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PROPELLENT POWDER

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This invention relates generally to explosives, and particularly to a process of handling nitroglycerine and treating propellant powders with the same.

In propellant powders of the smokeless type, which may be, for instance, of a nitrocellulose base and which are treated with a deterrent in order to achieve a progressive rate of combustion and thereby attain improved ballistic characteristics, it sometimes happens that the presence of the deterrent operates to retard the ignition of the powder and consequently it is necessary to employ primers which will produce a hotter flame than would otherwise be necessary. The use of a primer of too high an energy content sometimes imposes an undesirable condition, in that excessive variations in pressure and velocity are occasionally encountered. The higher energy of the primer, although designed to off-set the deficiencies in the ignitibility of the powder, sometimes promotes conditions in the powder charge which are conducive to more rapid combustion of the powder, and, hence, to the development of excessively high pressures. Experience has indicated that it is preferable to avoid excessive use of difficultly ignitable coatings on the powder. In order to render such powders more readily ignitable and at the same time more non-hygroscopic such powders may also be surface treated with nitroglycerine which operates as an accelerator in that it renders the powder more readily ignitable and consequently primers without such exceptionally hot flame characteristics may be successfully employed with the propellant charge.

It is among the objects of the present invention to provide a process of handling nitroglycerine and treating powder with the same wherein the hazards heretofore encountered in such processes will be substantially eliminated.

The object of the present invention, generally stated, is to provide a safe process of treating smokeless powder with an agent, such as nitroglycerine, which renders the powder more readily ignitable and/or more non-hygroscopic.

A further object is to provide a process of treating smokeless powders concurrently with an accelerator, such as nitroglycerine, and a deterrent.

Another object of this invention is to provide a smokeless propellant powder grain, the surface of which is gelatinized by both an accelerator and a deterrent.

A still further object is to provide nitroglycerine in a form in which it may be safely and economically handled.

A more specific object of this invention is to provide a process of surface treating propellant powder grains wherein the procedure is such as to avoid the necessity of temperatures such as would tend to cause explosions.

Other objects will become apparent to those skilled in the art when the following description is read.

Generally stated, in accordance with the present invention, a propellant powder which may be, for instance, of a type having a nitrocellulose base, may be treated with an accelerator, or easily ignitable material, such, for instance, as nitroglycerine with, or without, a deterrent. This may be accomplished by treating the nitrocellulose in a non-solvent bath, such as water, in which the nitroglycerine with, or without, deterrent is distributed. The nitroglycerine content of the bath may be regulated so as to control the amount which will be taken up by the nitrocellulose, it being understood that due to the great affinity of nitroglycerine for nitrocellulose, substantially all nitroglycerine in the bath will be taken up by the nitrocellulose. In order to achieve uniformity of treatment of the nitrocellulose the nitroglycerine may be added to the bath, in the form of a solution not miscible or only partially miscible with the bath, or as an emulsion, and the entire mixture agitated sufficiently to cause the nitroglycerine and nitrocellulose to be distributed therein. Because of the affinity between the nitrocellulose and nitroglycerine there is a tendency for them to gelatinize, the nitroglycerine being thus separated from its solvent. After gelatinization has progressed to a desired extent, the bath in which the treatment has taken place, and still containing some of the solvent for the nitroglycerine, may be discharged from the treated grains, for instance, as by draining or wringing in a centrifuge. In this manner coating of the grains with a highly explosive accelerator is accomplished without subjecting the treating bath to temperatures of such order as would tend to cause explosions of the nitroglycerine. If desired, any suitable deterrent or other material designed to modify the surface or internal characteristics of the grains may be added to the treating bath along with the nitroglycerine and is conveniently dissolved in the same solvent which carries the nitroglycerine into the bath. These additional materials may serve several purposes. They may, for example improve the resistance to penetration of moisture. They may improve the flowing characteristics of the grains and promote more uniform loading condi-

tions. They may avoid development of static electricity. They may aid the nitroglycerine to penetrate the grain.

