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MANUFACTURE OF DETONATING EXPLOSIVES AND PRODUCTS OBTAINED THEREBY

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The present invention relates to the provision of new or improved high velocity detonating explosive compositions useful for demolition and like uses, and characterised by high power and satisfactory sensitiveness to initiation by detonation, but by low sensitiveness to initiation by friction or impact.

The explosive compositions prepared according to our invention when freshly made are plastic, and can, if desired, be shaped by extrusion. Some of them retain their plasticity on storage and can be used for purposes for which plastic properties are a requisite, while others assume on storage a solid or even friable consistency and can be employed for purposes with which such a consistency is compatible. The explosive compositions made according to our invention consist essentially of crystalline explosive polynitric esters solid at normal temperatures desensitised by means of a liquid aromatic nitrobody composition rendered viscous by the presence of a proportion of nitrocellulose dissolved therein, whereby exudation of the nitrobody is prevented, the nitric ester being present as the predominating ingredient of the composition, optionally with minor proportions of other ingredients. In order to produce such a mixture having a satisfactory low sensitiveness to friction and shock, it is necessary that the solid crystalline nitric ester should be present in a finely crystalline condition intimately distributed throughout the nitrobody gel, and this requirement renders the ordinary incorporation methods for the preparation of explosives inapplicable or dangerous for the manufacture of the desired compositions, since the nitric esters are sensitive to friction.

According to the present invention, a process for the manufacture of an explosive of the character hereinbefore described comprises mixing with the liquid aromatic nitrobody a mixture of the crystalline polynitric ester and a nitrocellulose co-precipitated from solution in a mutual organic solvent by means of a precipitating liquid miscible with said solvent, the nitrobody and the nitrocellulose being such that the nitrocellulose is capable of being gelatinised by the nitrobody.

The invention is applicable to crystalline polynitric esters which do not become liquid at ordinary or at storage temperatures up to about 50° C., for example pentaerythritol tetranitrate, erythritol tetranitrate, hexanitromannitol, enneahexitol hexanitrate, dipentaerythritol hexanitrate, anhydo-ennea-heptitol penta-nitrate, dulcitol hexanitrate and nitro sucrose.

As nitrocelluloses there may conveniently be

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employed those having a nitrogen content of 11.8 per cent. and upwards.

As the nitrobody there may be employed liquid mono-, di-, and polynitro-aromatic compounds; nitro aromatic compounds that are solid at atmospheric temperatures may be used in admixture with other nitro aromatic compounds provided that the mixture is liquid at ordinary temperatures or increased temperatures up to about 50° C. Mono-, di-, and polynitrated aromatic hydrocarbons or mixtures thereof are especially suitable.

In putting the invention into effect the co-precipitated mixture of the nitrocellulose and the solid nitric ester may most conveniently be obtained by introducing the precipitating liquid progressively into a bulk of the solution containing the dissolved solid polynitric ester and nitrocellulose. The precipitation may advantageously be carried out at atmospheric temperature, and the mutual organic solvent and the precipitating liquid are advantageously volatile so that the co-precipitated mixture may be dried off directly. Alternatively, the liquid associated with the co-precipitated mixture may be displaced by treatment with a volatile nonsolvent liquid facilitating the drying of the co-precipitated mixture. Complete removal of the adherent liquid is not always necessary, and when this liquid has the property of assisting the gelatinisation of the nitrocellulose by the nitrobody, it may be advantageous to effect only partial evaporation thereof.

For dissolving and co-precipitating the solid nitric ester and nitrocellulose, it is usually convenient to employ acetone as the solvent. As the precipitating liquid water is especially suitable as it reduces the risk of fire. On the other hand, if the water is displaced by alcohol the co-precipitated mixture can be dried at lower temperatures. Complete removal of the alcohol is not necessary as it assists the gelatinisation of the nitrocellulose. Other indifferent solvents such as methyl ethyl ketone, ethyl acetate, ethyl lactate, nitrobenzene; and other precipitants, for example alcohol, petroleum ether or methyl alcohol may also be employed depending on the solubility characteristics of the particular polynitric ester and the nitrocellulose used.

For the preparation of compositions intended to retain their plasticity it is preferred to employ nitrocelluloses of low viscosities such as are used in the manufacture of industrial lacquers. It will be understood that even in the case of such nitrocelluloses, of which a higher proportion can, if desired, be worked into the composition than of

high viscosity nitrocelluloses, the proportion of nitrocellulose to the composition as a whole will ordinarily be quite small, and usually it will not exceed about 20 per cent. of the weight of the aromatic nitrobody. In order to achieve the solution or gelatinisation of the nitrocellulose, and obtain an explosive of the desired properties, the treatment of the co-precipitated mixture of crystalline polynitric ester with the liquid nitrobody necessitates only a gentle mechanical manipulation. The invention is further illustrated by the following examples, in which the parts are parts by weight.

Example 1

1.4 parts of nitrocellulose having a nitrogen content of 12.0 per cent. and a viscosity of 30 cgs. units in 40 per cent. solution in 95 per cent. acetone at 20° C. are dissolved in 80 parts acetone; with the resulting solution is mixed a solution containing 90.8 parts pentaerythritol tetranitrate in 545 parts acetone and the solution is filtered into a precipitating vessel fitted with mechanical stirring. 409 parts of water are run into the vessel with stirring at a uniform rate over a period of 35 minutes, and a further 409 parts water are then run into the vessel at an increased rate over 10 minutes. The resulting suspension is then decanted, filtered and washed with water, and transferred to a drying stove in which it is dried at a temperature of 45° C. for 72 hours. The dry co-precipitate is then mixed in an Atlas mixer with 26.3 parts of a mixture having a composition—dinitrotoluenes 75 per cent. and trinitrotoluene 25 per cent. Cartridges of the resulting plastic explosive extruded in 3 inches by 1¼ inches diameter from a worm type extrusion machine have a velocity of detonation approaching 8,000 metres per second and a power approximately 89 per cent. of that of blasting gelatine and are sensitive to initiation by a No. 6 lead azide detonator. They maintain their sensitiveness and plasticity and suffer little diminution in velocity of detonation and power even after three months' storage at 35° C. They are not initiated by the impact of a .303 rifle bullet Mark VII fired at a range of 30 yards and are not ignited in a rawhide mallet and birch slab test, and only about once in ten trials in a rawhide mallet and whinstone slab test.

