(No Model.) 2 Sheets—Sheet 1.

F. G. DU PONT.

PROCESS OF MAKING SMOKELESS EXPLOSIVES.

No. 503,587. Patented Aug. 22, 1893.

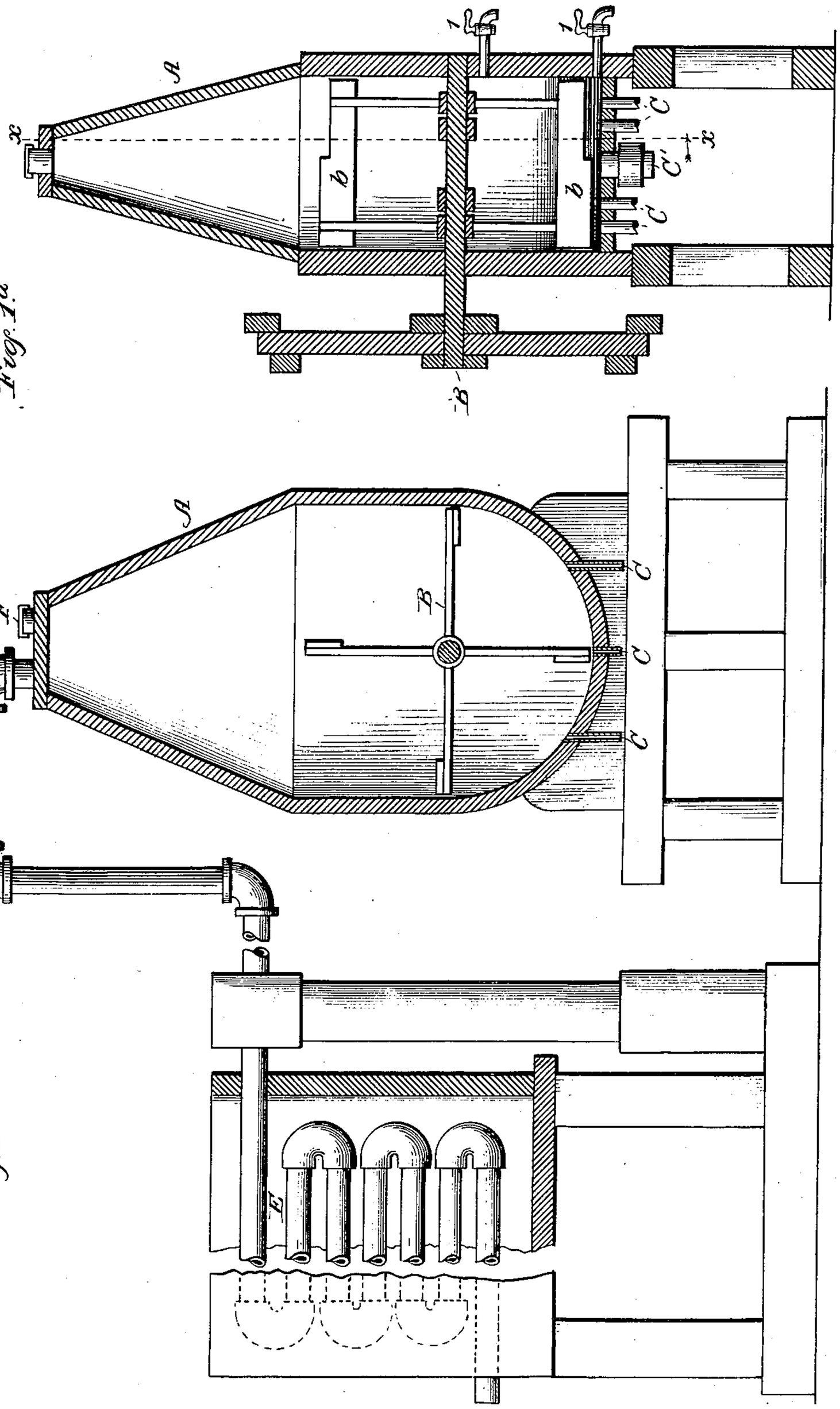

Fig. 1a.

