(No Model.) 2 Sheets—Sheet 1.

F. G. & P. S. DU PONT.

PROCESS OF MAKING SMOKELESS EXPLOSIVES.

No. 503,583. Patented Aug. 22, 1893.

Fig. 1a.

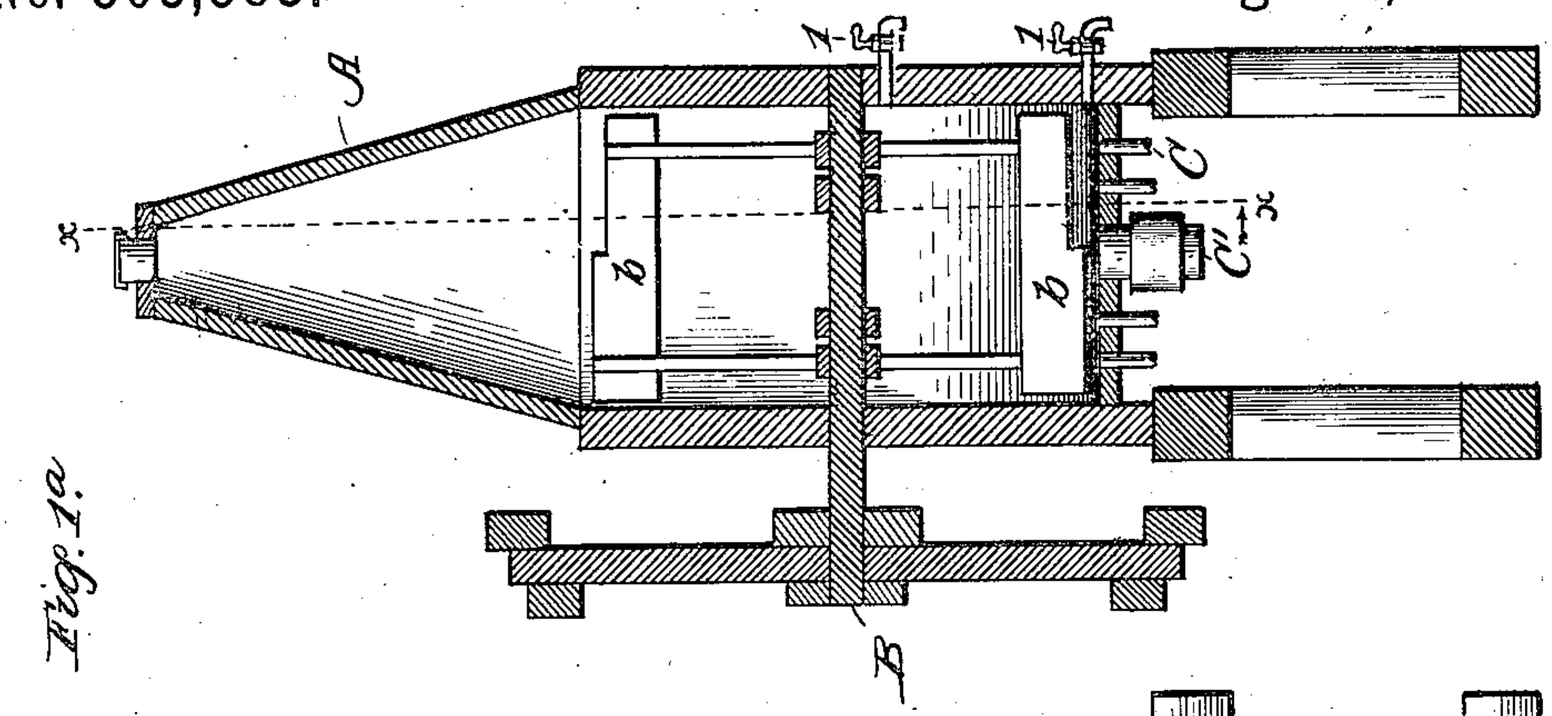
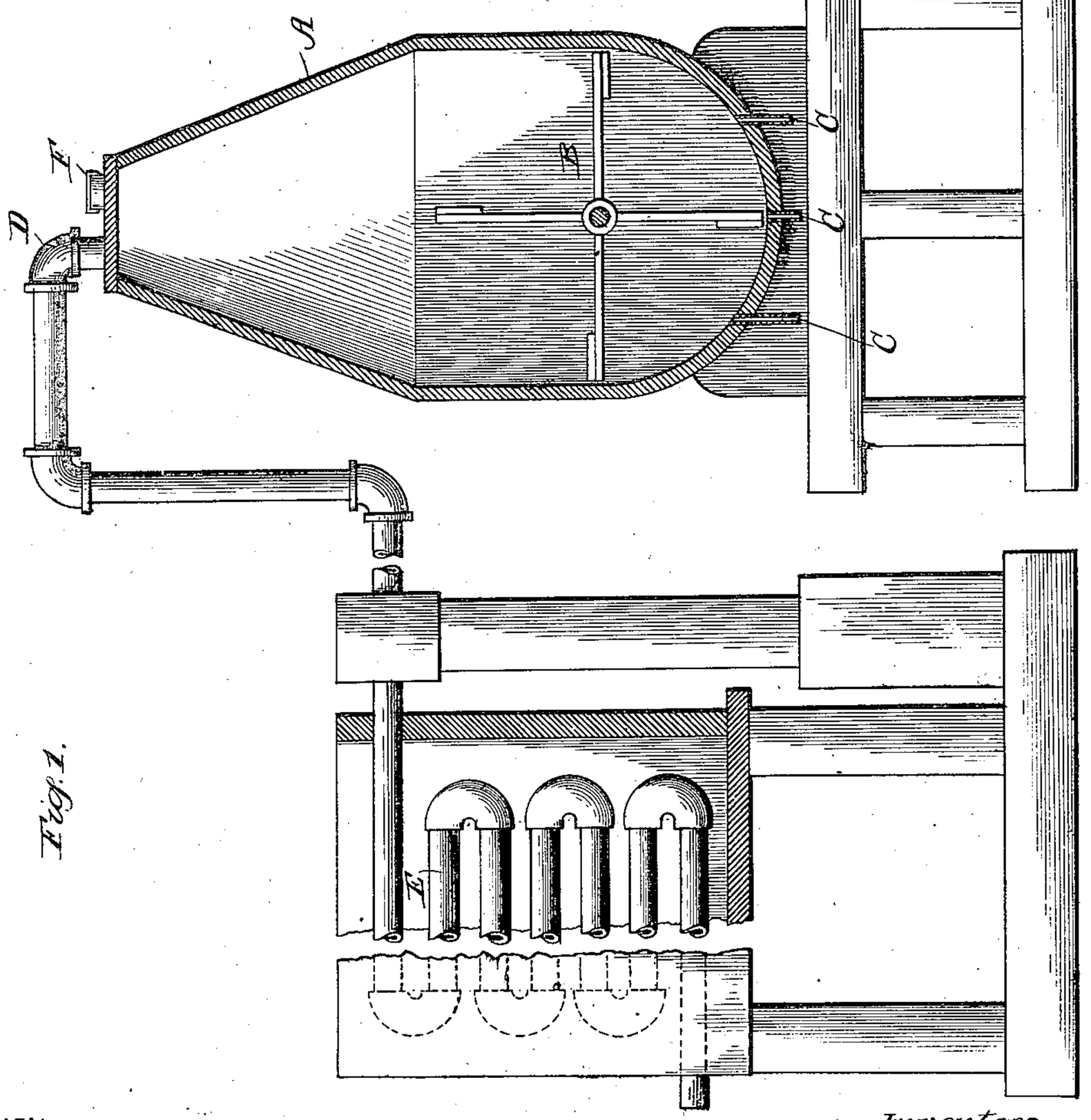


Fig. 1.



