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MANUFACTURE OF SMOKELESS POWDER

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This invention relates generally to the manufacture of explosives and particularly to the manufacture of smokeless powder. As to common subject matter this application is a continuation of the prior copending applicaion of Fredrich Olsen et al., Serial No. 620,302, filed June 30, 1932, as well as application of Fredrich Olsen, Serial No. 753,503, filed November 17, 1934.

It is among the objects of this invention to provide a process whereby deteriorated and partially unstable powder may be so treated and renovated as to render it adaptable for use in ammunition. A more specific object of the present invention is to provide a process of making smokeless powders suitable for use in small arms ammunition wherein deteriorated cannon powder having unstable regions may be employed as the raw material.

Another object is to provide a process whereby the production of grains of the desired shape and size may be controlled.

Another object is to provide a process of manufacturing smokeless powders wherein the procedure is safe and economical.

Another object is to provide a process for making smokeless powder wherein the resulting grains will be uniform as to their ignition characteristics or rate of combustion.

According to another phase of the invention, the object, generally stated, is to provide a process of making propellant powder in which fine particles of smokeless powder are agglomerated together through the agency of an explosive which is a plasticizer for nitrocellulose.

Another object of this invention is to provide a process of agglomerating particles of nitrocellulose into grains through the agency of nitroglycerine with or without other explosive plasticizers.

A further object of this invention is to provide a smokeless powder grain composed of particles of dense colloided nitrocellulose powder together with nitroglycerine and containing another explosive plasticizer of the nitrotoluene group.

Other objects will become apparent to those skilled in the art when the following description of the process of this invention is read and understood. It is to be understood, however, that the embodiments hereinafter described are illustrative only of various embodiments of which this invention is susceptible. In the following description, as far as possible, an explanation will be given of what is believed to be the action taking place from a theoretical standpoint; it is to be understood, however, that in giving such explanation the theory and explanation is advanced for the purpose of facilitating an understanding from a theoretical standpoint; and it is understood that this invention is not to be limited to any advanced theory or explanation from a theoretical standpoint.

Generally stated, in accordance with the present invention, finely divided particles of dense colloided smokeless powder, such for example as scrap cannon powder, may be agglomerated together and formed into grains of the desired size and ignition characteristics in order to adapt them for use as the propellent in a variety of types of ammunition. Such agglomeration of fine particles of dense smokeless powder, such as completely gelatinized nitrocellulose, may be accomplished by treating the particles with a suitable agglomerating agent, which may advantageously be a material which is a solvent or plasticizing agent for the dense smokeless powder particles. When such a solvent is employed, the finely divided particles may be surface plasticized and cohered together through the medium of their mutual colloid, in order to accomplish the agglomeration. This operation may conveniently be carried out in a bath of water, and it is especially advantageous to employ an agglomerating solvent which is substantially insoluble in the water. When such a mixture is vigorously agitated, a multitude of the particles of dense smokeless powder are gathered together by the respective globules of solvent, and upon removal of the solvent, as by vaporization, the various particles of dense smokeless powder become autogenously welded into grain form.

During the removal of the solvent, such for instance as by evaporation, the tendency is for the several particles of nitrocellulose dust to coalesce or form into small clusters. The extent of this coalescence may be controlled by controlling the violence of the agitation during the removal of the solvent, and the gravimetric density of the resultant grains may be controlled by regulation of the rate of temperature increase and the period of time employed to completely remove the solvent by vaporization. Furthermore the amount of solvent employed affects not only the size, shape and surface of the resultant grains, but also the gravimetric density thereof.

The desired amount of solvent may be conveniently added to the slurry of nitrocellulose dust in the form of a spray and in this manner small globules of the solvent may be readily distributed throughout the mixture. During agitation and heating the surfaces of the particles of nitro-

cellulose dust become softened due to the gelatinizing action of the solvent and tend to coalesce forming clusters comprising a number of dust particles. This action may be explained since it is well known that all liquid or plastic two phase system tend to a condition of minimum surface energy which can be accomplished by coalescence. In the practice of the present invention suitable substances descriptively termed protective colloids and which may include emulsifying and de-emulsifying agents as well as electrolytes, may, if desired, be incorporated in the mixture in order to effect modification in the granulation which will be accomplished. Among the features which contribute to the resultant physical characteristics of the resultant powder grains are the following:—Small amounts of solvent give a small rough cluster of particles as the grains while larger amounts of solvent give more spherical smoother grains. The faster and more violent the agitation, the smaller the resultant grains. While, as pointed out hereinbefore, the rate at which the temperature is raised during evaporation of the solvent affects both the granulation and density of the resultant powder. In this respect the rapidity with which certain temperatures, such for instance as the boiling point of the mixture of water and solvent, are reached has a particular effect upon the density of the powder.

