heating at lower temperatures may also be satisfactory and in some cases heating may be dispensed with. The residue remaining after distillation will, in general, contain more or less titanous salts and may be reused in a subsequent treatment of further amounts of titanium tetrachloride.

The following examples are illustrative:

Example I.—100 parts of yellow titanium tetrachloride and 5 parts of titanium trichloride were vigorously agitated and heated under a reflux condenser at a temperature of 135° C. for a period of two hours. The treated mixture was then distilled rapidly in an atmosphere of dry CO_2. The product produced was water white.

Example II.—100 parts of yellow titanium tetrachloride and 3 parts of titanium trichloride were vigorously agitated and heated under a reflux condenser at a temperature of 135° C. for a period of four hours. The treated mixture was then distilled rapidly in an atmosphere of dry CO_2. The product produced was water white.

Example III.—100 parts of yellow titanium tetrachloride and 5 parts of titanium dichloride were vigorously agitated and heated under a reflux condenser at a temperature of 135° C. for a period of two hours. The treated mixture was then distilled rapidly in an atmosphere of dry nitrogen. The product produced was water white.

The titanium compound may be introduced into the tetrachloride in any convenient manner. By one very effective method, I may reduce a portion of the tetrachloride by suitable means, for example, by introduction of hydrogen, thereby generating the lower valent chloride in situ. However, the compound may be added in the form of a solid or as a solution as desired. Aqueous solutions of the compound preferably should be avoided due to the rapidity with which the tetrachloride hydrolyzes.

Although the invention has been described in connection with the specific details of certain embodiments thereof, it is not intended that such details shall be regarded as limitations upon the scope of the invention except insofar as included in the accompanying claims.

I claim:

1. A method of decolorizing colored titanium tetrachloride which comprises contacting the same with a lower valent titanium compound in an amount sufficient and for the time required

to remove at least a substantial portion of the color from the tetrachloride.

2. A method of decolorizing colored titanium tetrachloride which comprises contacting the same with a lower valent titanium halide in an amount sufficient and for the time required to remove at least a substantial portion of the color from the tetrachloride.

3. A method of decolorizing colored titanium tetrachloride which comprises contacting the same with a lower valent titanium chloride in an amount sufficient and for the time required to remove at least a substantial portion of the color from the tetrachloride.

4. A method of decolorizing colored titanium tetrachloride which comprises contacting the same with titanium trichloride in an amount sufficient and for the time required to remove at least a substantial portion of the color from the tetrachloride.

5. A method of decolorizing colored titanium tetrachloride which comprises contacting the same with titanium dichloride in an amount sufficient and for the time required to remove at least a substantial portion of the color from the tetrachloride.

6. A method of decolorizing colored titanium tetrachloride which comprises heating the same in the presence of a lower valent titanium compound in an amount sufficient to remove at least a substantial portion of the color from the tetrachloride and subsequently distilling the heated titanium tetrachloride.

7. A method of decolorizing colored titanium tetrachloride which comprises heating the same in the presence of a lower valent titanium halide in an amount sufficient to remove at least a substantial portion of the color from the tetrachloride and subsequently distilling the heated titanium tetrachloride.

8. A method of decolorizing colored titanium tetrachloride which comprises heating the same in the presence of a lower valent titanium chloride in an amount sufficient to remove at least a substantial portion of the color from the tetrachloride and subsequently distilling the heated titanium tetrachloride.

9. A method of decolorizing colored titanium tetrachloride which comprises heating the same in the presence of titanium trichloride in an amount sufficient to remove at least a substantial portion of the color from the tetrachloride and subsequently distilling the heated titanium tetrachloride.

10. A method of decolorizing colored titanium tetrachloride which comprises heating the same in the presence of titanium dichloride in an amount sufficient to remove at least a substantial portion of the color from the tetrachloride and subsequently distilling the heated titanium tetrachloride.

11. A method of decolorizing colored titanium tetrachloride which comprises heating the same in the presence of titanium dichloride in an amount sufficient to remove at least a substantial portion of the color from the tetrachloride and subsequently distilling the heated titanium tetrachloride and decolorizing further quantities of titanium tetrachloride by treating said quantities with the residue from said distillation.

12. A method of decolorizing colored titanium tetrachloride which comprises heating the same with a lower valent titanium compound in an amount sufficient and for the time required to remove at least a substantial portion of the color from the tetrachloride.

13. A method of decolorizing colored titanium tetrachloride which comprises heating the same with titanium trichloride in an amount sufficient and for the time required to remove at least a substantial portion of the color from the tetrachloride.

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