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(No Model.) 2 Sheets—Sheet 2.

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Fig. 2a.

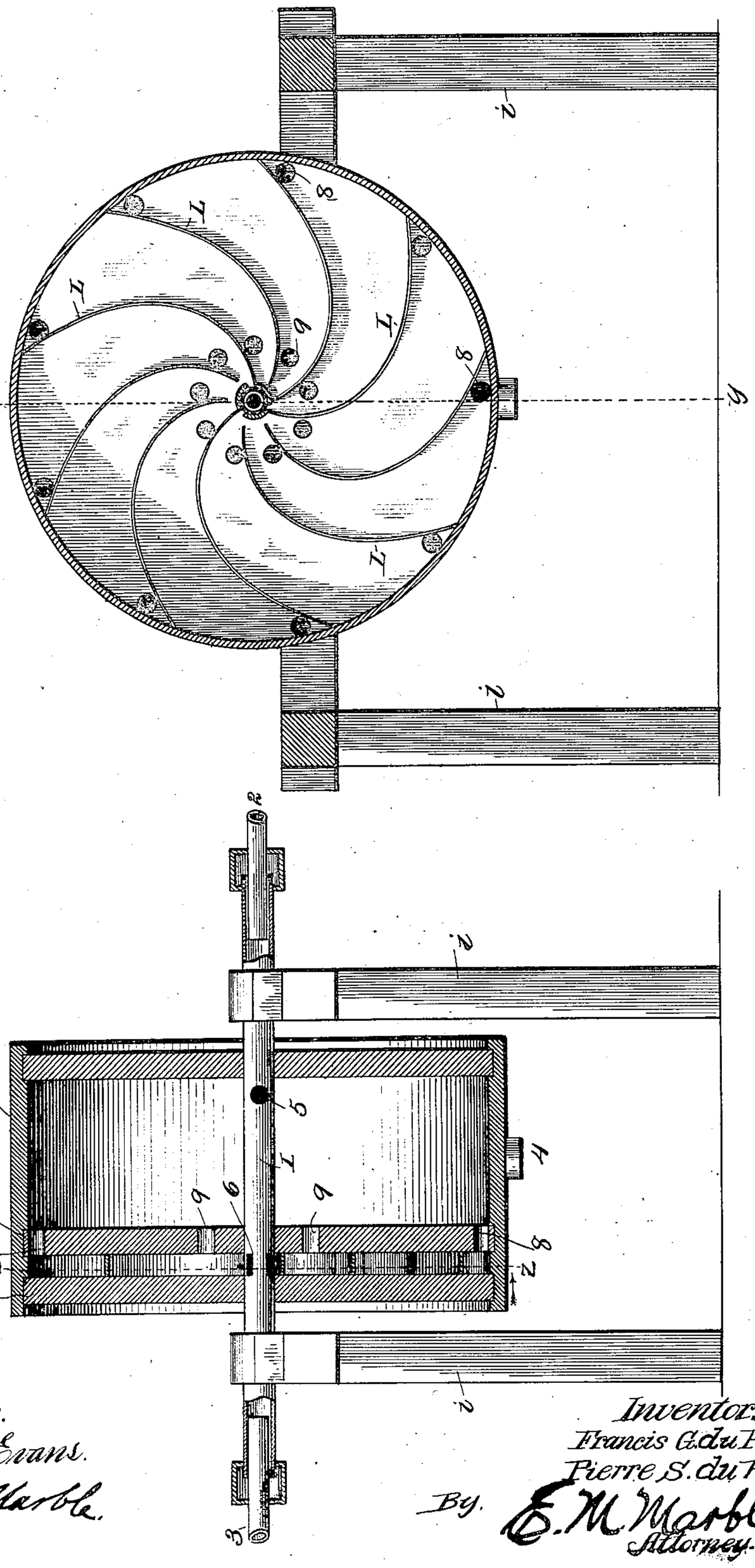

Fig. 2.