Example 2

The process of manufacture is carried out as in Example 1, except that 5 parts of nitrocotton are used instead of 1.4 parts. The properties of the explosive closely resemble those of the explosive described in Example 1.

Example 3

The process is carried out as in Example 2, except that the proportions of the ingredients used are:

Pentaerythrite tetranitrate-----	74½	65
Nitrocellulose -----	5	
Liquid nitrobody mixture-----	20	
Chalk -----	½	

per 100 parts of the finished explosive, and that the nitrocellulose used is blasting soluble nitrocellulose. The extruded explosive hardens to a friable solid consistency on storage, but remains very insensitive to percussion and frictional blows and also to impact from a rifle bullet.

As many seemingly widely different embodi-

ments of the invention will be apparent without departing from the spirit and scope thereof it must be understood that the invention is not limited to any specific embodiment except as defined in the appended claims.

We claim:

1. A process for the manufacture of an explosive which comprises forming in solution a mixture of dissolved solid crystalline polynitric ester and a nitro cellulose, co-precipitating the mixture by the progressive addition of liquid miscible with the solvent, separating the precipitate containing the polynitric ester in fine crystalline condition, and mixing the same with a liquid aromatic nitrobody capable of gelatinising the nitro cellulose with the polynitric ester crystals intimately distributed therethrough said polynitric ester having a melting point of at least approximately 50° C.

2. A process for the manufacture of an explosive which comprises forming in a mutual solvent a mixture of dissolved pentaerythritol tetra nitrate and nitro cellulose, co-precipitating the mixture by the progressive addition of liquid miscible with said solvent, separating, washing, and drying the precipitate, and subsequently mixing the same with a liquid aromatic nitrobody capable of gelatinising the nitro cellulose with intimate distribution of the crystals therethrough.

3. A process for the manufacture of an explosive which comprises forming in a mutual solvent a mixture of dissolved pentaerythritol tetra nitrate and nitro cellulose, co-precipitating the mixture by the progressive addition of liquid miscible with said solvent, separating and drying the precipitate, and subsequently mixing the same with a liquid aromatic nitrobody capable of gelatinising the nitro cellulose with intimate distribution of the crystals therethrough.

4. A process as claimed in claim 1 in which the aromatic nitrobody has a melting point below 50° C.

5. A process as claimed in claim 1 in which the crystalline explosive polynitric ester is pentaerythritol tetranitrate.

6. A process as claimed in claim 1 in which the crystalline explosive polynitric ester is erythritol tetranitrate.

7. A process as claimed in claim 1 in which the crystalline explosive polynitric ester is hexanitro-mannitol.

8. A process as claimed in claim 1 in which the nitrobody is a mixture comprising dinitrotoluene and trinitrotoluene.

9. A process as claimed in claim 1 in which the nitrobody is a mixture containing about 75 per cent. of dinitrotoluenes and about 25 per cent. of trinitrotoluene.

10. A process as claimed in claim 1 in which the precipitating liquid and the mutual organic solvent are both volatile.

11. A process for the manufacture of an explosive which comprises forming in solution in acetone a mixture of an explosive crystalline polynitric ester and a nitrocellulose, capable of being gelatinised by the nitrobody used later in the process, co-precipitating the mixture by the progressive addition of water to the solution, separating and drying off the acetone and water from the precipitate and then mixing the precipitate with a liquid aromatic nitrobody said polynitric ester having a melting point of at least approximately 50° C.

12. A process as claimed in claim 11 in which

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the rate of addition of water is accelerated towards the end of the co-precipitation step.

13. A process for the manufacture of an explosive which comprises forming in solution in a mutual organic solvent a mixture of an explosive crystalline polynitric ester and a nitrocellulose, capable of being gelatinised by the nitrobody used later in the process, co-precipitating the mixture by the progressive addition of a precipitating liquid to the solution, displacing at least a major portion of the precipitating liquid and organic solvent with a more volatile liquid, separating and drying off the volatile liquid from the precipitate and then mixing the precipitate with a liquid aromatic nitrobody said polynitric ester having a melting point of at least approximately 50° C.

14. An explosive composition comprising an explosive crystalline polynitric ester in the form of fine crystals distributed throughout a gel made up of liquid aromatic nitrobody and nitrocel-

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lulose said polynitric ester having a melting point of at least approximately 50° C.

15. An explosive composition as claimed in claim 14 in which the aromatic nitrobody is one which can be liquefied by heating to a temperature below 50° C.

16. An explosive composition as claimed in claim 14 in which the explosive polynitric ester is pentaerythritol tetranitrate.

17. An explosive composition as claimed in claim 14 in which the explosive polynitric ester is erythritol tetranitrate.

18. An explosive composition as claimed in claim 14 in which the explosive polynitric ester is hexanitromannitol.

19. An explosive composition as claimed in claim 14 in which the nitrobody is a mixture containing dinitrotoluene and trinitrotoluene.

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