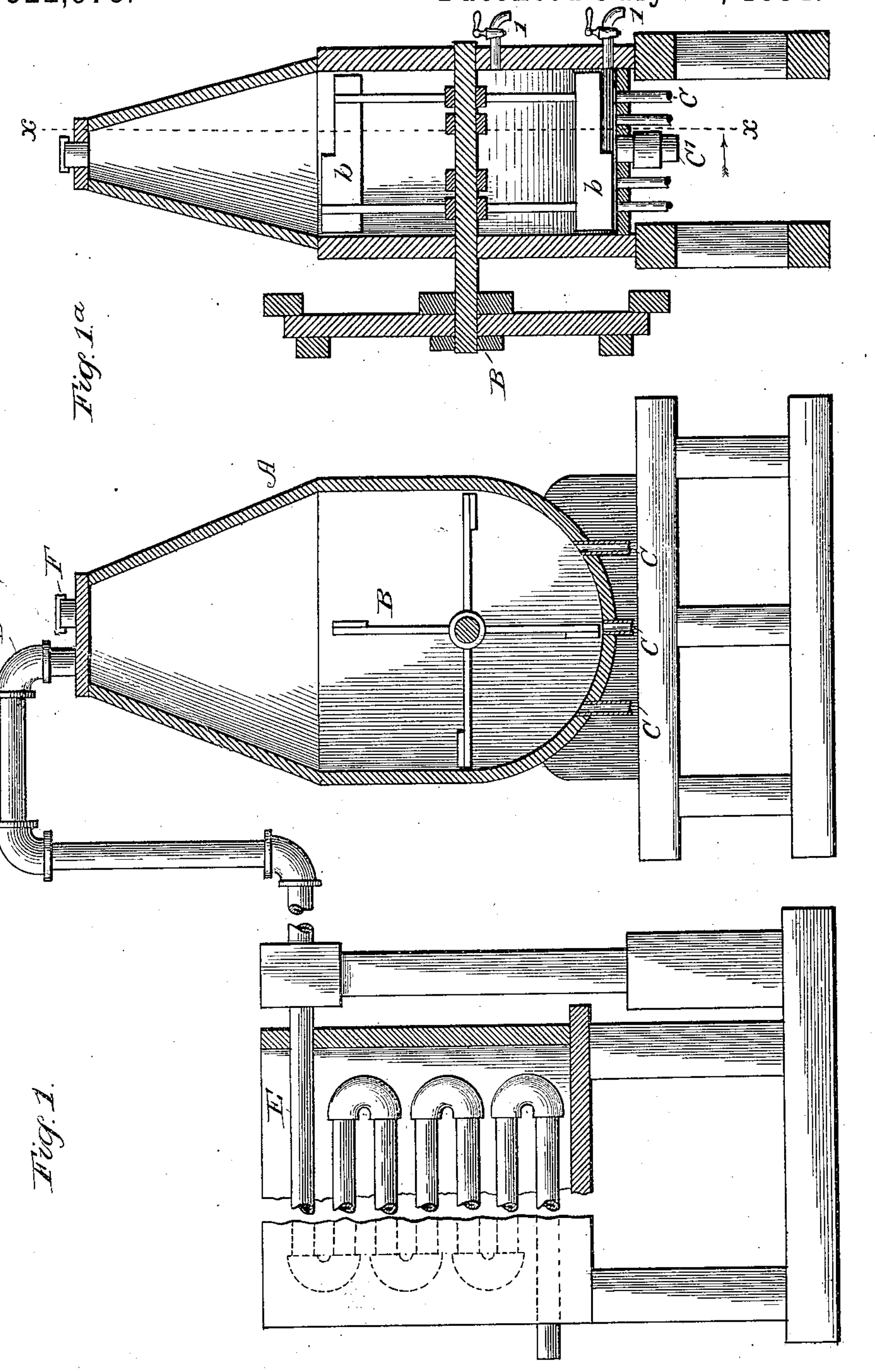
(No Model.) 3 Sheets—Sheet 1.