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UNITED STATES PATENT OFFICE.

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PROCESS OF MAKING SMOKELESS EXPLOSIVES.

SPECIFICATION forming part of Letters Patent No. 503,583, dated August 22, 1893.

Application filed December 21, 1892. Serial No. 455,901. (No specimens.)

To all whom it may concern:

Be it known that we, FRANCIS G. DU PONT and PIERRE S. DU PONT, citizens of the United States, residing at Wilmington, in the county of New Castle and State of Delaware, have invented certain new and useful Improvements in Explosives; and we do hereby declare the following to be a full, clear, and exact description of the invention, such as will enable others skilled in the art to which it appertains to make and use the same.

Our invention relates to improvements in explosives to be used in small arms, large guns, as a bursting charge for shells, or for any other explosive use to which it may be suited, and it consists in an improved process for producing an explosive from nitro-cellulose, consisting in granulating the nitro-cellulose by agitating therewith, when suspended in a liquid, a suitable solvent of the same in proper proportions, which will be hereinafter fully described, and particularly pointed out in the claims.

The main object of our invention is to produce an explosive which will be smokeless, or nearly so; which, while imparting a high muzzle velocity to the projectile, will not cause undue pressure in the gun barrel at the moment of firing, and which can be easily and safely handled and stored.

Nitro-cellulose in some of its forms, usually that of gun cotton, has frequently been used as the basis of smokeless powders, because it unites in itself so many of the qualities which good smokeless powders must possess; that is to say, it is very stable when properly prepared, burns or explodes with very little smoke, possesses great power as an explosive, and is not hydroscopic enough to impair its keeping qualities. The explosive force of gun cotton, or other nitrated fiber, when used by itself is so great, however, and the difficulties of securing an even rate of combustion so hard to overcome, that its use by itself is dangerous in fire arms.

Many attempts have been made to control or diminish the explosive force of gun cotton, while preserving all of its advantageous properties. These attempts have in general proceeded along three lines, viz: First: That of subjecting the gun cotton to great pressure, by which all air spaces, in which the explosive gases might collect, are removed from it, and it is rendered solid and compact. Second: That of mixing with the gun cotton certain substances which lessen its rate of combustion, the substances added being less violent in explosive force than the gun cotton. For this purpose nitro-benzole, nitro-naphthaline, camphor, and the high boiling ketones and acetates have been used. Third: The solvents of gun cotton which have been added may be entirely evaporated, leaving the gun cotton in a state resembling celluloid, in which its condition is changed from the fibrous to the amorphous state, in which latter state it is more compact than the original gun cotton.

Our invention belongs to the third of the classes above specified, but it differs from all other processes now known or used in that the gun cotton is treated in a wet way, by suspending it in a fluid and mixing with it a suitable solvent, which produces a granular condition hereinafter described.

We have discovered that the various solvents of gun cotton, especially the nitro-derivatives of the aromatic group, have a tendency, when mixed with a fluid in which finely divided gun cotton is suspended, to seize the gun cotton and leave the water clear, forming in the water a more or less coherent mass; and that when a suitable solvent is added in proper proportions, and the mass is even slightly agitated, a well defined granular condition is the result, the solvent collecting the particles of nitro-cellulose into grains, and forming a coating around the same. These grains, after they have undergone a process of hardening by rotation in a barrel, and removal of excess of solvent and water contained in the grains by heat, the said heat being applied either before or during the process of rotation, or by rotation in a barrel in an atmosphere of steam, in which case the removal of the excess of solvent and water contained in the grains will take place at the same time as the rotation, become sufficiently firm to be graded according to size, and constitute a valuable smokeless powder, the violence of whose explosive action can be diminished by varying the duration of the above

processes, or it may be still further diminished by the addition of a substance to moderate its action. We have found nitrated rosin and nitrated turpentine especially suited for this purpose, and we add them in the manner and proportion which will be hereinafter stated.

