(No Model.) 2 Sheets—Sheet 1

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PROCESS OF MAKING SMOKELESS EXPLOSIVES.

No. 503,585. Patented Aug. 22, 1893.

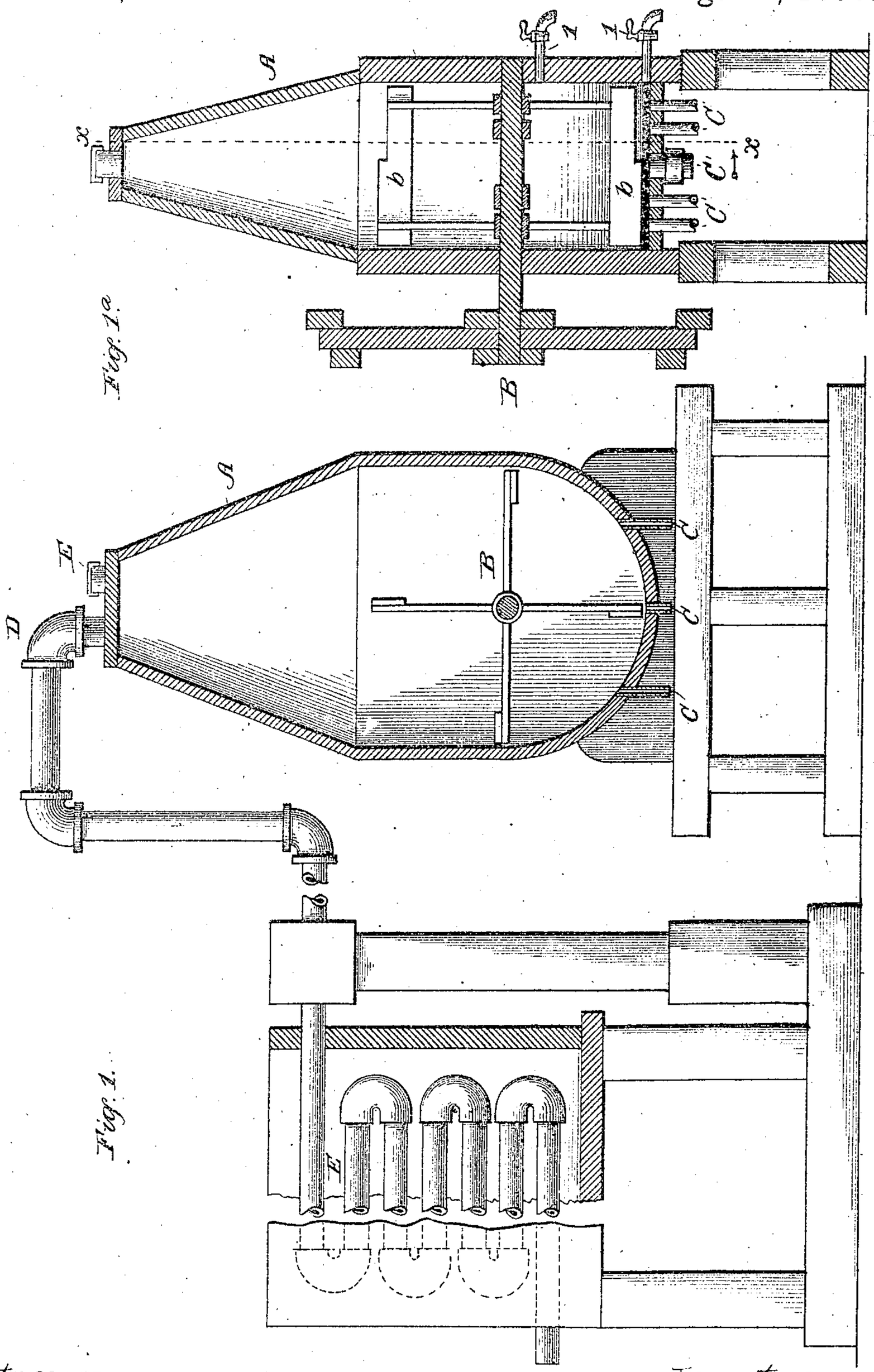

Fig. 1.