The nitrocellulose to be treated may be in the form of substantially completely gelatinized grains as obtained by the extrusion or rolling processes followed by suitable granulating by cutting into discs, flakes, cylinders, etc.; or, the grains may be only partially gelatinized as in the case of bulk powder grain as used in shot-shell loads; or, ungelatinized grains of nitrocellulose, as obtained by cutting sheets of cellulose into grains or flakes either prior to or after nitration, may be used. Other forms and composition of propellents of various granulation, degrees of gelatinization, and density may be employed. The nitrocellulose, for example, may be of any desired nitrogen content or a blend of nitrocellulose of various nitrogen contents. Instead of nitroglycerine, the nitrates of ethylene, diethylene, or other glycols may be used, or mixtures of them, with nitroglycerine. Other mixtures containing penta-erythrite-tetranitrate, tetranitro-diglycerine may be used in various combinations with, or without, nitroglycerine.

As remarked above, the nitroglycerine may be introduced either as a solution or as an emulsion. The solvent employed may be one which is, or is not, a solvent for the nitrocellulose. If it is desired to harden the surface of the grains more than would be accomplished by the nitroglycerine with, or without, deterrent, then a solvent which is also a solvent for the nitrocellulose may be used. Ordinarily, however, it is desired to avoid softening the grains as much as possible and this dictates the use of a solvent for the nitroglycerine which is non-solvent for the nitrocellulose. Ethyl alcohol, benzol, toluene or mixtures thereof, may be used in the latter event while ethyl, butyl, or propyl acetate may be used where additional hardening is desired.

Toluene possesses a very distinct advantage in that more concentrated, yet safe, solutions of nitroglycerine may be formed in it than in any other known solvent for nitroglycerine. A solution composed of 50 parts nitroglycerine and 50 parts toluene is safe to handle at low temperatures, and in addition such a solution will stand ignition and combustion with less chance of detonation. The toluene seems to burn off more slowly and allow concurrent slow combustion of the nitroglycerine. By adding to the solution 5-10% by weight of a deterrent substance, such as dinitrotoluene, a dialkylphthalate, or other well known equivalent, the tendency of the nitroglycerine to detonate, even after the solvent has burnt off is very considerably reduced. The presence of such a deterrent substance causes combustion to take place more uniformly, and, hence, reduces the tendency of the nitroglycerine to detonate.

The solvent used in the treatment of nitrocellulose in accordance with this invention may be selected according to the properties desired in the powder and economical and safety considerations. For instance, greater penetration of the nitroglycerine into the grains may be obtained by using toluene than is obtained from an alcohol or benzol. Moreover, when alcohol or alcohol-benzol is used, some of the nitroglycerine may be precipitated due to the solution of alcohol in the water of the bath. This precipitated nitroglycerine will usually be in a very finely dispersed form and will act upon the nitrocellulose surface uniformly and in a manner

appreciably different from that encountered in the attack of the nitrocellulose by a benzene or toluene solution of nitroglycerine. It, therefore, depends to a large degree upon the type of propellant powder grains desired, as to what specific surface characteristics are deliberately produced, and as to what solvents are employed. If alcohol is used, it will almost inevitably be lost, due to dilution by the water used in separating the powder grains. The solvents such as toluene and benzene, which are largely non-miscible with water, can be separated to some extent and recovered, but it may frequently be of an advantage to use such proportions of solvent with respect to powder, nitroglycerine, and water that substantially all of the toluene, for example, will penetrate the grains and will be removable from the grains during subsequent drying treatments.

The solution of nitroglycerine may be introduced directly into the treating bath or may be first emulsified with a small amount of water and then introduced. Suitable emulsifying agents such as gum arabic, starch, dextrine and soaps may be used in the latter event.

As a practical embodiment which is illustrative of the present invention a charge of nitrocellulose powder grains, for example, 300 pounds, may be incorporated in a suitable non-solvent, such for instance as water. The ratio of water to powder grains may be from 3-1 to 10-1 by weight, a practical proportion being 4 parts of water to 1 part of powder grains. This may be agitated to produce a good distribution of the powder grains in the water and then the nitroglycerine may be added in the form of a solution. Water to powder ratios of even less than 3-1 may, in some instances, be used in suitable equipment.