In the drawings accompanying and forming a part of this application, we have represented the apparatus which we use in carrying out our process.

Figure 1, which is taken on the line *x x* Fig. 1ª, looking in the direction of the arrow, illustrates the churn in which the mixture of the gun cotton with the solvent takes place, Fig. 1ª being a sectional view of the same, and Fig. 2 being a representation of the rotating barrel in which the grains formed in the churn shown in Fig. 1 are hardened and rounded, and thus fitted for use. It is taken on the line *y y* Fig. 2ª. Fig. 2ª is a view taken on the line *z z* Fig. 2, looking in the direction of the arrow. It shows the double series of apertures in the inner bulkhead of the barrel, and also the series of cycloidal chutes which conduct the water of condensation, received through the outer series of apertures in the bulkhead, to the apertures in the central pipe forming the axis of the barrel, by which pipe the said water is conveyed away.

Before proceeding with a description of our process, we will describe the apparatus used in carrying it out.

Referring to Figs. 1 and 1ª, A represents a churn, in which, on a shaft B, are rotated the blades *b*, each of which is formed with a notched outer surface to promote currents from side to side in the contents of the churn during rotation. Steam inlet pipes C are provided which are relatively small in diameter, so that the steam will be well distributed through the contents of the churn, and a single steam outlet pipe D, which leads to the refrigerating coils E, where the vapors which it conducts may be condensed. An opening F is provided for the admission of material, and an opening C′ to aid in flushing the interior of the churn when desired. Test cocks 11 are also provided, in order that the condition of the contents of the churn may at any time be inspected.

In Figs. 2 and 2ª the rotating barrel is represented in which the grains of explosive produced in the churn just described are hardened and prepared for use. This barrel, H, is provided with a tubular axis I, journaled in the supports *i i*, to which axis steam is conducted by the pipe 2, and from which steam is conveyed by the pipe 3. The barrel is designed to facilitate the escape of both water and steam from the interior of the same, the steam carrying off the solvent used, and to this end is formed with an inner bulkhead K, which is placed in close proximity to the bulkhead H^2 of the barrel. In the inner bulkhead are formed two series of apertures, one around its periphery 8 for the separation of water from the contents of the barrel, and one near its center 9 for the escape of steam from the same. Between the two bulkheads K and H^2 is arranged a series of cycloidal guides or chutes L, all of which converge toward the central axis.

In the operation of the barrel, steam is admitted to its interior through the aperture 5 in the central axis, and passes out through the apertures 8 and 9 in the bulkhead K, and thence through the axis I and pipe 3, carrying with it, as will be hereinafter explained, the excess of the solvent found with the grains of explosive. Water of condensation, as fast as formed, passes through the lower series of apertures in the bulkhead K at the lower stage of the revolution of the barrel into the space between said bulkhead K and bulkhead H^2, and is conducted by the cycloidal chutes toward the axis of the barrel, then passing out through the apertures 6 formed at this point.

Proceeding now with our process, we place in the churn A about ninety liters of a suitable liquid, such as water, which may either be pure, or mixed with any salt. When water is used, we sometimes dissolve in it chloride of calcium, common salt, saltpeter, or other salt, as we can thus modify in many ways the characteristics of the grains formed. The proportion in which these ingredients are added depends somewhat upon which one is used. In the case of salpeter, about a half saturated solution will answer very well, or even one of greater strength, say a saturated solution at a temperature of 60° Fahrenheit. We add to the liquid about six kilograms of finely divided gun cotton, beating it up with the water in the churn until a thorough mixture has taken place, and the gun cotton is uniformly suspended in the liquid. When this has been accomplished, we add to the mixture a suitable solvent of gun cotton, such as nitro-benzole, to the extent of about three times the weight of the gun cotton. By the action of the solvent upon the suspended gun cotton a plastic, flocculent mass is formed, which by slight agitation becomes granulated. When the agitation in the churn is now continued, a well defined granular product is produced, the solvent (in this case nitro-benzole) collecting the particles of gun cotton, and forming them into grains, which are as yet somewhat soft and pulpy as they contain considerable water, and as there is an excess of solvent. The agitation aids the granulation by keeping the particles of gun cotton suspended, and thus enabling the action of the solvent to be carried on uniformly upon all the particles of gun cotton in the churn. During this action, steam may be admitted through the bottom of the churn, and this, as it passes out through the refrigerating coils, carries with it a portion of the solvent, which is recovered in the said coils. This action of the steam is not, however, essential to the production of the

grains, though it assists in their formation, and may be omitted without detriment to the product.