After the mixture has been heated for a time sufficient to accomplish removal of as much of the solvent as may be economically accomplished at this time, the grains may be removed from the still in which the distillation has been carried out and the water removed therefrom as by centrifuging or wringing. Thereafter the grains may be given a coating for modifying the burning characteristics of the powder and rendering the same non-hygroscopic if desirable. This coating may, for instance, comprise nitroglycerine which acts as an accelerator in that it renders the powder more readily ignitable and also acts as a water proofing agent, rendering the powder non-hygroscopic. A suitable deterrent such, for instance, as dibutylphthalate may be used along with the nitroglycerine or other accelerator and water proofing agent. After this is accomplished, the grains may be dried in the usual manner and if desired, glazed and blended in accordance with the usual practice.

As an illustrative embodiment of the present invention completely gelatinized nitrocellulose powder, such, for instance, as cannon powder which may be to some extent deteriorated and contain unstable regions may be finely subdivided in a suitable machine, such for instance as a beater, and preferably to a fineness such as will permit it to pass a sixty-five mesh screen. In accordance with the present invention the raw material employed may be, for instance, nitrocellulose powder dust which has passed through a sixty-five mesh screen and has been caught on a hundred mesh screen. Furthermore material which is passed through a hundred mesh screen is suitable for use as the raw ingredient for the process of the present invention. For certain purposes it is desirable that the raw material be of a more uniform fineness than under other conditions and accordingly under these circumstances the raw material may be caused to pass through an eighty mean screen and caught on a hundred mesh screen. It is to be understood, however, that for some purposes the raw material may be of a larger size than sixty-five mesh.

In the case of grains containing only spots of deteriorated material, these acidic portions are likewise broken up thoroughly during the grinding operatings and similarly dispersed or neutralized. Powder on which deterioration has not proceeded to such an extent that visible regions of discolored material are detectable is, nevertheless, improved in stability through the grinding operation and the contact which is afforded between the newly cut and exposed surfaces and the mild alkalinity of the water used in the grinding. The degree of stability of the resultant ground powder depends to some extent upon the degree of fineness to which the material has been ground.

Under ordinary circumstances, however, deteriorated cannon powder which may be used for this purpose will not contain so great a proportion of unstable regions of concentrated acidity that the acidity, when diffused throughout the entire mass, will be excessive. The local concentrations of acid which are responsible for the unstable character of the gelatinized nitrocellulose powder used as a raw material in the process of this invention are disposed of in this manner. Furthermore, it may be said that the nitrated diphenylamines are responsible to some extent for the unstable character of the powders. Diphenylamine is a neutral compound, generally employed as a stabilizer for nitrocellulose powders, but which upon nitration becomes progressively more acidic in character. Enough diphenylamine is usually present in a grain of ordinary cannon powder to produce a relatively stable mass if the nitrated and unnitrated diphenylamine were diffused throughout the mass which is accomplished in accordance with this invention. After grinding and especially if a mildly basic substance, such as calcium carbonate is added to the slurry, the resultant mass of nitrocellulose dust is not only stable but the acidity thereof is not above the allowable.

Upon completion of the grinding operation the small particles of cannon powder, which are descriptively termed dust, may be screened in order to obtain particles of the desired fineness. This may be accomplished in any suitable manner, but under ordinary conditions in accordance with the present invention, the dust which is in the form of a mud may be caused to pass a sixty-five mesh screen and be caught on a hundred mesh screen; under some circumstances, however, it will be understood that particles either above or below these limits may be employed as the raw material for the process of the present invention.