As a specific example of a suitable solution, thirty pounds of nitroglycerine may be dissolved in a vehicle consisting of seventy-five pounds ethyl alcohol and twenty-five pounds benzol or toluene. The nitroglycerine solution thus formed is suitable for treating about 300 pounds of nitrocellulose powder grains, of such a size and character that the resultant powder will give suitable ballistics in a 12 gage shot shell.

If desired, a suitable deterrent, such for instance as dibutylphthalate or dinitrotoluene, may also be dissolved in the nitroglycerine solution. It will be understood that the quantity of deterrent employed will depend upon the circumstances and the type of ammunition into which the resultant powder is to be loaded.

After the nitroglycerine solution, which may, or may not, contain a deterrent, has been added to the water-nitrocellulose mixture the same may be agitated in a suitable vessel at room temperature for a period of about thirty minutes. The temperature of the bath may then be gradually raised to a suitable temperature at which gelatinization of the nitrocellulose powder by nitroglycerine and deterrent, if the latter is employed, may progress. A suitable and safe temperature is 65° C. which may be reached gradually, as for instance, over a period of forty-five minutes. The mixture may be held at this temperature for a period of time sufficient to accomplish the desired surface gelatinization. The nitroglycerine (and deterrent) tends to attack and gelatinize the nitrocellulose leaving the solvent entrained in the bath. However, part of the nitroglycerine probably remains with the solvent, such as benzene or toluene, and pene-

trates the nitrocellulose grain in the form of a solution of nitroglycerine. After surface gelatinization and impregnation by the nitroglycerine and deterrent has progressed to the desired extent which may require, for instance, a period of one hour, the mixture may be cooled down to a suitable temperature for handling as, for instance, 40° C.

The liquid content may then be discharged from the treated grains in any suitable manner as by draining and/or wringing in a centrifuge. The wringing operation may be continued until the moisture content of the treated powder grain has been reduced to less than 30%. In this manner the spent solvent having given up substantially all its nitroglycerine and deterrent, if the latter is employed, is discharged with the water and may be disposed of as is desired.

As another illustrative embodiment, nitroglycerine, containing from 5-10% of its weight of dinitrotoluene, may be dissolved in toluene to give a solution as concentrated as 30 pounds of nitroglycerine in 30 pounds of toluene. This solution may be emulsified with water using a suitable emulsification agent, such as gum arabic, to give a liquid comprising very finely dispersed droplets of nitroglycerine-dinitrotoluene-toluene solution in water. This emulsion is easily distributed in a very uniform manner throughout the water nitrocellulose mixture and is sufficient to treat about 300 pounds of dense powder for sporting ammunition. The remaining operations are substantially as described above.

As illustrating the use of a nitroglycerine solvent which is also solvent for nitrocellulose so as to obtain additional hardening of the grains, 30 pounds of nitroglycerine may be dissolved in 300 pounds ethyl acetate. This may be distributed in a bath containing about 300 pounds of powder. Advantageous results are obtained with this, as well as the other embodiments, if about one-fourth percent of an emulsifying agent, such as gum arabic, starch, dextrine, soap, and the like, is introduced into the treating bath with the nitroglycerine. Such materials act as protective colloids and prevent the grains under treatment from sticking together. Moreover, by the aid of emulsifying agents the nitroglycerine may be emulsified with water and a sufficiently fine dispersion obtained without any solvent.

From the foregoing description it is apparent that the invention accomplishes its objects and that the process is such as to avoid temperatures such as might tend to cause explosions of the nitroglycerine. Although it is to be understood that the temperatures and periods of treatment above referred to are merely for the purpose of illustration, they may be varied within limits, it being preferable, however, that substantially none of the solvent employed for the nitroglycerine, with, or without, the deterrent, be evaporated. By thus maintaining the temperatures of treatment below the vaporizing temperature for the nitroglycerine solvent, it is apparent that the hazardous possibility of nitroglycerine distilling over with the solvent is avoided and the danger of operation thus greatly reduced. In accordance with the present invention substantially all of the solvent employed for the nitroglycerine, after giving up its nitroglycerine and deterrent (if the latter is employed) is discharged as a liquid with the water of the treating bath. Moreover, the powder grains resulting from the proc-

ess of the present invention, when a deterrent is employed, will have their surfaces gelatinized by both nitroglycerine and the deterrent in view of the fact that the same have been deposited concurrently. In this manner the ballistic characteristics may be more readily controlled while favorable ignition properties are maintained and a progressive burning or combustion rate achieved.

