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NITROCELLULOSE-CONTAINING COMPOSITION AND PROCESS OF PREPARING SAME

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The present invention relates to a new and improved nitrocellulose preparation. These nitrocellulose preparations can be used for the manufacture of blasting explosives of the kind that include a gelatinous solution of nitrocellulose in a liquid explosive nitric ester and a proportion of ammonium nitrate, along with other ingredients.

When dry nitrocellulose is used as one of the raw materials for the manufacture of blasting explosives, the hazards and expense involved in the drying operation and the handling and storage of the dry nitrocellulose must be undertaken. While nitrocellulose in a water wet form containing a proportion of the water resulting from the nature of the manufacture of nitrocellulose is often used in limited amounts in the manufacture of blasting explosives for the purpose of thickening the liquid nitric ester, the presence of the water, which amounts to about 25 to 33 per cent of the weight of the water wet nitrocellulose, has a desensitising effect on the resulting explosive, and may render it necessary to work the mixture for a longer time than usual in order to bring about the solution of the nitrocellulose in the liquid explosive nitric ester. If ammonium nitrate is present as an ingredient of the explosive, the presence of the water may also accelerate the tendency of the composition to set. It is also known to employ spirit wet nitrocellulose, which is obtained by displacing the water from water wet nitrocellulose with somewhat diluted alcohol, but the use of spirit wet nitrocellulose causes the liquid nitric ester to dissolve the nitrocellulose with the formation of a solution characterised at least when freshly made by excessive capacity for extension and adhesiveness, properties which introduce difficulties in the conduct of the mixing operation and in the subsequent extrusion operation.

According to the present invention the improved nitrocellulose preparation consists of nitrocellulose of the kind soluble in liquid explosive nitric esters, in uncolloided condition and moist with a solution comprising at least 30% by weight of a mixture of ammonium nitrate and urea in binary eutectic proportion in an aqueous alcoholic solvent substantially richer in alcohol than in water, the amount of water being not more than 20% on weight of the nitrocellulose.

It is to be understood that the phrase "a mixture of ammonium nitrate and urea in binary eutectic proportion" includes mixtures in which the proportion of ammonium nitrate and urea is only approximately a binary eutectic one.

The weight of the said solution may be for

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instance from about 0.9 to 1.3 times the weight of the uncolloided nitrocellulose, and of this solution the mixture of ammonium nitrate and urea of substantially the binary eutectic composition preferably amounts to approximately 35 to 50%.

The binary eutectic composition is ammonium nitrate 53% urea 47%; and this composition melts at 44.7° C. and requires the addition of about 10% water to bring it into the form of a viscid solution at room temperature. Nitrocellulose moistened with the resulting solution dissolves too slowly in explosive liquid nitric esters to be useful for the manufacture of explosive compositions. The solution can however, be further diluted with industrial spirit without precipitating either of the solids; and uncolloided nitrocellulose moist with a damping solution as herein before specified dissolves rapidly in liquid explosive nitric esters without forming tacky solutions such as would lead to mixing and extrusion difficulties. The desensitising and settling difficulties experienced when water is used as damping medium for nitrocellulose are also minimised.

It is possible to make the improved nitrocellulose preparation directly from water wet nitrocellulose by treating the latter with strongly alcoholic aqueous solution of ammonium nitrate and urea under such conditions that the displacement of the water is nearly complete. This usually necessitates the introduction as the displacing liquor of an alcoholic solution of ammonium nitrate and urea of such low water content as only to be free from crystallisation at temperatures substantially above atmospheric. It is however, more convenient to make the product from the water wet nitrocellulose indirectly, that is to say from spirit wet nitrocellulose, since it is then unnecessary to achieve a high degree of displacement of the spirit to attain in the product the required low water content. It is even possible to attain the required composition by treatment of the spirit wet nitrocellulose with a strong aqueous solution of the ammonium nitrate and urea, which need in this case contain no alcohol, without rejection of any of the liquors at all, that is to say by a process wherein the whole of the spirit of the spirit wet nitrocellulose is retained and mixes with the incoming liquor. Whether the final composition is attained by dilution alone or by a combination of dilution and displacement of the spirit in the spirit wet nitrocellulose it is easy to arrange that the liquor that is added will contain sufficient water to maintain it free from crystallisation at ordinary temperature while also containing sufficient ammonium nitrate and urea

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to allow its dilution by the retained spirit of the spirit wet nitrocellulose.

The manufacture may be carried out, for instance, by spraying spirit wet nitrocellulose with the substantially eutectic mixtures dissolved in a suitably limited amount of water, or by washing the spirit wet nitrocellulose in a centrifuge with a solution of the substantially eutectic mixture in an alcohol-water mixture of say 60-70% alcoholic strength and centrifuging away a predetermined quantity of the liquor.

The following examples illustrate a method of producing improved nitrocellulose preparations in accordance with the invention and their application to the production of explosives. The percentages and parts are by weight.

Example 1

A nitrocellulose preparation containing:

	Per cent
Uncolloided nitrocellulose of 12.1% nitrogen content	48
Alcohol	25
Water	6.5
Ammonium nitrate	10.7
Urea	9.8

is produced in the following manner.

An aqueous solution containing:

	Parts
Ammonium nitrate	12
Urea	10.5
Water	2.5

is first formed by adding the ammonium nitrate and urea to water in said proportions and effecting dissolution in a hot water bath at about 90° C. 9.5 parts of this aqueous solution is sprayed uniformly on to 20 parts (dry weight) of uncolloided nitrocellulose of 12.1% nitrogen content, previously dehydrated in a centrifugal to an alcohol-water content of 38%, in which alcohol-water mixture the quantity of alcohol is 86%.