After the water of the grinding slurry has been permitted to drain from the mass of particles a charge of, for instance 325 pounds of the ground powder or dust (on a dry weight basis, it being understood that the ground product even after draining is in the form of a mud) may be placed in a still with from seven and one-half to eight times its weight of water. The mixture may be agitated in the still to form a thin slurry and during this time a suitable solvent, such for instance as ethyl acetate, may be added preferably in the form of a spray. It will be understood that the solvent which may be ethyl acetate may also contain in solution therewith suitable deterrents, stabilizers or other agents for modifying the burning characteristics or hygroscopicity of the powder. As hereinbefore stated, nitroglycerine is such an agent, and hence may be employed. In accordance with one embodiment of

the present invention the amount of solvent employed is from one and one-fourth to two times the dry weight of the raw material comprising the charge. If desired, a suitable quantity of calcium carbonate which is generally employed as a neutralizer may also be added to the still. After the solvent and other agents, if desired, have been sprayed into the still preferably at room temperature, the temperature of the still may be gradually increased until the ethyl acetate or other solvent is completely removed from the slurry in the still.

As pointed out hereinbefore, the characteristics of the resultant powders especially with regard to their granulation and density are to some extent affected by the degree of fineness of the nitrocellulose dust, the rate at which the solvent is added or sprayed into the slurry, and by the schedule of distillation, that is to say, by the manner in which the temperature is varied throughout the period of time over which the distillation extends. The distillation schedule which may be found appropriate and effective to achieve the desired grain characteristics will depend upon various other features such as, for instance, the time and amount of solvent employed. As a general rule it will be found that a shortening of the distillation schedule will have the effect of reducing the gravimetric density. This is especially true of the time period during which the solvent-water mixture is passing through its boiling point which, in the case of ethyl acetate is 69° to 84° C. The same effect may be encountered, however, by shortening the period of time required for raising the temperature of the mixture in the still through any selected temperature bracket, but under most circumstances it is most noticeable during the period at which the solvent-water mixture is passing through its boiling point. As a suitable schedule of distillation where ethyl acetate is employed as the solvent the following is given for the purpose of illustration:

	Minutes
From room temperature to 60° C	45
From 60° to 74° C	45
From 74° to 84° C	60
From 84° to 95° C	45

In the case of ethyl acetate substantially all of the solvent will be found to be removed in accordance with the foregoing schedule of distillation when the temperature reaches 95° C. The solvent may, of course, be recovered in accordance with the usual practice.

Upon completion of the distillation just referred to the contents of the still may be withdrawn and the water removed therefrom by any suitable process, such for instance as by centrifuging whereupon the powder will be found to exist in the form of grains, each comprising a cluster of the dust particles being substantially uniform in size and density, each of which characteristics may be controlled in the manner hereinbefore set forth.

In accordance with the present invention the contents of the still are withdrawn while a slight trace of the solvent still remains therein, and after the excess water has been removed in the manner just referred to the grains containing a small amount of solvent may be coated with suitable modifying and water proofing agent. For instance, the grains may be given a coating of a deterrent such for instance as dibutylphthalate with or without an accelerator to render the grains more readily ignitable. Nitroglycerine is a suitable accelerator since it not only renders the grains more readily ignitable but also renders the same non-hygroscopic. Furthermore nitroglycerine is a good gelatinizer for nitrocellulose, so that it operates to form a relatively impervious surface film on the grain. The nitroglycerine and dibutylphthalate may be applied as by dissolving them in alcohol with or without benzol. When the dibutylphthalate is employed alone the solvents may be dispensed with and the substance emulsified with water. This solution may then be applied to the grains which are preferably carried in a medium such, for instance, as a water slurry. Although the nitroglycerine and dibutylphthalate may be applied to the grains in the same still in which agglomeration of the dust particles has been carried out, it is preferable that these operations be carried out in separate vessels. The proportion of nitroglycerine which is added to the powder may be from 5% to 20% of the dry weight of the powder, a practical example being 10%, it being understood that the nitroglycerine is preferably handled in the form of a 20% to 30% solution of alcohol and benzol. The dibutylphthalate may be added up to 10% of the dry weight of the powder, a practical proportion being 5%. The resultant grains will be provided with a relatively impervious surface film containing the nitroglycerine which increases the ignitability and renders the powder non-hygroscopic and with it the dibutylphthalate which operates as a deterrent.