Fig. 1.

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Fig. 2a.

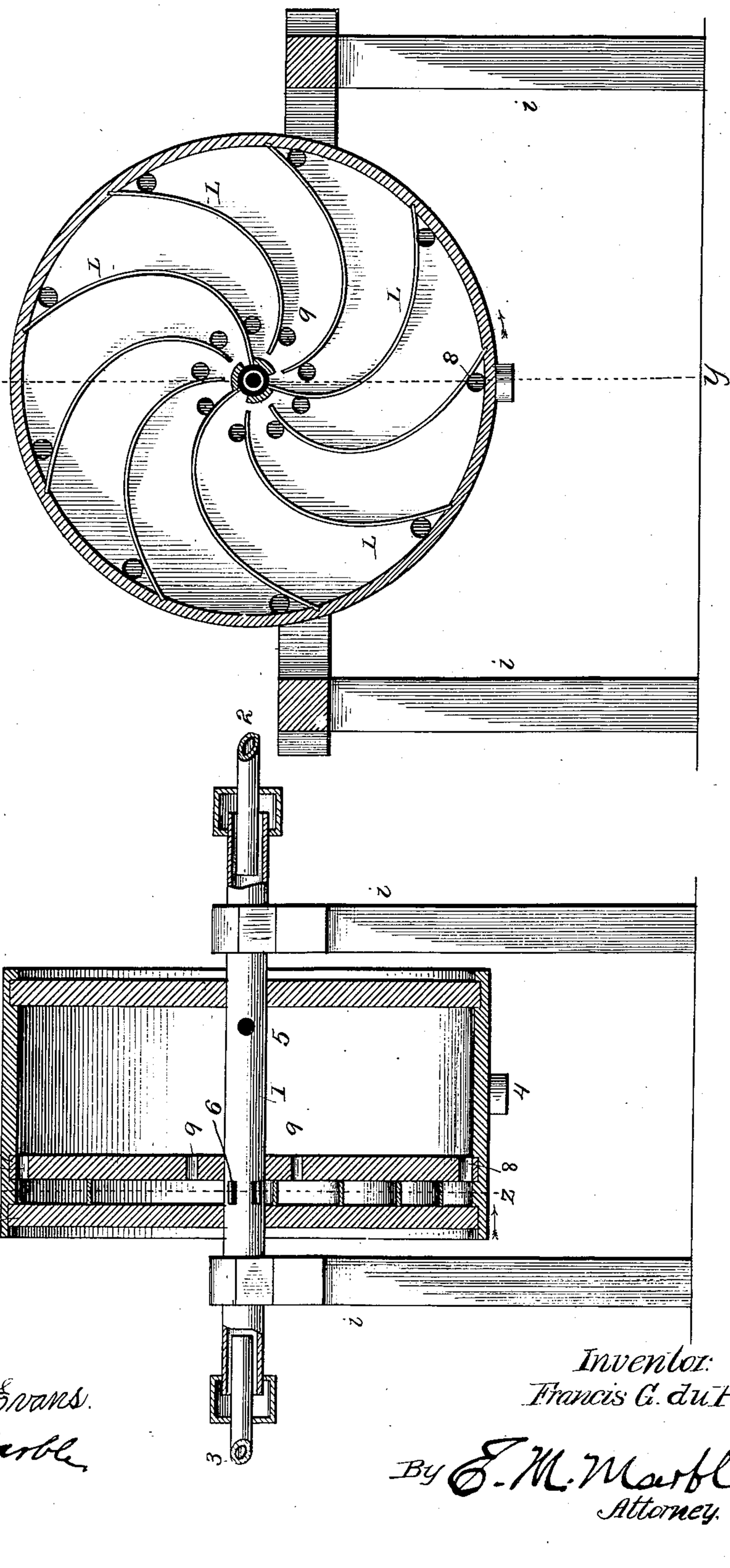

Fig. 2.

