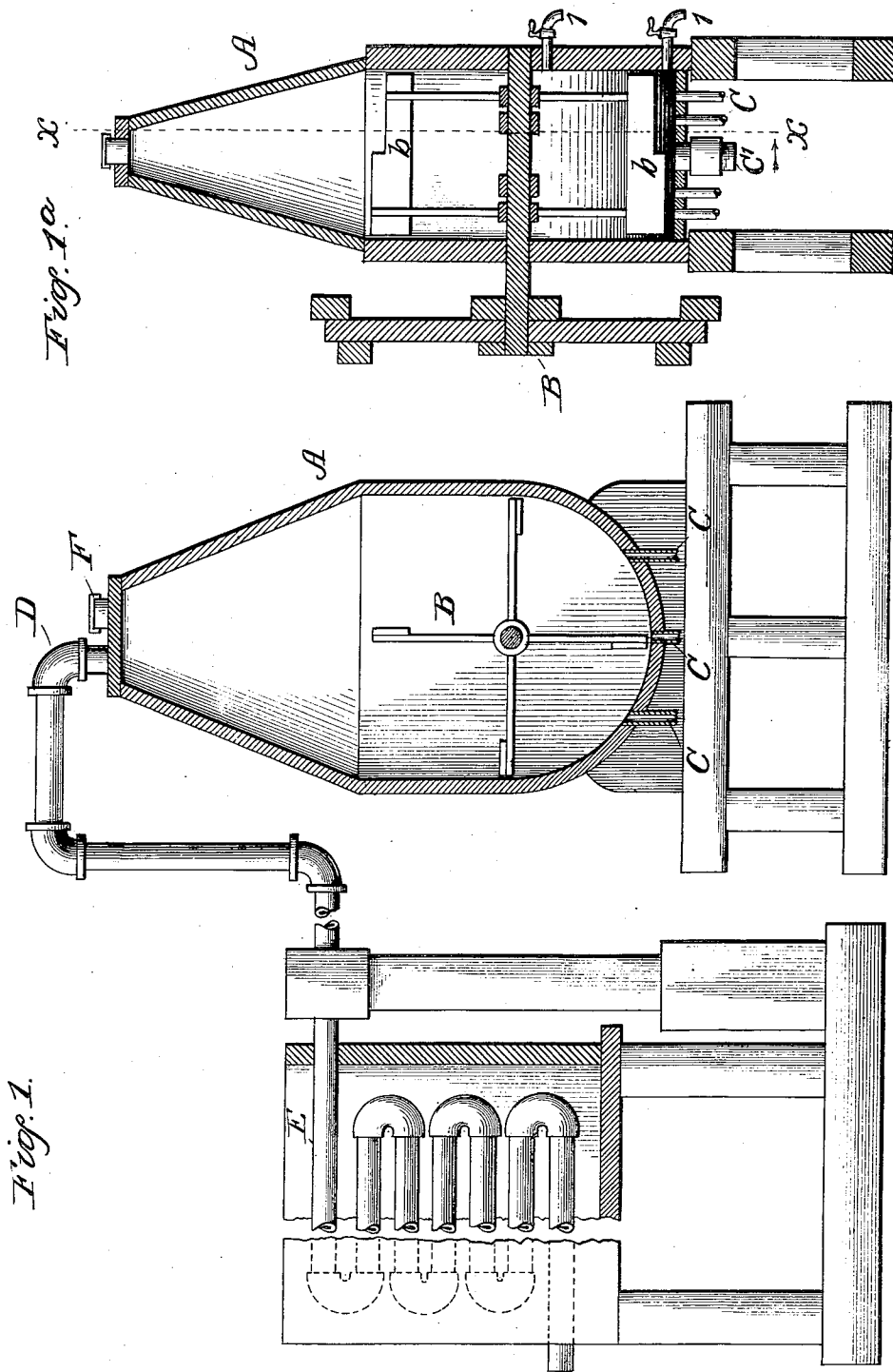


F. G. DU PONT.
MANUFACTURE OF SMOKELESS POWDER.

No. 519,702.