Instead of using nitro-benzole to produce the granulation nitro-toluol or nitro-xylol may be used, or other solvents. The solvent used, however, must not be miscible to any great extent in the liquid in which the gun cotton is suspended, as if it is so miscible, it will not leave the liquid readily and form the desired granulation. The suitability of the solvent is thus determined. We do not confine ourselves to the proportions mentioned of the gun cotton and solvent, although these have been found to give good results, as the proportion may be altered and still a good granulation is obtained. The contents of the churn are now removed, and the grains are placed in the revolving barrel shown in Figs. 2 and 2ª. By rotating the barrel for a greater or less time without the admission of steam, the grains are compacted and rounded. The barrel is then partially filled with water, and rotated in such a direction as to prevent the cycloidal chutes from carrying off the water, steam being admitted, and the rotation continued until a further portion of the solvent is carried off. When this action has proceeded sufficiently far, the direction of rotation is changed, and the flow of steam continued until the excess of solvent is entirely removed. The action of this latter process, namely the rotation in an atmosphere of steam, is to increase to a very great degree the density of the grain, and to round and compact the same, fitting it for its destined use as an explosive.

The density and also the size of the grain formed may be varied by changing the proportions of the ingredients, and by altering the length of time of the preliminary rolling in the revolving barrel. The principle at the root of the matter is this: If the original grains formed in the churn have a large quantity of nitro-benzole, or other solvent used, they will be quite plastic, and during the preliminary rolling in the barrel will tend to gather up any loose material which may happen to be among the grains, thus becoming larger; while if the proportion of the nitro-benzole, or other solvent, to the gun cotton in the churn be made smaller, the grains formed will be less plastic, and will be apt to remain nearly the same size as when produced in the churn for the reason that they are less plastic, and have more consistence, and consequently do not tend to pick up material as they will if more plastic. Furthermore, the quantity of solvent used in proportion to the gun cotton has some influence upon the preliminary grains in the churn, the grains in the churn always being larger when a greater percentage is used from the fact that the combination is of a more plastic nature, and tends to unite in larger particles. We may also remove the grain at any stage of the beforementioned processes, and evaporate the solvent by boiling the grain with water in the ordinary manner. The construction of barrel described, however, is well suited for the purpose of hardening and rounding the preliminary grains formed in the churn.

In order that the grains formed by the above process may be tempered in explosive force to any degree required, we dissolve in the solvent used in the granulation process from two and one-half to ten per cent. of a moderating agent, and find especially adapted for this purpose nitrated rosin or nitrated turpentine, *i. e.*, rosin or Venice turpentine treated with nitric acid, though we do not confine ourselves to the use of these substances, or to the proportions mentioned. Instead of adding the moderating agent in the manner described, it may be added to the grain after it is finished, by means of solution in a volatile solvent, or in any other manner.

While we have described the gun cotton as being diffused in water, it is evident that any other fluid which will cause a better or as good a distribution of the particles of gun cotton in the churn may be used instead, and we do not confine ourselves to the use of water as a suspending liquid; nor do we confine ourselves to the use of the particular form of nitro-cellulose known as gun cotton. We also do not confine ourselves to the particular solvents mentioned, or to the exact proportions in which they are added, or to the particular apparatus employed.