F. G. DU PONT.

SMOKELESS EXPLOSIVE.

No. 522,978. Patented July 17, 1894.



Witnesses.
Victor J. Evans.
L. M. Marble.

Inventor:
Francis G. du Pont.

By. E. M. Marble
Attorney.

(No Model.) 3 Sheets—Sheet 2.

F. G. DU PONT.

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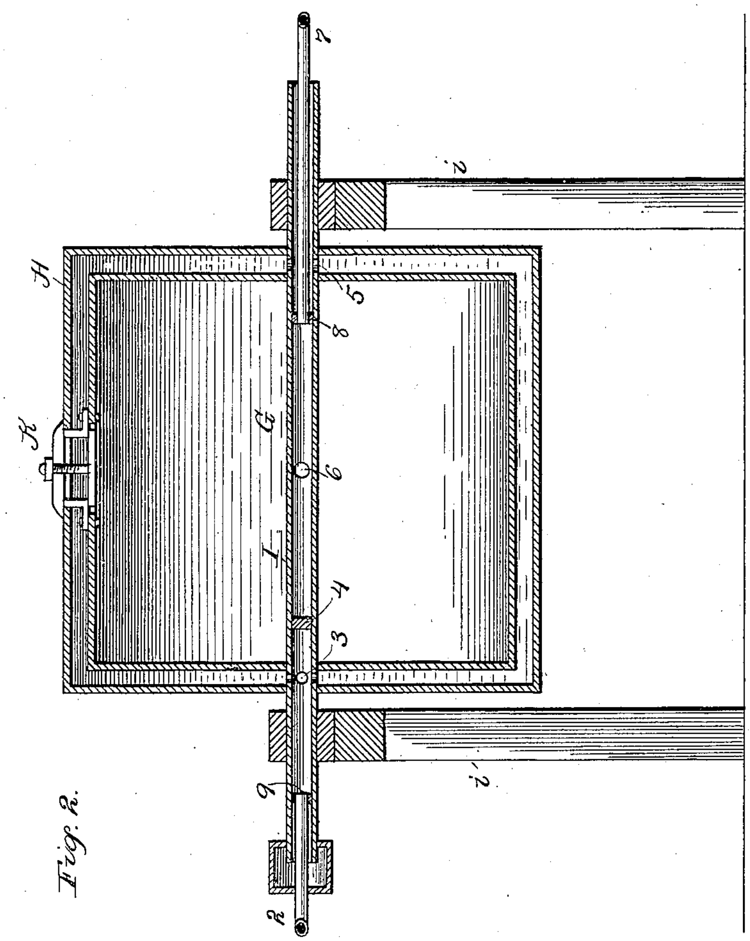

Fig. 2.

Witnesses.
Victor J. Evans.
L. M. Marble

Inventor.
Francis G. du Pont.

By *E. M. Marble*
Attorney.

(No Model.)

3 Sheets—Sheet 3.

F. G. DU PONT.

SMOKELESS EXPLOSIVE.