Patented May 15, 1894.



Witnesses.
Victor J. Evans.
L. M. Marsh

Inventor.
Francis G. du Pont.
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Attorney.

(No Model.)

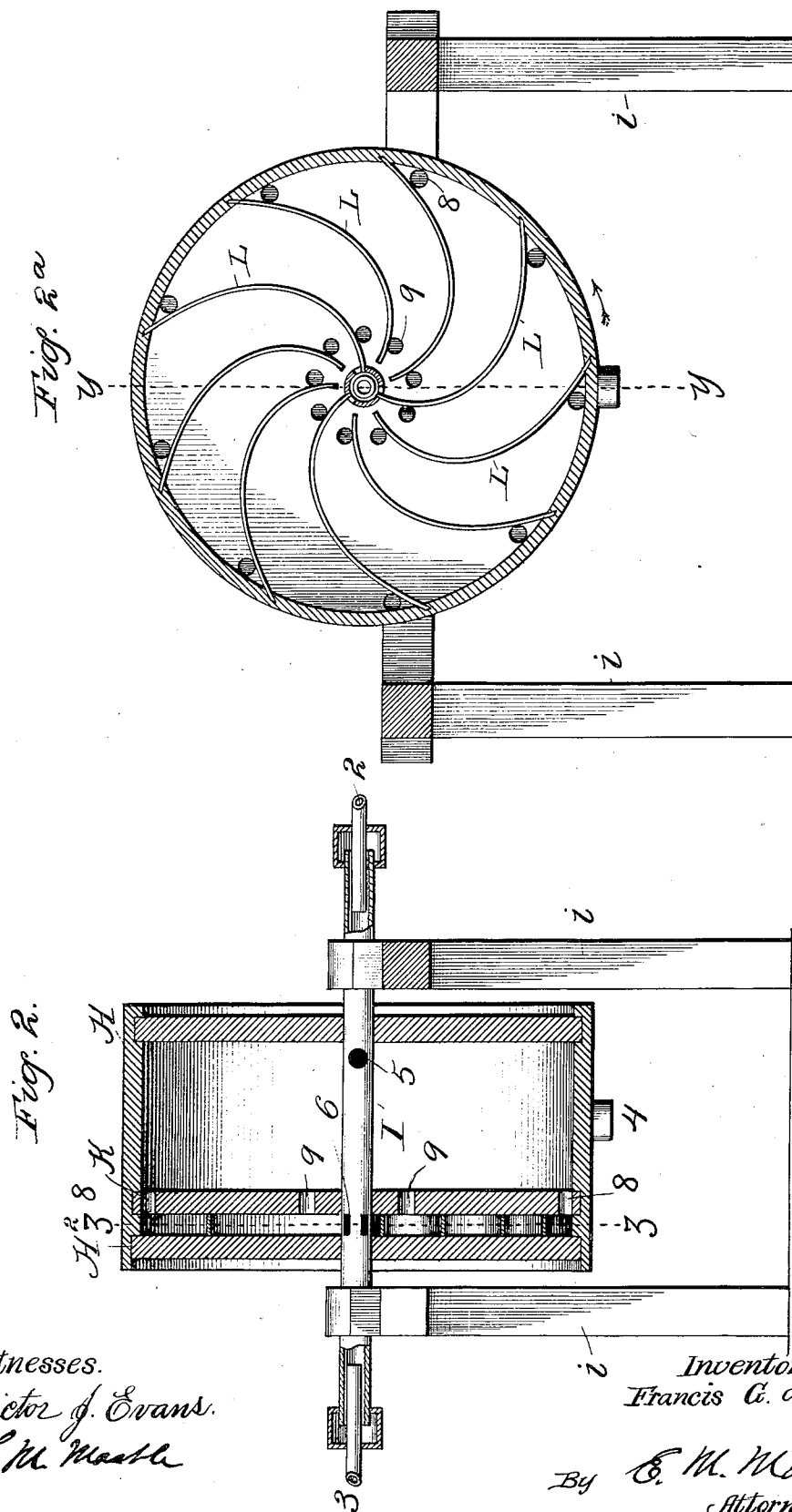