From the foregoing description it will be understood that action of the ethyl acetate is such as to cause the finely divided particles to agglomerate into clusters, forming small grains, and also to swell the grains depending upon the amount of solvent employed and the rapidity with which it is distributed throughout the water-nitrocellulose slurry. The amount of swelling which is accomplished determines the final gravimetric density of the powder produced. The agglomerating process of the present invention is admirably adapted for use where a slightly deteriorated cannon powder containing unstable regions is employed for the raw material, since it is apparent that diffusion of the nitrated and unnitrated diphenylamines and also of the acidity of the unstable regions continues during the treatment in the presence of the solvent. It is probable that the swollen condition of the nitrocellulose due to the treatment with ethyl acetate permits the ready diffusion of acid within that swollen nitrocellulose into the water with which the nitrocellulose is in intimate contact. It is obvious that, if desired, a suitable amount of neutral diphenylamine may be added to the powder, as for instance with solvent in order to render the powder more stable.

As an illustration of another embodiment of the process of this invention (claimed in the aforesaid application of Olsen, Tibbitts and Kerone, but not claimed herein) there may be incorporated with the nitrocellulose slurry a small quantity of a protective colloid, such, for instance, as starch. The amount of such protective colloid which may be employed may be as low as one-half to one percent relative to the weight of the water in the slurry of nitrocellulose dust and by use of such protective colloid flocculation is restricted to an extent such that the resultant grains are finer and of more uniform size.

In accordance with another phase of the present invention, the fine particles of dense colloided

nitrocellulose powder may be agglomerated together and formed into grains by the use of an explosive plasticizer such, for instance as nitroglycerine, dinitrotoluene, or trinitroluene which plasticizes the surfaces of the individual particles and causes them to weld together into a grain. The particles of nitrocellulose may be those resulting from comminution of dense colloided nitrocellulose such as scrap cannon powder which may be ground to a suitable fineness, as for instance fine enough to pass through a 65 mesh screen. Larger or smaller particles may, however, be used under some circumstances but for a maximum of convenience particles passing a 65 mesh screen are recommended for the raw material to be used in the process of the present invention.

As an illustrative embodiment employing an explosive solvent, in whole or in part, as the agglomerating agent of the present invention the fine particles of nitrocellulose which may be termed "dust" may be suspended as by agitation in a bath of water. The quantity of water may conveniently be from 7½ to 8 times the dry weight of the nitrocellulose dust. This mixture may be agitated in a suitable still to form a thin slurry or suspension and, while the agitation continues, the solvent may be introduced in any suitable manner. For instance, the desired amount of nitroglycerine, as for instance 5 to 20% of weight of nitrocellulose, may be introduced as a straight oil or as a solution thereof in a suitable solvent, such as ethyl acetate or, if desired, the straight oil or solution may be first emulsified in a convenient quantity of water and this emulsion introduced into the bath containing the suspension of nitrocellulose particles.

If the nitroglycerine is employed in solution in a volatile solvent, such as ethyl acetate, isoproply acetate, butyl formates, or other water insoluble formates and insoluble ketones, such as methyl isobutyl ketone, a quantity of solvent about equal to the weight of nitrocellulose to be treated may be employed. In this the desired quantity of nitroglycerine, depending upon the ballistic properties desired, may be dissolved. Each of the aforementioned solvents is substantially insoluble in water and sufficiently volatile that it may be removed by distillation from the water bath below the boiling point of water.

As pointed out above, the solution of nitroglycerine may be introduced into the water-nitrocellulose suspension while the latter is being agitated. The nitroglycerine solution may be injected as through a suitable spraying device and the continuing agitation of the water nitrocellulose suspension will disperse the nitroglycerine solution in the form of globules throughout the bath. When safety conditions permit the straight nitroglycerine may be likewise introduced in the form of a spray into the bath and the agitation therein may be relied upon to accomplish the desired dispersion in the form of globules. In this case suitable emulsifying agents, such as the oleates, Turkey red oil, Saponin, etc. may be employed, if desired, to accomplish the degree of dispersion desired.

As an alternative procedure the straight nitroglycerine oil or the nitroglycerine solution may be first emulsified in the convenient quantity of water with suitable emulsifying agents, as mentioned above, if desired. Thereafter this emulsion or dispersion of nitroglycerine or nitroglycerine solution in water may be introduced into the water nitrocellulose mixture while agitation of the latter continues.