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(No Model.) 2 Sheets—Sheet 2.

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Fig. 2.

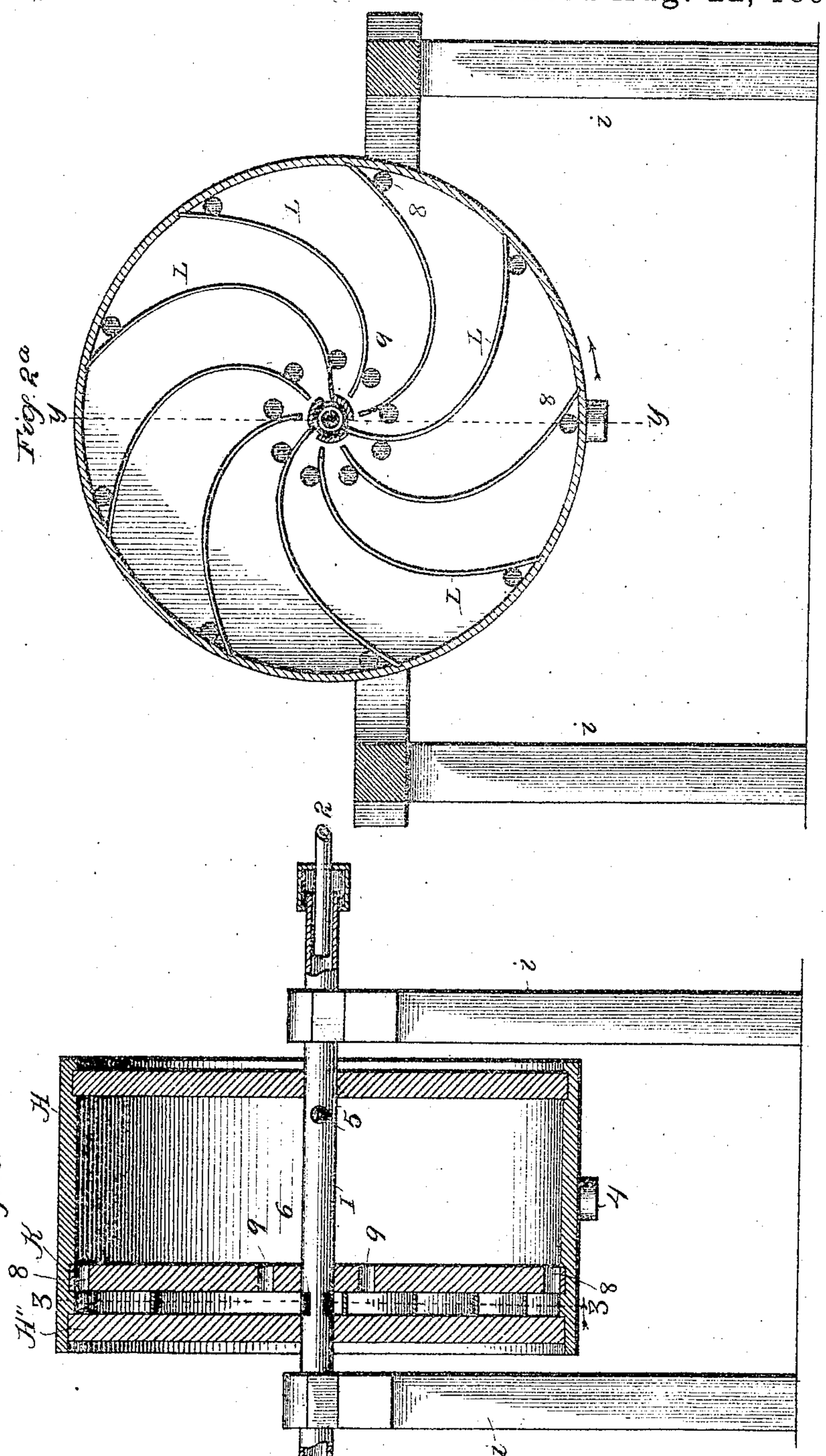

Fig. 2a.

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UNITED STATES PATENT OFFICE.