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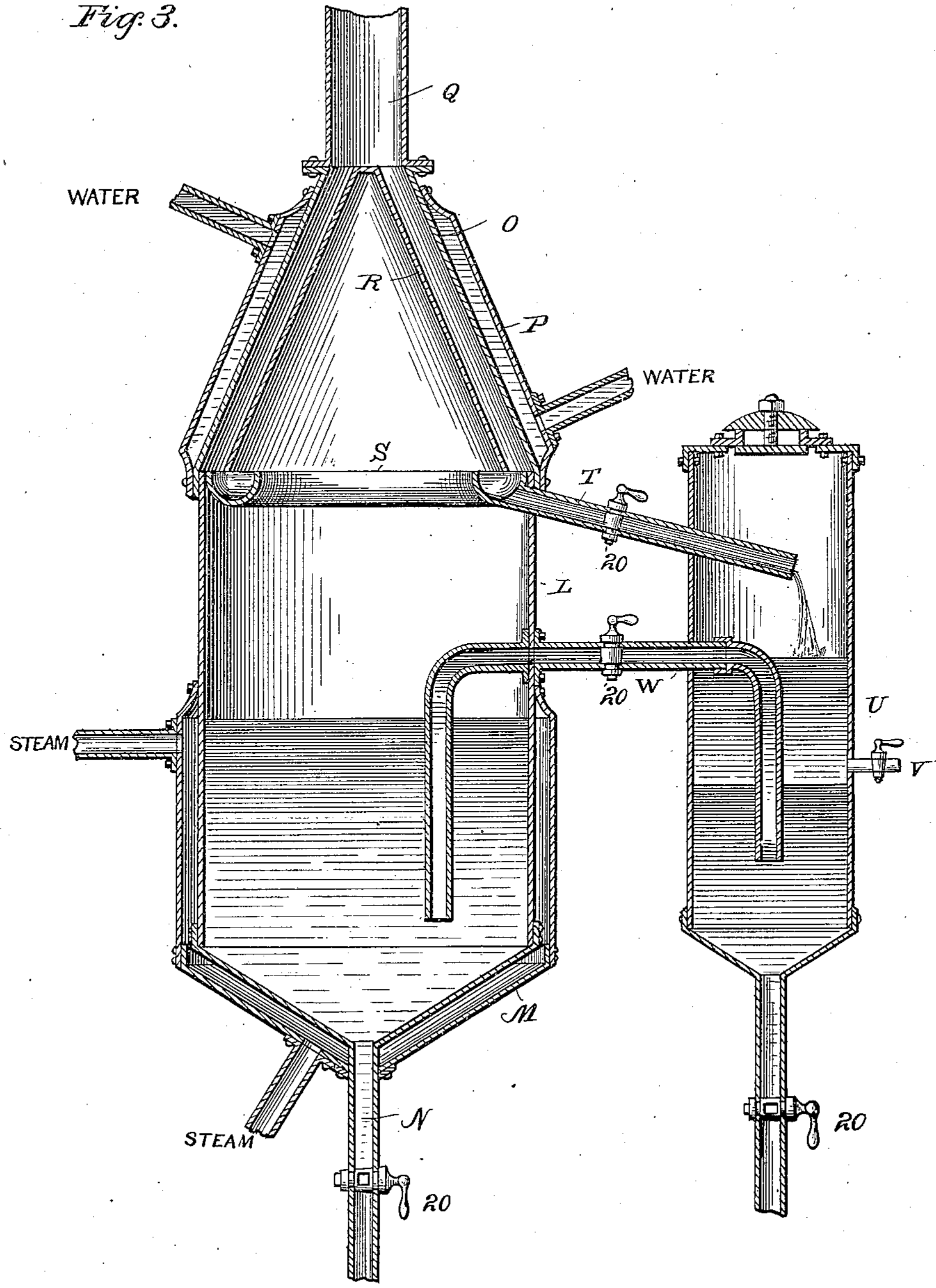
Fig. 3.



Witnesses.
Victor J. Evans
L. M. Marble

Inventor
Francis G. du Pont.
By. *E. M. Marble*
Attorney.

UNITED STATES PATENT OFFICE.

FRANCIS G. DU PONT, OF WILMINGTON, DELAWARE.

SMOKELESS EXPLOSIVE.

SPECIFICATION forming part of Letters Patent No. 522,978, dated July 17, 1894.