When the nitroglycerine is employed in the form of a solution in a volatile solvent the temperature of the bath containing the various ingredients may be gradually elevated to a temperature sufficient to vaporize and drive off the volatile solvent from the bath. During this time the agitation continues and may be controlled so as to secure the desired degree of dispersion of the nitroglycerine. In general, the more violent the agitation the smaller the clusters or agglomerates of fine particles which produce the grains. The size of the resultant grains may be controlled by controlling the size of the droplets of the solvent globules since, in most cases, the size of the resultant powder grains is slightly larger than the globules of the nitroglycerine dispersion. Indeed, if the solvent is emulsified with a convenient quantity of water before being introduced into the water nitrocellulose suspension, practically all of the resulting grains will be slightly larger than the globules of solvent; while, if the solvent is dispersed directly by agitation in the water nitrocellulose suspension, about 90% of the grains formed will ordinarily be larger than the droplets or globules of solvent.

While the bath is maintained in a suitable state of suspension and dispersion the temperature may be raised so as to drive off the volatile solvent. This may be accomplished over a period of three hours or longer, finally reaching the temperature of boiling of the volatile solvent (but below the boiling point of water).

In the event that the nitroglycerine is employed in the form of straight oil rather than the solution in a volatile solvent, it will be understood that no distillation is carried out and hence it is only necessary to heat the bath to a sufficient temperature and for a sufficient length of time to secure the desired degree of hardening and penetration of the nitroglycerine into the nitrocellulose particles. For instance, the temperatures may be gradually raised to about 65° C. over a period of thirty to sixty minutes.

The solution of nitroglycerine which is introduced into the bath may also contain suitable amounts of other modifying agents, such as deterrents, for instance dinitrotoluene, trinitrotoluene, dialkylphthalates, etc., and may also contain a small quantity of a stabilizing agent, such as diphenylamine, if desired. In some cases where it is not desired to introduce nitroglycerine into the grains the trinitrotoluene or dinitrotoluene may be dissolved in the solvent and may be introduced in the same manner as described above with reference to the nitroglycerine. The amount of such deterrent will be determined by the ballistic characteristics sought but may be up to about 15%. For instance, 8% T. N. T. may be employed with or without the desired quantity of nitroglycerine.

As a specific illustration a solution of 30 pounds of nitroglycerine, 15 pounds of D. N. T. or T. N. T. and 1½ pounds of diphenylamine may be dissolved in about 300 pounds of ethyl acetate. This solution is sufficient for the treatment of about 300 pounds of nitrocellulose dust.

Moreover, a deterrent substance, such as dinitrotoluene or trinitrotoluene which is normally solid may be employed to agglomerate particles of the nitrocellulose dust so as to produce grains even without employing a volatile solvent as hereinbefore described. This may be accomplished by melting the D. N. T. or T. N. T. and

introducing it into the water nitrocellulose suspension in that condition. Hence the molten D. N. T. or T. N. T. may be introduced directly into the suspension of nitrocellulose in water and the desired amount of dispersion accomplished by agitation of the suspension. In this case, however, care should be taken that the temperature of the water containing the nitrocellulose in suspension is above the melting point of the plasticizing deterrent employed whether D. N. T. or T. N. T. Another convenient method, however, is to emulsify the molten D. N. T. or T. N. T. in a suitable quantity of hot water so as to secure the desired degree of dispersion or size of globules as a separate emulsion and then introduce this into the nitrocellulose water suspension while the latter is being agitated.

In carrying out the process according to this embodiment, wherein the temperature of the bath is maintained above the melting point of an explosive plasticizer, such as D. N. T. or T. N. T., until the desired agglomeration has taken place, hardening of the grains may be expedited by reducing the temperature of the bath below the melting point of the explosive plasticizer. This reduction in temperature should, however, not take place until after the particles have agglomerated to form grains of the desired size and become welded to each other through the agency of the nitrocellulose surface colloid. When the temperature of the bath is reduced below the melting point of the explosive plasticizer the plasticized nitrocellulose becomes hardened so as to form a permanent bond between the particles.

However, unless a large proportion of explosive plasticizer is employed, it is not necessary to so reduce the temperature in order to harden the grains since penetration of the plasticizer into the particles after surface colloiding will tend to harden the surface layers and weld the particles together.