FRANCIS G. DU PONT AND PIERRE S. DU PONT, OF WILMINGTON, DELAWARE.

PROCESS OF MAKING SMOKELESS EXPLOSIVES.

SPECIFICATION forming part of Letters Patent No. 503,585, dated August 22, 1893.

Application filed March 17, 1893. Serial No. 466,488. (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 which are particularly adapted for imparting a high initial velocity to the projectiles of small bore rifles, such as the Mannlicher, the Krag-Jorgensen and the English .303, and it consists in an improved process for producing an explosive from nitro-cellulose, consisting of granulating the nitro-cellulose by agitating therewith, when suspended in a fluid, a solvent of the same in suitable 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 will be unaffected by being kept for a considerable space of time, 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. Our invention has for its special aim, however, the attainment of a higher initial velocity than has formerly been attainable by the use of smokeless explosives, with the aim of adapting the explosive to use with small bore rifles, such as those mentioned above.

In our application for Letters Patent for improvements in explosives, filed December 21, 1892, Serial No. 455,901, we described a process for producing smokeless powder, the distinguishing feature of which was the granulation of pure nitro-cellulose by mixing therewith a solvent of the same in suitable proportions. Basing our process on the tendency which we found was inherent in the various solvents of the nitro-celluloses, and especially the nitro-derivatives of the aromatic group, when mixed in a fluid in which finely divided nitro-cellulose is suspended, to seize the nitro-cellulose and leave the water clear, forming in the water a more or less coherent mass, we discovered that when the solvent is added in proper proportions, (and we found that the proportions of three parts of the solvent to one of the nitro-cellulose by weight was a good working proportion,) and the mass is slightly agitated a well defined granular condition was 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 boiling, 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 as to size preparatory to being placed on the market, 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 stated in said application. We have discovered, however, that increased hardness and consolidation of the grains thus formed may be obtained by subjecting the grains, either directly after they have been removed from the churn in which they were formed, or after they have been subjected to partial treatment by rotation in an atmosphere of steam in order to remove the excess of solvent, to the action of a gentle heat, not sufficient to cause vaporization of the solvent, but to remove some of the water condensed in the grains, this discovery being based upon the property, which we have found the grain possesses, of giving up its contained water before it parts with the solvent used in its preparation, and at a lower heat.

In the drawings accompanying and forming a part of this application, we have repre-

sented 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, illustrating the churn in which the mixture of the nitro-cellulose 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 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, by 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 1 1 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 away by the pipe 3. This barrel is designed to facilitate the escape of both water and steam, the latter 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 H² of the barrel. In this inner bulkhead are formed two series of apertures, one around its periphery S, 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 K² are arranged a series of cycloidal chutes or guides 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, passing out through the apertures 9 in the bulkhead K, from which it passes through the axis I and the pipe 3. Water of condensation, as fast as formed, and also the water separated from the grains of explosive, passes through the outer series of apertures in the bulkhead K at the lower part of the revolution of the barrel into the space between the said bulkhead K and bulkhead K², 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 water, or other liquid, 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. We add to the water about six kilograms of gun cotton, beating it up with the water in the churn until a thorough mixture has taken place. When this has been accomplished, we add to the mixture nitro benzole, or other suitable solvent of gun cotton not miscible to any great extent in the liquid used to suspend the gun cotton, such as nitro-toluol and nitro-xylol, to the extent of about three times the weight of the gun cotton. When the agitation of the contents of the churn is continued, the gun cotton assumes a well defined granular state, the grains formed being soft and containing considerable water. During this agitation 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. The size of the grains may be varied by changing the proportions of the ingredients and the duration of treatment. The above is an example of proportions which give good results. Instead of placing the grains thus formed directly into the rotating barrels shown in Figs. 2 and 2ª, and rotating said barrel with ingress of steam until the excess of volatile solvent has been entirely evaporated from the grains, as we do in the process described in the application for Letters Patent for improvements in explosives already referred to, we have found that better results as to the hardness and degree of consolidation of the resulting grains may be obtained by subjecting the grains to a gentle heat, ranging say from 120° to 180° Fahrenheit, either before the grains are placed in the rotating barrel, or after they have been partially treated in the same. This result is due to a property which the grains possess of parting with the water condensed in their fiber before they part with the solvent used in their preparation. By heating the said grains to a point not sufficient to carry away the solvent, but only to evaporate