What we claim as new, and desire to secure by Letters Patent, is—

1. The herein described process of making a smokeless explosive, which consists in suspending nitro-cellulose in a liquid not a solvent of the same, granulating the nitro-cellulose by agitating therewith in proper proportions a suitable solvent not miscible to any great extent in the liquid used to suspend the nitro-cellulose, and solidifying the grains thus formed, substantially as described.

2. The herein described process of making a smokeless explosive, which consists in suspending nitro-cellulose in a liquid not a solvent of the same, granulating the nitro-cellulose by agitating therewith in proper proportions a suitable solvent not miscible to any great extent in the liquid used to suspend the nitro-cellulose, and solidifying the grains thus formed by heat, substantially as described.

3. The herein described process of producing a smokeless explosive, which consists in suspending nitro-cellulose in a liquid not a solvent of the same, granulating the nitro-cellulose by agitating therewith in proper proportions a suitable solvent not miscible to any great extent in the liquid used to suspend the nitro-cellulose, and solidifying the grains thus formed by agitation in an atmosphere of steam, substantially as described.

4. The herein described process of producing a smokeless explosive, which consists in suspending nitro-cellulose in a liquid not a solvent of the same, granulating the nitro-cellulose by agitating therewith in proper pro-

portions a suitable solvent not miscible to any great extent in the liquid used to suspend the nitro-cellulose, and solidifying the grains thus formed by rotation in an atmosphere of steam, substantially as described.

5. The herein described process of producing a smokeless explosive, which consists in suspending nitro-cellulose in a liquid not a solvent of the same, granulating the nitro-cellulose by agitating therewith in proper proportions a suitable solvent not miscible to any great extent in the liquid used to suspend the nitro-cellulose, hardening and rounding the grains thus formed by rotation, and further solidifying the grains by rotation in an atmosphere of steam, substantially as described.

6. The herein described process of producing a smokeless explosive, which consists in suspending nitro-cellulose in a liquid not a solvent of the same, granulating the nitro-cellulose by agitating therewith in proper proportions, accompanied by injection of steam, a suitable solvent not miscible to any great extent in the liquid used to suspend the nitro-cellulose, and solidifying the grains thus formed by agitation in an atmosphere of steam, substantially as described.

7. The herein described process of producing a smokeless explosive, which consists in suspending nitro-cellulose in a liquid not a solvent of the same, granulating the nitro-cellulose by agitating therewith in proper proportions a suitable solvent not miscible to any great extent in the liquid used to suspend the nitro-cellulose, solidifying the grains thus formed, and moderating the action of the same by the addition, during the above operation, of a suitable moderating agent, substantially as described.

8. The herein described process of producing a smokeless explosive, which consists in suspending nitro-cellulose in a liquid not a solvent of the same, granulating the nitro-cellulose by agitating therewith in proper proportions a suitable solvent not miscible to any great extent in the liquid used to suspend the nitro-cellulose, and in which is dissolved a moderating agent, and solidifying the grains thus formed, substantially as described.

9. The herein described process of producing a smokeless explosive, which consists in suspending nitro-cellulose in a liquid, such as water, which is not a solvent of the same, and in which is dissolved a suitable salt, granulating the nitro-cellulose by agitating therewith in proper proportions a suitable solvent not miscible to any great extent in the liquid used to suspend the nitro-cellulose, and solidifying the grains thus formed, substantially as described.

10. The herein described process of producing a smokeless explosive, which consists in suspending nitro-cellulose in a liquid not a solvent of the same, granulating the nitro-cellulose by agitating therewith in proper proportions, during injection of steam, a suitable solvent not miscible to any great extent in the liquid used to suspend the nitro-cellulose and in which is dissolved a moderating agent, and solidifying the grains thus formed by rotation, and afterward by rotation in an atmosphere of steam, substantially as described.

In testimony whereof we affix our signatures in presence of two witnesses.

FRANCIS G. DU PONT.
PIERRE S. DU PONT.

Witnesses:
J. H. HOFFECKER, Jr.,
FRANK M. SMITH.