Although in the foregoing description reference has been partly made to the use of particles of dense colloided nitrocellulose, it is to be understood that a double base powder, such for instance as Ballistite (nitrocellulose-nitroglycerine) may be ground to the desired fineness and agglomerated to produce grains in the same manner as that hereinbefore described with reference to dense colloided nitrocellulose generally. For example 300 pounds of Ballistite ground to a fineness of, for instance 65 mesh, may be suspended in water and while the agitation continues 300 pounds of ethyl acetate may be introduced whereupon an agglomeration of the particles of Ballistite will take place and hardened grains will result upon evaporation of ethyl acetate.

After the nitroglycerine or other plasticizing solution is introduced into the water nitrocellulose suspension and the solution becomes dispersed therein in the form of droplets, a plurality of particles of the nitrocellulose dust will agglomerate about the globules of nitroglycerine or solution and will become sufficiently plasticized so as to be stuck together to produce a grain. After the volatile solvent has been driven off, as described above, or after sufficient hardening has been accomplished, to secure a firm grain, the heating and agitation may be discontinued. Thereafter suitable coatings, as for instance modifying agents, either deterrents or accelerators, or both, may be applied in the usual manner to the grains in order to achieve the desired ballistic characteristics as described in the prior copending applications hereinbefore mentioned.

From the foregoing description, it is apparent that the present invention accomplishes its objects and provides a method for making propellant powder grains whereby individual dense colloided powder particles are agglomerated together through the agency of an explosive plasticizer so as to produce a grain containing not only nitrocellulose but another explosive, such as nitroglycerine, dinitrotoluene or the like therein.

The powder grains produced in accordance with the present invention are, therefore, small clusters of completely gelatinized particles of nitrocellulose dust. The respective particles which form the individual grains are cemented together by the action of the solvent to such an extent that, although the resultant grain may be of small size, it is of substantially spherical shape although undulations will exist in the surface thereof.

It is to be understood that this invention is not limited to the details of procedure hereinbefore specifically described for the purpose of illustration but that variations and modifications may be made without departing from the spirit of this invention and that other equivalent explosive plasticizers may be employed with or without those mentioned herein. For instance, nitroglycols or a mixture of nitroglycols with nitroglycerine may be employed wherever nitroglycerine has been described. The words "coagmented" and "coagmenting", as used in the appended claims, are to be undertsood as implying a condition in which a plurality of individual particles of dense smokeless powder are adhered together through the agency of a foreign substance, or cohered together through the agency of the mutual colloid of the particles, or by a combination of such cohesive and adhesive agencies; but without restriction as to the agent, or combination of agents, employed for accomplishing such adhesion or cohesion, or both. It is to be distinctly understood, therefore, that such variations and modifications, or the use of such individual features or subcombinations of features as do not depart from the spirit of this invention are, although not specifically described herein, contemplated by and within the scope of the appended claims.

Having described the invention, what is claimed is:

1. A propellant powder grain composed of particles of dense smokeless powder coagmented together.

2. A propellant powder grain composed of particles of dense smokeless powder coagmented together, said grain containing a combustion modifier.

3. A propellant powder grain composed of particles of dense smokeless powder coagmented together, said grains being coated with a combustion modifying agent.

4. A propellant powder grain composed of particles of dense smokeless powder coagmented together, said grain being coated with nitroglycerine.

5. A propellant powder grain composed of coagmented particles of dense smokeless powder and containing a combustion modifying agent between the particles.

6. A propellant powder grain composed of coagmented particles of dense smokeless powder and containing a deterrent between the particles.

7. A propellant powder grain composed of par-

ticles of dense smokeless powder agglomerated with nitroglycerine.

8. A propellant powder grain composed of particles of dense smokeless powder agglomerated with nitroglycerine and a nitrotoluene.

9. A propellant powder grain composed of agglomerated particles of dense smokeless powder and containing trinitrotoluene between the particles.

10. A propellant powder grain composed of agglomerated particles of dense smokeless powder and containing nitroglycerine between the particles.

11. A double base smokeless powder in the form of grains comprising finely divided particles of dense colloided nitrocellulose agglomerated by means of an explosive solvent therefor.

12. As a new article of manufacture, grains of smokeless powder suitable for use as propellant powder, said grains comprising, particles of dense colloided nitrocellulose agglomerated together with nitroglycerine.