It is apparent that the various features of this invention are not limited in their application to the specific embodiments hereinbefore described, but it is to be understood that the terms nitrocellulose, nitroglycerine deterrent solvent, etc., have been used in an illustrative and not in a limiting sense. It is to be understood, therefore, that the invention is not limited to the specific details herein set forth for the purpose of illustration, but that such modifications and the use of such individual features and sub-combinations of features as may occur to those skilled in the art without departing from the spirit of this invention are, although not specifically described herein, contemplated by and within the scope of the appended claims.

Having thus described the invention, what is claimed is:

1. In the art of making propellant powders, the process comprising, treating nitrocellulose powder grains while suspended in water with a solution of nitroglycerine and a deterrent.

2. In the art of making propellant powders, the process comprising, treating nitrocellulose in a non-solvent bath having a solution of nitroglycerine distributed therein.

3. In the art of making propellant powders, the process comprising, forming an aqueous suspension of nitrocellulose, and distributing a solution of nitroglycerine throughout the slurry.

4. In the art of making propellant powders, the process comprising, treating nitrocellulose powder grains while distributed in a bath of water with nitroglycerine dissolved in a vehicle until the grains are surface gelatinized, elevating the temperature of the mix to an extent sufficient to promote gelatinization of the nitrocellulose by the nitroglycerine but not high enough to vaporize either solvent, the nitroglycerine or the water and removing the vehicle as a liquid.

5. In the art of making propellant powders, the process comprising, treating nitrocellulose powder grains while suspended in water with nitroglycerine and a deterrent dissolved in a vehicle which is non-solvent for the nitrocellulose.

6. In the art of making propellant powders, the process comprising, treating smokeless powder grains with nitroglycerine dissolved in toluene.

7. In the art of making propellant powders, the process comprising, treating smokeless powder grains with nitroglycerine and dinitrotoluene dissolved in toluene.

8. In the art of making propellant powders, the process comprising, surface treating smokeless powder grains with a nitroglycerine solution in a non-solvent bath, and discharging the bath with the entrained spent nitroglycerine solution from the grains after surface treatment has progressed to the desired extent.

9. In the art of making propellant powders, the process comprising, suspending smokeless powder grains in a non-solvent vehicle, adding a solution of modifier having an affinity for the grains, agitating the mixture until the modified

gelatinizes the grains leaving the solvent entrained in the vehicle, and discharging the vehicle with the entrained modifier solvent.

10. In the art of making propellant powders, the process comprising, concurrently surface treating smokeless powder grains while suspended in a non-solvent bath with an accelerator and a deterrent in a solvent capable of penetrating the grain.

11. In the art of making propellant powders, the process comprising, concurrently surface treating smokeless powder grains while suspended in a non-solvent bath with nitroglycerine and a detergent in a solvent capable of penetrating the grain.

12. In the art of making propellant powders, the process comprising, concurrently surface treating smokeless powder grains while suspended in a non-solvent bath with nitroglycerine and

dibutylphthalate in a solvent capable of penetrating the grain.

13. In the art of making propellant powders, the process comprising, concurrently surface treating smokeless powder grains while suspended in a non-solvent bath with nitroglycerine and dinitrotoluene in a solvent capable of penetrating the grain.

14. The process of producing a propellant powder which comprises, preparing a water bath containing smokeless powder grains and a liquid nitric ester of a polyhydric alcohol dissolved in a solvent therefor, and agitating the bath and contents sufficiently to distribute the powder grains and ester solution therein, such agitation being maintained until the ester is substantially all absorbed by the powder grains.

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CERTIFICATE OF CORRECTION.

Patent No. 2,344,516.

March 21, 1944.

GEORGE A. McBRIDE.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 3, second column, line 75, claim 9, for "modified" read --modifier--; page 4, first column, line 14, claim 11, for "detergent" read --deterrent--; and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 16th day of May, A. D. 1944.

Leslie Frazer

(Seal)

Acting Commissioner of Patents.

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