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PROCESS FOR PURIFICATION OF PENTAERYTHRITOL TETRANITRATE

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6 Claims. (Cl. 260—144)

This invention relates to a process for purifying normally solid organic explosive compounds by removal of the impurities by means of suitable organic solvents. It relates more particularly to purification of such compounds by treatment with solvents not miscible with water and having relatively high boiling points, in which case the final traces of solvent are removed from the compound by steam distillation.

The manufacture of solid explosive organic nitrocompounds, or organic nitrates, which are the substances covered by my invention, involves the nitration of the raw material with nitric acid, either alone or together with sulfuric acid. The solid nitration product is removed from the spent acid by filtration, decantation or other method, and is washed with water, and frequently with a dilute alkaline solution, for example ammonia, or an aqueous solution of sodium carbonate.

The product obtained above often contains an undesirable amount of impurities, particularly impurities of an acid nature due to the inclusion of foreign acidic material within the crystals. The presence of such impurities has an undesirable effect on the stability of the explosive product, as shown by various heat tests, etc.

The object of my invention is to provide an improved process for the purification of pentaerythritol tetranitrate, trinitrotoluene, tetryl and other normally solid organic explosive compounds. A further object is to provide a process for the purification of such compounds using a solvent giving off no considerable amounts of inflammable vapors, at ordinary temperatures, to form explosive mixtures with air. A still further object is to provide a process using a solvent immiscible with water but capable of removal from the purified material by distillation with steam. Other objects will be apparent as the invention is subsequently described.

I have discovered that the above objects may be attained if the organic explosive compounds to be purified are dissolved in a solvent which is not miscible with water and which has a boiling point over 100° C., the compounds to be purified being isolated from the solution after free acid and any insoluble impurities have been removed therefrom. The free acid may be removed by washing with water or alkaline solutions, and before or after removal of the insoluble impurities by filtration or other means. While various solvents are adapted for such use, for example high boiling point aliphatic hydrocarbons, aniline, aromatic hydrocarbons such as xylene, and like compounds, I prefer to use nitrobenzene.

The solubility of pentaerythritol tetranitrate, for example, in nitrobenzene is such as to make the latter solvent a desirable one for use. Since this solvent has a high boiling point, the vapor pressure is low at ordinary temperatures, and explosive mixtures are not formed with air. After purification of the pentaerythritol tetranitrate by nitrobenzene and removal of the impurities by filtration, the nitrobenzene may be removed from the explosive compound by steam distillation.

My process, as outlined above, may be applied to any one of a number of normally solid organic explosive compounds, for example tetryl, trinitrotoluene, trinitrobenzene, trinitronaphthalene, tetranitroaniline, pentaerythritol tetranitrate etc.

A further improvement in my process may be introduced by the use of a mild alkali dissolved in the water used for washing the solution of the organic explosive compounds; for example ammonia or a dilute sodium carbonate solution may be used. Thus when the organic solvent is steam distilled off it may be conveniently done in presence of a dilute alkaline solution. If desired, ammonia may be introduced into the distillation liquid by addition to the steam entering the vessel. The use of the above alkali insures the complete elimination of acidity from the explosive compound being purified.

By way of illustration the following specific example is given to illustrate my invention more fully.

Twenty-five lbs. of moist crude pentaerythritol tetranitrate, containing 5 lbs. of water, are brought into solution in 100 lbs. of nitrobenzene by agitation at a somewhat elevated temperature. The hot mixture is filtered to remove the solid undissolved impurities, and the filtrate is then steam distilled, together with a considerable quantity of a warm 1% sodium carbonate solution, for example. The distillation is continued until the condensate is free from nitrobenzene. At this time the aqueous solution should still be alkaline.

With the removal of the nitrobenzene from the distillation vessel, the purified pentaerythritol tetranitrate separates out. The crystals are filtered, washed, and dried at 100° C.

Pentaerythritol tetranitrate, produced according to the above process, possesses a high degree

of purity and shows high stability when tested by the usual heat tests.

The method of purification described heretofore has many advantages over the acetone purification process generally used according to the prior art. Acetone is a very volatile solvent giving off inflammable vapors, so that the use of hot acetone as a purifying agent adds a fire risk to the process. In addition, there is a loss of solvent by evaporation during the filtration of the solution from impurities, which frequently causes clogging of the filter because of premature crystallization of the explosive compound. Moreover, such crystals prematurely formed are not sufficiently pure and must be subsequently redissolved.

A further advantage in my process is that the solvent used is obtained free from water after the steam distillation, and can be used subsequently without further treatment. In the case of acetone, however, considerable difficulty is involved in separating the acetone from the water and obtaining a solution sufficiently concentrated for further use in the process. While I have described my invention in the foregoing with particular reference to pentaerythritol tetranitrate, it should be understood that it applies also to processes for purifying other organic explosive compounds, such as for example trinitrobenzene, trinitrotoluene, tetryl, trinitronaphthalene, tetranitroaniline, etc. It should be understood also that other compounds than nitrobenzene may be used as purifying agents, the only requirement being that they have solvent power for the explosive compounds, that they be not miscible with water, and that they have an elevated boiling point, above 100° C.

I claim:

1. The process of purifying pentaerythritol tetranitrate, which comprises crystallizing said compound from nitrobenzene.

2. The process of purifying pentaerythritol tetranitrate, which comprises crytallizing said compound from nitrobenzene, the nitrobenzene being subsequently recovered by distillation with steam.

3. The process of purifying pentaerythritol tetranitrate, comprising the crystallization of said compound from nitrobenzene, the nitro benzene being subsequently separated from the pentaerythritol tetranitrate by distillation with steam, said steam distillation being carried out in the presence of an alkaline reagent.

4. The process of purifying pentaerythritol tetranitrate comprising the crystallization of said compound from nitrobenzene, the nitrobenzene being subsequently separated from the pentaerythritol tetranitrate by distillation with steam, said steam distillation being carried out in the presence of ammonia.

5. The process of purifying pentaerythritol tetranitrate obtained by nitrating pentaerythritol, which comprises treating it with a solvent which is substantially immiscible with water, which has a higher boiling point than water and which is volatile in steam, and isolating solid pentaerythritol tetranitrate from its solution in the said solvent after free acid and any insoluble impurities have been removed therefrom.

6. The process of purifying pentaerythritol tetranitrate obtained by nitrating pentaerythritol, which comprises treating it with a solvent which is substantially immiscible with water, which has a higher boiling point than water and which is volatile in steam, removing any insoluble impurities from the solution of pentaerythritol tetranitrate, and subjecting the said solution to steam distillation in the presence of a dilute aqueous alkaline solution.

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