13. A double base smokeless powder in the form of grains comprising single base nitrocellulose powder fines surface treated with nitroglycerine and agglomerated.

14. A double base smokeless powder in the form of grains comprising single base nitrocellulose powder fines of a size not exceeding about thirty mesh surface treated with nitroglycerine and agglomerated into a plurality of grains.

15. A double base smokeless powder in the form grains comprising single base nitrocellulose powder fines surface colloided with nitroglycerine and agglomerated and bound together through the medium of their surface colloid into grains.

16. In the art of making propellant powder grains, the process comprising, coagmenting particles of dense colloided nitrocellulose dust together.

17. In the art of making propellant powder grains, the process comprising, comminuting dense smokeless powder to form fine particles, treating the particles with a solvent and agglomerating a plurality of individual particles into grains.

18. In the art of making propellant powder grains, the process comprising, comminuting dense smokeless powder to form fine particles, treating the particles with an agglomerating agent and agglomerating the particles into grains.

19. In the art of making propellant powder grains, the process comprising, coagmenting particles of gelatinized nitrocellulose dust together, and coating the resultant grains with a deterrent.

20. In the art of making propellant powder grains, the process comprising, agglomerating particles of gelatinized nitrocellulose dust, and coating the resultant grains with an accelerator.

21. In the art of making propellant powder grains, the process comprising, comminuting dense smokeless powder to form fine particles, treating the particles with nitroglycerine, and agglomerating a plurality of particles together to form grains.

22. The process of making a propellant powder comprising treating fine particles of dense colloided nitrocellulose with nitroglycerine and agglomerating a plurality of individual particles into grains.

23. The process of making a propellant powder comprising treating fine particles of colloided nitrocellulose with an explosive solvent therefor and agglomerating a plurality of individual particles into grains.

24. In the art of making propellant powder grains, the process comprising, suspending finely divided particles of dense colloided nitrocellulose in a body of water, suspending a combustion modifying agent in liquid form in the body of water with the particles of dense colloided nitrocellulose, and agitating the mixture until the combustion modifying agent attacks and agglomerates the particles of nitrocellulose.

25. A process of making grains of powder suitable for use as propellant powder, said process comprising, surface plasticizing finely divided dense colloided nitrocellulose with a combustion modifying agent mixed with a volatile solvent for the nitrocellulose while both the nitrocellulose and combustion modifier are suspended by agitation in water, continuing said agitation until a plurality of particles of surface plasticized nitrocellulose have united together in grain form, then driving off the volatile solvent for the nitrocellulose by heat.

26. The process of making grains of powder suitable for use as propellant powder, the process comprising, suspending finely divided dense colloided nitrocellulose in water, mixing an explosive modifying agent with a liquid miscible therewith which is substantially immiscible with water and is likewise a solvent for nitrocellulose, suspending said admixture in the water with the nitrocellulose by agitation, the speed of agitation in relation to the quantity of water used being such as to provide globules of modifying agent and solvent suspended in the water, applying heat to drive off the solvent, and maintaining the suspension by agitation until hardened colloided powder grains of nitrocellulose and modifying agent have been formed.

27. A process of making grains of powder suitable for use as propellant powder, said process comprising, treating finely divided dense colloided nitrocellulose with an explosive modifying agent mixed with ethyl acetate while both the nitrocellulose and modifier are suspended by agitation in water, continuing said agitation until grains have been formed, and driving off the ethyl acetate by heat.

28. The process of making grains of powder suitable for use as propellant powder, which comprises, maintaining finely divided dense colloided nitrocellulose in suspension in water by agitation, maintaining a mixture of combustion modifying agent and a volatile solvent for both the combustion modifying agent and the nitrocellulose in suspension in a body of water by agitation, combining said suspensions, continuing the agitation until colloided grains of nitrocellulose and combustion modifying agent have been formed, and driving off the volatile solvent by heat.

29. The process of making grains of powder suitable for use as propellant powder, said process comprising, suspending finely divided dense colloided nitrocellulose in water, suspending nitroglycerine in water by agitation, controlling the agitation to produce globules of nitroglycerine slightly smaller than the desired powder grains, admixing said suspensions, and continuing the agitation until the nitroglycerine has colloided the nitrocellulose into powder grains.