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UNITED STATES PATENT OFFICE.

FRANCIS G. DU PONT, OF WILMINGTON, DELAWARE.

PROCESS OF MAKING SMOKELESS EXPLOSIVES.

SPECIFICATION forming part of Letters Patent No. 503,587, dated August 22, 1893.

Application filed April 15, 1893. Serial No. 470,549. (No specimens.)

To all whom it may concern:

Be it known that I, FRANCIS G. DU PONT, a citizen 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 I 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.

My 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 the improved process for producing an explosive from nitro-cellulose, by agitating therewith, when suspended in a liquid, in suitable proportions, an emulsion of a solvent of the same in a suitable liquid, and subsequently hardening the grains thus produced, which will be hereinafter fully described, and particularly pointed out in the claims.

In the joint application of myself and Pierre S. du Pont for Letters Patent for improvements in explosives, filed December 21, 1892, Serial No. 455,901, a process for producing a smokeless explosive was described, the distinguishing feature of which consisted in the granulation of the nitro-cellulose by agitating therewith, when suspended in a suitable fluid, a solvent of the same in proper proportions. Basing the process on the tendency which was found to be inherent in the solvents of nitro-cellulose, and especially the nitro-derivatives of the aromatic group, when mixed with a fluid in which finely-divided nitro-cellulose is suspended, to seize the nitro-cellulose and leave the fluid clear, forming in the fluid a more or less coherent mass, it was discovered that when the solvent is added in proper proportions, (and the proportion of three parts by weight of the solvent to one of the nitro-cellulose was found to be 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 put upon 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 can be still further diminished by the addition of a substance to moderate its action. For this latter purpose nitrated rosin and nitrated turpentine, *i. e.*, rosin or Venice turpentine treated with nitric acid, were found to be especially suited, and they were added in the manner and proportion stated in said application.

The essential feature of the above process, and that which determines the character of the explosive produced, is the granulation of the nitro-cellulose. This granulation is effected, in the process described in said application, by pouring nitro-benzole, or other solvent of nitro-cellulose, into a churn in which a quantity of finely divided nitro-cellulose is suspended in a suitable liquid, said nitro-cellulose having been uniformly distributed through said liquid by the action of the churn. After the solvent has been added to the mixture in the churn, the agitation of the contents of the same is continued, until the nitro-cellulose has been deposited in the bottom of the churn in the form of granules, which are somewhat pulpy, as they contain in the fiber of the nitro-cellulose considerable water, and as there is an excess of solvent in connection with the same.

It is of the utmost importance, in determining the characteristics of the grains produced, that the granulation be uniform, and that all of the nitro-cellulose be precipitated, as otherwise there is a loss of material, and the grains formed are uneven in structure.

I have discovered that complete and uniform granulation of the nitro-cellulose may be effected by emulsifying the nitro-benzole, or other solvent of nitro-cellulose used, before adding the said solvent to the mixture in the churn, with a suitable liquid. Water may be used as such liquid, and may either be pure, or may have in solution in it any of the

usual substances which are used to produce emulsions, such as soap, or carbonate of soda liquor may be used, or any other suitable liquid. I have found by experiment that by the use of the emulsion thus produced a more uniform granular condition is brought about, and also a more uniform and perfect precipitation of the nitro-cellulose obtained, than when the unemulsified solvent is used.

In the drawings accompanying and forming a part of this application, I have represented the apparatus which I use to carry out my 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 pipes forming the axis of the barrel by which the said water is conveyed away.