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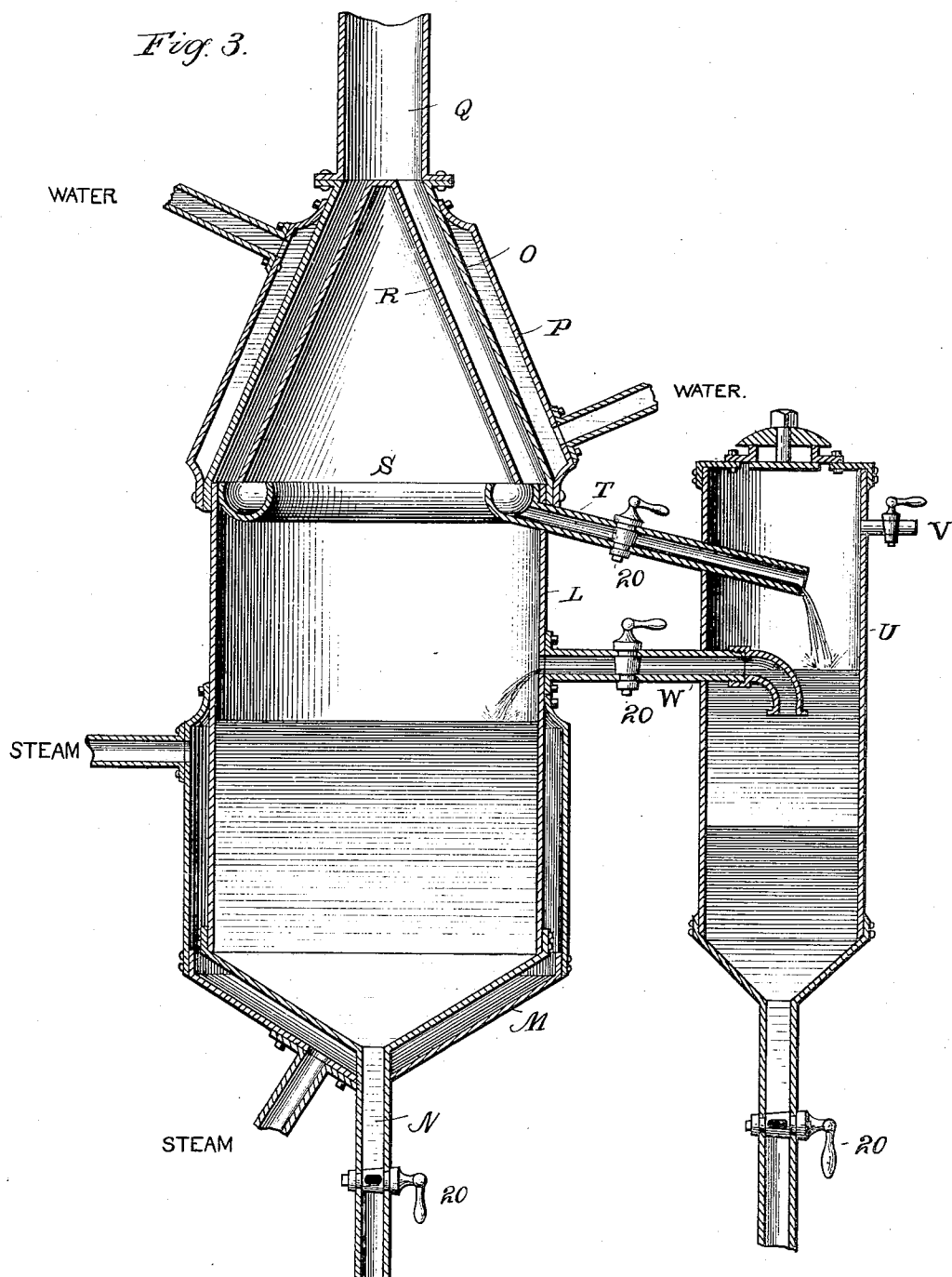
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UNITED STATES PATENT OFFICE.