30. The process of making grains of powder suitable for use as propellant powder, the process comprising, mixing nitroglycerine with a liquid miscible therewith which is substantially immisci-

ble with water and is likewise a solvent for nitrocellulose, suspending said admixture in water by agitation, the speed of agitation in relation to the quantity of water used being such as to provide globules of nitroglycerine and solvent suspended in the water, the size of the globules being slightly smaller than the size of the desired powder grains, suspending finely divided dense colloided nitrocellulose in water, admixing the two suspensions while maintaining the nitroglycerine and solvent in said globular condition, applying heat to drive off the solvent, and maintaining the suspension by agitation until hardened colloided powder grains of nitrocellulose and nitroglycerine have been formed.

31. A process of making grains of powder suitable for use as propellant powder, said process comprising, surface plasticizing finely divided dense colloided nitrocellulose with nitroglycerine mixed with ethyl acetate while both the nitrocellulose and nitroglycerine are suspended by agitation in water, continuing said agitation until grains have been formed, and driving off the volatile solvent for the nitrocellulose by heat.

32. The process of making grains of powder suitable for use as propellant powder, the process comprising, mixing nitroglycerine with a liquid miscible therewith which is substantially immiscible with water and is likewise a solvent for nitrocellulose, suspending said admixture in water by agitation, the speed of agitation in relation to the quantity of water being such as to provide globules of nitroglycerine and solvent suspended in the water, the size of the globules being slightly smaller than the size of the desired powder grains, suspending finely divided dense colloided nitrocellulose in water, adding the former suspension to the latter while maintaining the nitroglycerine and solvent in said globular condition, applying heat to drive off the solvent, and maintaining the suspension by agitation until hardened colloided powder grains of nitrocellulose and nitroglycerine have been formed.

33. A process of making grains of powder suitable for use as propellant powder, said process, comprising, agitating nitroglycerine in water containing finely divided dense colloided nitrocellulose so as to produce globules of nitroglycerine slightly smaller in size than the desired powder grains, and maintaining said globules of nitroglycerine in said condition for a period sufficient to enable them to surface plasticize the particles of finely divided nitrocellulose and agglomerate the finely divided nitrocellulose into grains.

34. In the process of forming propellant powder grains from colloided nitrocellulose and nitroglycerine in water suspension, a method of determining the size of the powder grains produced, which method comprises, agitating nitroglycerine in an aqueous medium to disperse the nitroglycerine in the form of globules, and maintaining the nitroglycerine in the form of globules of size slightly smaller than the size of the desired powder grains during the grain forming process.

35. The process of making grains of powder suitable for use as propellant powder, said process, comprising, treating finely divided dense colloided nitrocellulose in water suspension with globules of nitroglycerine slightly smaller in size than that of the desired powder grains.

36. The process of making grains of powder suitable for use as propellant powder, said process, comprising, treating finely divided dense colloided nitrocellulose in water suspension with globules of nitroglycerine and a nitroglycerine miscible substantially water immiscible solvent for the nitrocellulose, said globules being slightly smaller in size than that of the desired powder grains.

FREDRICH OLSEN.

CERTIFICATE OF CORRECTION.

Patent No. 2,235,298. March 18, 1941.

FREDRICH OLSEN.

It is hereby certified that error appears in the printed specif
of the above numbered patent requiring correction as follows: Page 4
column, line 39, for "isoproply" read --isopropyl--; page 6, first c
line 33, claim 15, for the word "form" read --form of--; line 60, cla
for "agglomerating" read --coagmenting--; line 61, same claim, after
and before the comma insert --together--; and that the said Letters
should be read with this correction therein that the same may conf
the record of the case in the Patent Office.

Signed and sealed this 29th day of April, A. D. 1941.

Henry Van Arsdale
Acting Commissioner of P

(Seal)

CERTIFICATE OF CORRECTION.

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It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 4, first column, line 39, for "isoproply" read --isopropyl--; page 6, first column, line 33, claim 15, for the word "form" read --form of--; line 60, claim 20, for "agglomerating" read --coagmenting--; line 61, same claim, after "dust" and before the comma insert --together--; 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 29th day of April, A. D. 1941.

(Seal)

Henry Van Arsdale,
Acting Commissioner of Patents.