Before proceeding with a description of my process, I 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 *l l* 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 steam and water, the former 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 this 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 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 apertures 9 in the bulkhead K, from whence it passes through the axis I and the pipe 3. Water of condensation, as fast as formed, passes through the outer series of apertures in the bulkhead K at the lower part of revolution of the barrel into the space between said bulkhead 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 my process, I 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, I sometimes dissolve in it chloride of calcium, common salt, saltpeter, or other salt, as the characteristics of the grains produced can thus be modified in many ways. I then add to the water about six kilograms of nitro-cellulose, beating it up with the water in the churn until a thorough mixture has taken place. In another churn similar to that shown in Figs. 1 and 1ª, or in any other suitable machine, I prepare an emulsion of the solvent of the nitro-cellulose which I desire to use with water, or with other emulsifying liquid. When water is used, I add it to the solvent, such as nitro-benzole, in about the proportions of three times the weight of the nitro-benzole. When carbonate of soda liquor is used, I add one pound of the carbonate of soda to a gallon of water, and use about the same weight of liquor as in the case of pure water. When soap is used to aid emulsion, I add it in the proportion of about one ounce to twenty-five pounds of nitro-benzole, and use about the same weight of water as before. I do not restrict myself to the use of the emulsifying agents above mentioned, stating them merely as examples, nor to the proportions here stated of adding the same to the solvent of the nitro-cellulose, or those in which the various ingredients are added to water. The emulsion thus produced is added to the mixture in churn A, a sufficient quantity of it being added so that the proportion of the nitro-benzole, or other solvent used, to the nitro-cellulose is by weight about three to one, and then the agitation of the contents of the churn is continued until the granulation of the nitro-cellulose is complete. This granulation takes place on account of the tendency of the nitro-benzole to gather together the particles of nitro-cellulose into granules, and form a coating around the same, the coating when formed preventing the grains from becoming attached to each other, or otherwise forming one mass. The action of the solvent when emulsified in the

manner above stated is much more rapid and complete than when such solvent is added in an unemulsified form, and results in the more perfect granulation hereinbefore described. The grains thus formed, when removed from the churn, may be treated with steam to remove the excess of solvent and the water contained in the grains, and then be used with success for some explosive uses, but a more perfect grain is produced by treatment of the grains produced in the churn in the rotating barrel shown in Figs. 2 and 2ª. This treatment proceeds either in the manner described in the joint application of myself and Pierre S. du Pont above referred to, by rotating the barrel for a greater or less time without the admission of steam, until the grains are compacted and rounded, then filling the barrel partially with water, and rotating it with admission of steam in such a direction as to prevent the water from being carried away, and then changing the direction of rotation and continuing the flow of steam until the solvent is entirely removed; or in the manner described in the joint application of myself and Pierre S. du Pont filed March 17, 1893, Serial No. 466,488, in which the grains are, at some time before their final rounding and compacting treated with heat, but of a degree below that of the point of vaporization of the solvent used in the production of the grains, to remove more effectually the water condensed in the fibers of the grains. The latter method of treatment is preferred, as resulting in a more finished product. The explosive force of the grains thus produced may be tempered in explosive force to any degree required by the addition of a suitable moderating agent, in the manner described in the joint applications before referred to.

While I have described the nitro-cellulose as being diffused in water, it is evident that any other fluid which will cause a better, or as good a distribution of the nitro-cellulose in the churn may be used instead.

I do not confine myself to the exact proportions used, nor to the particular apparatus described, as these can be varied and changed without affecting the merits of this invention.

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

1. 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 an emulsion of a solvent of the nitro-cellulose, which is not miscible to any great extent in the liquid used to suspend the same, in a suitable liquid in proper proportions, and solidifying the grains thus formed, substantially as described.

2. 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 an emulsion of a solvent of the same, which is not miscible to any great extent in the liquid used to suspend the nitro-cellulose, with water in proper proportions, and solidifying the grains thus formed, 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 an emulsion of nitro-benzole, which is not miscible to any great extent in the liquid used to suspend the nitro-cellulose, with a suitable liquid in proper proportions, and solidifying the grains thus formed, substantially as described.

In testimony whereof I affix my signature in presence of two witnesses.

FRANCIS G. DU PONT.

Witnesses:

FRANCIS I. DU PONT,

ELIZA SIMONS.