FRANCIS G. DU PONT, OF WILMINGTON, DELAWARE.

MANUFACTURE OF SMOKELESS POWDER.

SPECIFICATION forming part of Letters Patent No. 519,702, dated May 15, 1894.

Application filed August 5, 1893. Serial No. 482,448. (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 for which it may be suited, and it consists of an improved process of producing an explosive from nitro-cellulose by agitating therewith in suitable proportions, when suspended in a liquid not a solvent of the nitro-cellulose, an emulsion of a suitable liquid with a mixture of a suitable solvent of nitro-cellulose and a volatile oil, such as hydro-carbon oil, in proper proportion, and subsequently hardening the grains thus produced, which will be herein after 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 of producing a smokeless explosive was described, the distinguishing feature of which consisted in the granulation of the nitro-cellulose by the agitation therewith, when suspended in a suitable liquid not a solvent of the nitro-cellulose, of 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 which explosive action can be diminished by the addition of a substance to moderate its action. For this latter purpose nitrated rosin and nitrated turpentine, *i. e.*, rosin and 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 into 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 considerable water, and as there is an excess of solvent in connection with the same.

My present invention aims to provide means for enabling a more perfect granulation to take place than was possible by the process described in the application above referred to, for enabling the density of the grains formed to be controlled as desired, and for enabling the grains formed to withstand a far greater degree of heat than those formed by said process could without fusing together. I have discovered that if a volatile oil, and preferably a hydro-carbon oil, of which benzine may be mentioned as a type, be mixed with the solvent in proper proportions, and

then an emulsion of this mixture with a suitable emulsifying liquid, such as water be produced and added to the contents of the churn in the manner above described, it is possible to obtain a much better grain than has heretofore been obtainable, and also to regulate its density with much greater precision than has heretofore been possible. Furthermore, while the grain produced by the original process referred to will not withstand the action of 212° Fahrenheit heat without fusing into a solid mass, the grain produced in the manner just above stated has the remarkable property of being able to withstand the application of a much greater degree of heat without damage. The addition of the oil to the solvent appears to cause a repelling action among the grains, which entirely prevents the heat acting upon them, and making them stick together.

The apparatus employed in carrying out my process is fully represented in the drawings accompanying and forming a part of this application, in which the same reference letters and figures refer to the same or corresponding parts, and in which—

Figure 1, taken on the line *xx* Fig. 1^a, looking in the direction of the arrow, is a view of the churn in which the mixture of the nitro-cellulose with the solvent takes place. Fig. 1^a is a sectional view of the same. Fig. 2 is a sectional view of the rotating barrel in which the grain formed in the churn shown in Figs. 1 and 1^a is rounded and hardened. Fig. 2^a is a sectional view taken on the plane of the cycloidal chutes shown in Fig. 2. It shows the double series of apertures in the inner partition of the barrel, and also the form of the series of cycloidal chutes which conduct the water of condensation, received through the outer series of apertures in the partition, to the apertures in the central pipe forming the axis of the barrel, by which the said water is conveyed away. Fig. 3 is a view of the still in which the oil is removed from the grain.

Before proceeding with a description of my process, I shall describe the apparatus used in carrying it out.

Referring to the drawings, and especially to Figs. 1 and 1^a, A represents a churn, in which, on a shaft B, are mounted 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 the rotation of the blades. 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 it conducts may be condensed. The pipes C are bent upward or otherwise formed so as to prevent the escape of the contents of the churn through the same. 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 ascertained.

In Figs. 2 and 2^a the rotating barrel is represented in which the grains of explosive produced in the churn just described are hardened and rounded. 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 partition K, which is placed in close proximity to the head H² of the barrel. In this inner partition 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 partition K and head H² is 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 axis I and the pipe 3. Water of condensation, as fast as formed, passes through the outer series of apertures in the partition K at the lower part of the revolution of the barrel into the space between said partition and head H², and is conducted by the cycloidal chutes toward the axis of the barrel, then passing out through the apertures 6 formed at this point.