The spraying of the aqueous solution can be carried out by passing the said solution through the rose of a watering can. The nitrocellulose, equivalent to 32.5 parts wet weight, is uniformly spread in a pan of area 10 square feet thereby permitting uniform spraying of an appreciable area of the nitrocellulose. The mass is turned over intermittently by hand during the spraying operation to effect approximate uniformity of wetting throughout the mass.

2.5 parts of the nitrocellulose preparation thus formed, 2.7 parts waxed woodmeal, 0.5 part woodmeal, 28 parts of an 80:20 mixture of nitroglycerine and ethylene glycol dinitrate and 1.8 part of a liquid dinitrotoluene-trinitrotoluene mixture are mixed together in a Talley mixer for about 3 minutes by which time the nitrocellulose is dissolved to give a solution of somewhat gelatinous character. 42 parts ammonium nitrate, 19 parts sodium nitrate, 3 parts sulphur, 1 part starch and 0.5 parts chalk are worked into the mixture while running the mixer for about another 5 minutes. The resulting non-tacky gelatine explosive composition is removed from the mixer, and is extruded and cartridgeed in known manner without difficulty. It is satisfactorily sensitive to a No. 6 commercial detonator. In a double cartridge gap test, in which two $\frac{7}{8}$ " diameter cartridges are arranged end to end separated by a gap, the maximum gap over which detonation of the receptor cartridge can be induced was 3 to 4 inches when the cartridges are freshly made

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and 3 to 4 inches after 3 months dry storage at 35° C.

Example 2

5 An improved preparation containing:

	Per cent
Nitrocellulose as described in Example 1	51.5
Alcohol	27.5
Water	6.0
Ammonium nitrate	8.0
Urea	7.0

is prepared as in Example 1, except that 6.5 parts of the aqueous solution of ammonium nitrate and urea is uniformly sprayed on to 20 parts (dry weight) of the uncolloided nitrocelluloses of an alcohol-water content of 38%, in which alcohol-water mixture the quantity of alcohol is 86%.

6 parts of the above nitrocellulose preparation and 61 parts of an 80:20 mixture of nitroglycerine and ethylene glycol dinitrate are mixed together in a Talley mixer for about 5 to 10 minutes, thus forming a fairly stiff jelly. Into this jelly are worked in the course of another 5 minutes 6.5 parts woodmeal, 26.2 parts sodium nitrate and 0.3 part chalk. The resulting non-tacky explosive composition is removed, extruded and cartridgeed without difficulty, in known manner. It is satisfactorily sensitive to a No. 6 commercial detonator. In a double cartridge gap test, in which two $\frac{7}{8}$ " diameter cartridges are arranged end to end separated by a gap, the maximum gap over which detonation of the receptor cartridge can be induced is 11 to 12 inches when the cartridges are freshly made and 8 to 9 inches after 1 month's atmospheric temperature storage.

An explosive not in accordance with the invention similar in composition to that of Example 2, except that 5.5 parts water wet nitrocellulose (containing 3.5 parts dry nitrocellulose) are used instead of 6 parts of the above nitrocellulose preparation and the amount of ammonium nitrate is increased by 0.5 parts, gives a gap test of only 3 to 4 inches when the cartridges are freshly made and 2 to 3 inches after 1 month's atmospheric storage.

I claim:

1. An improved nitrocellulose containing preparation for the manufacture of explosives comprising uncolloided liquid nitric ester-soluble nitrocellulose moistened with a solution comprising at least 30% by weight of a substantially binary eutectic mixture of ammonium nitrate and urea in an aqueous alcoholic solvent, the said solvent being substantially richer in alcohol than in water and the amount of water present in the preparation being less than 20% by weight of the nitrocellulose.

2. An improved nitrocellulose containing preparation as claimed in claim 1 wherein the weight of the said solution is from 0.9 to 1.3 times the weight of the uncolloided nitrocellulose.

3. An improved nitrocellulose containing preparation as claimed in claim 1 wherein the said solution contains up to 50% of the substantially binary eutectic mixture of ammonium nitrate and urea.

4. A process for the preparation of an improved nitrocellulose containing composition for the manufacture of explosives comprising moistening spirit wet uncolloided liquid nitric ester-soluble nitrocellulose with at least 30% by weight of a solution comprising a substantially binary eutectic mixture of ammonium nitrate and urea dissolved in a solution comprising water, the amount of water

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present in the resulting preparation being less than 20% by weight of the nitrocellulose.

5. A process for the preparation of an improved nitrocellulose containing composition for the manufacture of explosives comprising moistening water wet uncolloided liquid nitric ester-soluble nitrocellulose with at least 30% by weight of a solution comprising a substantially binary eutectic mixture of ammonium nitrate and urea dissolved in a solution comprising water and alcohol, the amount of water present in the resulting preparation being less than 20% by weight of the nitrocellulose, and the preparation being substantially richer in alcohol than in water.

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REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
1,510,348	Snelling -----	Sept. 30, 1924
1,819,458	Dehn -----	Aug. 18, 1931
1,966,806	Spurlin -----	Aug. 17, 1934
2,063,572	Woodbury et al. -----	Dec. 8, 1936

FOREIGN PATENTS

Number	Country	Date
552,645	Great Britain -----	Apr. 19, 1943
43,911	Switzerland -----	June 29, 1908