the water contained in the fibers of the grains, most of the said contained water is separated from the grains before the grains become hard. This they do when treated by boiling for the removal of the excess of solvent. Since, therefore, by this means a greater proportion of the water condensed in the fibers is removed than is the case when the grains are boiled for removal of all excess of solvent without this primary or intermediate heating, the resulting grains are harder and more dense, and thus become fitted to produce the high initial velocity to the projectile which is the object of this process. It is not material whether the grains be subjected to this gentle heat before they have been treated by boiling for removal of excess of solvent, or after they have been partially so treated, it only being necessary that they be so treated before the outer envelope of the grains becomes so hard as not to permit the passage of water through the same.

We do not confine ourselves to the temperatures mentioned, although we find them good for the purpose intended, nor do we confine ourselves to any particular method of exposing the grains to heat, using a rotating drier such as is used in sugar refineries, or spreading the grains upon sieves, or adopting any other method for the desired purpose.

After this roasting process has been carried to a greater or less extent, dependent upon the condition of the grains and the degree of consolidation required, the grain is treated in the usual manner, the solvent being boiled off in the steam barrel, or in a still, or in the open air.

We have found that better results are obtained in the use of the barrel by creating a vacuum at the steam outlet pipe, as the vapors are thereby more rapidly conveyed away, and the amount of time required for the treatment in the barrel is reduced.

The explosive force of the explosive thus produced may be tempered in the same manner as was the explosive produced by our process for which application for Letters Patent has already been made, namely, by adding from two and one-half per cent. to ten per cent. of a moderating agent, such as nitrated rosin or nitrated turpentine, *i. e.*, rosin or Venice turpentine treated with nitric acid, although we do not confine ourselves to the use of these substances, nor to the proportions mentioned. We add this moderating agent by dissolving the same in the solvent used in the granulation process, or by adding it to the finished grain, or in any other suitable manner, but it must be added in the proportion which it is to bear to the finished product as it is non-volatile at the temperatures used.

We have described the gun cotton as being diffused in water, but 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 while gun cotton has been described as the form of nitro-cellulose employed in the process, it is evident that other forms of nitro-cellulose may be used instead if desired.

We do not confine ourselves to the exact proportions mentioned, nor to the particular apparatus described, as these can be varied and changed without affecting the merits of our process.

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

1. The herein described process for 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 of the same not miscible to any great extent in the liquid used to suspend the nitro-cellulose, and solidifying the grains thus formed by subjecting the same to a heat insufficient to vaporize the solvent but sufficient to vaporize the liquid for removal of the liquid contained in the grains, and then removing the excess of solvent, substantially as described.

2. The herein described process for 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 subjecting the same to a heat insufficient to vaporize the solvent but sufficient to vaporize the liquid for removal of the liquid contained in the grains, and then to a heat sufficient to vaporize the solvent for removal of the excess of solvent, substantially as described.

3. The herein described process for 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 with removal of liquid contained in the same and excess of solvent by subjecting them to a heat ranging from 120° to 180° Fahrenheit for removal of the liquid, and then boiling them for removal of excess of solvent, substantially as described.

4. The herein described process for 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 subjecting the same to a heat insufficient to vaporize the solvent but sufficient to vaporize the liquid for removal of the liquid contained in the grains, and then to a heat sufficient to vaporize the solvent for re-

moval of the excess of solvent, the latter operation being accompanied by agitation, substantially as described.

5. The herein described process for 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 of the same not miscible to any great extent in the liquid used to suspend the nitro-cellulose, and solidifying the grains thus formed by subjecting the same to a heat insufficient to vaporize the solvent but sufficient to vaporize the liquid for removal of the liquid contained in the grains, and then rotating the grains in an atmosphere of steam for removal of the excess of solvent, substantially as described.

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

FRANCIS G. DU PONT.
PIERRE S. DU PONT

Witnesses:
HENRY J. CRIPPEN,
FRANCIS H. HOFFECKER.