I have made a separate application for Letters Patent for the form of still shown in Fig. 3, said application being dated April 15, 1893, Serial No. 470,347, but I will briefly describe the same, so that its construction and operation may be understood. L represents the still, of any convenient size and construction, around the lower portion of which is placed an encircling steam jacket M, provided with steam inlet and outlet pipes. The pipe N, at the apex of the conical bottom of the still, serves for the introduction and removal of material. The still is provided with a conical top O, around which is placed the jacket P, which is provided with water inlet and outlet pipes, and between which, and the top O, a stream of cold water is constantly flowing when the still is in operation. The outlet Q is for the conduction of vapors escaping from the still to any suitable condenser, which condenser is so arranged that the fall of the pipes leading to the same is backward into the still, so as to drain condensed products into the still head. Inside of the top O, and of a corresponding shape, is suspended the inner shell or cone R, the function of which is to prevent the distillate coming from the condenser from falling into the still, and to determine its delivery, as well as that of all the vapors condensed in the still head, into the annular trough S, which surrounds the interior of the still at the base of the still head. The pipe T affords an exit from this

trough into the outer vessel U, which answers as a reservoir for the distillate. This reservoir has a conical bottom, and a pipe at the apex of the same, by means of which the reservoir may be emptied when desired. An outlet pipe V is also provided on the side of the reservoir, from which may be drawn from time to time the oil which has collected. Besides the connection afforded by pipe T, the reservoir is also connected with the still by the siphon pipe W, the lengths of the branches of which are so arranged that the water which collects in the bottom of the reservoir U will be automatically returned to the still L. Stop cocks 20 are provided to control the passage through the pipes leading to and from the still and reservoir.

Proceeding now with my process, I place in the churn A about ninety liters of water, 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 according to the properties of the ingredient added. The proportion in which these ingredients are added depends upon which one is used. In the case of saltpeter, about the half-saturated solution will answer very well, or even one of greater strength, say a saturated solution of the temperature of 60° Fahrenheit. 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 1^a, I place the quantity of solvent which I desire to use. I preferably use for such solvent some one of the nitro-compounds of the aromatic group, such as nitro-benzole, nitro-toluol, or nitro-xytol. For the sake of the present description, I will confine myself to nitro-benzole. I add to the nitro-benzole, the amount of which is determined by the working proportion of three parts of the nitro-benzole by weight to one of nitro-cellulose, a volatile oil, such as benzine, to the extent of from fifteen per cent. to twenty per cent. of the nitro-benzole used. By varying the amount of benzine added I can with accuracy and precision obtain any desired degree of density in the grains formed by my process, the grains being less dense in proportion as the percentage of oil is increased as other examples of volatile oil, may be added benzole, toluol, and xytol. I then prepare an emulsion of this mixture with a suitable emulsifying liquid. When water is used as such emulsifying liquid, I add it in about the proportion of three times the weight of the nitro-benzole. When a carbonate of soda liquor is used, I add one pound of the carbonate of soda to one gallon of water, and use about the same weight of liquid 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 liquids above mentioned, nor to the proportions there stated of adding the same to the nitro-benzole, or those in which the various ingredients are added to the water. The emulsion thus produced is added to the contents of the churn, and the agitation in the churn is continued until a perfect and complete granulation of the nitro-cellulose has taken place. The addition of the benzine to the nitro-benzole seems to have a diluting action upon the same which prevents it from acting so suddenly upon the nitro-cellulose, thus enabling a larger and a less dense grain, as well as a much more uniform grain, to be formed than is possible when nitro-benzole is used alone. The grain thus formed is quite complete, and may be used very well without the glazing in the rotating barrel which is next to be described. The contents of the churn are now placed in the rotating barrel shown in Figs. 2 and 2^a. The treatment now 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 the admission of steam in such a direction as to prevent the water from being carried away, and then changing the direction of the rotation and continuing the flow of steam; or in the manner described in the joint application of myself and Pierre S. du Pont, filed March 17, 1893, Serial No. 486,488, in which the grains are, at some time before the final rounding and compacting, treated with heat of a degree lower than that required to vaporize the solvent used to remove more effectually the water contained in the grains. The latter method of treatment is preferred, as resulting in a more finished product. I continue this rotation until the grain is completely formed, but as it would take a considerable length of time to complete the removal of the excess of solvent from the grain by simple rotation in an atmosphere of steam, I place the contents of the barrel, after the grain has been completely formed, rounded and hardened, into the still shown in Fig. 3. When it was attempted to drive off the excess of solvent from the grains produced by the process described in the joint application of myself and Pierre S. du Pont, Serial No. 455,901, to which reference has been made, by distillation, it was found to be necessary to use a vacuum in connection with the still to carry off the vapors as fast as formed, and to decrease the temperatures of volatilization of the contents of the still, because otherwise, if such grains were heated to 212° Fahrenheit, they would fuse into a solid mass. The addition of benzine to the solvent used has the peculiar property of enabling the grains to withstand the action of heat without fus-

ing together. A repellent action is produced between the grains which prevents them from sticking together. In the present case, therefore, it is not necessary to use a vacuum in connection with the exit pipe of the still, but by circulation of steam through the steam jacket surrounding the lower portion of the still, distillation at once commences, the benzine passing off with the nitro-benzole, and being collected with the same in the reservoir U. The pipe W is so arranged as to automatically return only the water to the main still, the nitro-benzole and the benzine being drawn off from the reservoir. As all of the benzine originally added to the nitro-benzole is thus recovered with it, it is not necessary to add a new portion of benzine to the nitro-benzole when it is reused. The grains thus produced can of course be modified to any desired extent in their explosive action by the addition of suitable moderating agents. Of such, nitro-rosin and nitro-turpentine, *i. e.*, rosin or Venice turpentine treated with nitric acid, may be mentioned, the proportion in which they are added varying from two and one-half per cent. to ten per cent. of the solvent used to granulate the nitro-cellulose. The moderating agent may be added either by allowing the finished grain to absorb a suitable quantity, or by addition to the contents of the churn while the granulation process is being carried on.

I do not confine myself to the use of the apparatus herein described, nor the particular solvents and oils mentioned nor to the exact proportions of the ingredients herein stated; but

What I do claim, 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 mixture, in suitable proportions, of a suitable solvent not miscible to any great extent in the liquid used to suspend the nitro-cellulose and a volatile oil, with a liquid calculated to aid emulsion 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 mixture, in

suitable proportions, of a suitable solvent not miscible to any great extent in the liquid used to suspend the nitro-cellulose and a hydro-carbon oil, with a liquid calculated to aid emulsion 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 a mixture, in suitable proportions, of a suitable solvent not miscible to any great extent in the liquid used to suspend the nitro-cellulose and a volatile oil, with a liquid calculated to aid emulsion in proper proportions, hardening and rounding the grains by agitation, and removing the excess of solvent and the oil by distillation, 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 proportions, an emulsion of a mixture, in proper proportions, of a suitable solvent not miscible to any great extent in the liquid used to suspend the nitro-cellulose and a volatile oil, with a liquid calculated to aid emulsion in proper proportions, hardening and rounding the grains by rotation, and removing the excess of solvent and the oil by distillation, 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, an emulsion of a mixture, in suitable proportions, of a suitable solvent not miscible to any great extent in the liquid used to suspend the nitro-cellulose and a volatile oil, with a liquid calculated to aid emulsion in proper proportions, hardening and rounding the grains by rotation in an atmosphere of steam, and removing the excess of solvent and the oil by distillation, substantially as described.

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

FRANCIS G. DU PONT.

Witnesses:

THOS. J. BOWEN, Jr.,
HENRY J. CRIPPEN.