Application filed September 26, 1893. Serial No. 486,519. (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 smokeless explosives to be used in rifles, shot guns, and other arms, as well as for other uses to which smokeless powder may be put; and it consists in an improved process for producing a smokeless explosive from nitro-cellulose, which consists in mixing in suitable proportions with the nitro-cellulose, when it is suspended in an oil, such as hydro-carbon oil, a solvent of the nitro-cellulose which, although it may be miscible with the suspending oil, is still capable of exerting a solvent action upon the nitro-cellulose, such as acetone and some of the high boiling point ketones, as dipropyl ketone, and subsequently hardening the grains thus produced, and 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, patented August 22, 1993, No. 503,583, a process for producing a smokeless powder was described, which consists in granulating the nitro-cellulose by agitating therewith, when suspended in a liquid not a solvent of the same, a solvent of the nitro-cellulose not soluble to any great extent in the liquid used to suspend the nitro-cellulose, and subsequently hardening the grains thus produced. In said application, while it was stated that any suitable solvent might be used to form the granulation thus produced, which was not soluble to any great extent in the liquid used to suspend the nitro-cellulose, attention was especially called to the nitro-compounds of the aromatic group, of which nitro-benzole was mentioned as a type, as being suitable solvents; and while it was stated that any suitable liquid, not a solvent of the nitro-cellulose, might be used to suspend the same, the granulation was described as taking place when the nitro-cellulose was suspended in water.

I have discovered that a parallel result may be accomplished by suspending the nitro-cellulose in liquids of the nature of oils, such as hydro-carbon oils, of which kerosene, benzine, and gasoline may be mentioned as especially suited for the purpose, and by forming the granulation of the nitro-cellulose by agitating with the same, thus suspended in an oil, a solvent of the nitro-cellulose which, though it may be miscible with the suspending oil, is still capable of exerting a solvent action upon the nitro-cellulose. It is thus not necessary that the solvent employed to granulate the nitro-cellulose be insoluble to a greater or less extent in the liquid used to suspend the same, but only that the relative attraction of the solvent for the oil and for the nitro-cellulose be such that there is a stronger tendency for nitro-cellulose, thrown into a solution of the solvent and the oil, to take the solvent, than for the oil to hold it. Of such solvents, acetone and some of the high boiling point ketones may be mentioned as examples.

Nitro-benzole cannot be conveniently used when an oil is used to suspend the nitro-cellulose, for the reason that it is so soluble in oils that, unless added in great excess, it will not leave the oil in sufficient quantity to form the granulation with the nitro-cellulose. In any case, the grains formed are imperfect. Nor can such a solvent of nitro-cellulose as acetone be used when the nitro-cellulose is suspended in water, because acetone is so soluble in water that it will not leave the water to cause the required granulation. But when the nitro-cellulose is suspended in an oil, and a solvent of the same is used which, although it may be completely miscible with the oil, will yet leave the oil to act upon the nitro-cellulose, a perfect granulation results. The grains thus formed are of the same general character as those produced by the process described in the joint application of myself and Pierre S. du Pont, to which reference has been made, that is, of uniform size, and hard and round but are somewhat denser, and more compact. The increased advantage gained by the use of an oil, such as kerosene oil, in which to suspend the nitro-cellulose, lies in the fact that thereby a more even and uniform distribution of the particles of nitro-cellulose throughout the liquid is obtained, and the granulation therefore takes place under more advantageous circumstances.

The apparatus employed in carrying out my process is fully represented in the drawings accompanying and forming a part of this application, in which—

Figure 1, taken on the line xx Fig. 1ª, 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. 1ª 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 1ª is rounded and hardened. 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 1ª, 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 admisssion 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 Fig. 2 the rotating barrel is represented in which the hardening of the grains produced by the granulation in the churn shown in Figs. 1 and 1ª is carried on. G is the rotating barrel, and is rotatively supported by the central hollow axis I, journaled in the supports i i. The barrel G is surrounded by a jacket H, also mounted on the axis I, and connected with the barrel G so as to revolve with it. Between the jacket and the barrel, a supply of water is kept constantly flowing, while the grains of explosive are being hardened. The water, which is heated to a suitable temperature, is introduced through the supply pipe 2, leading into one end of the central axis I, and passes into the space between the jacket and the barrel through the openings 3 in said axis, its flow through the axis being interrupted by the partition 4 formed in the same. After circulating through the jacket, the water passes out through the openings 5, formed at the other end of the axis, into the axis I, and is discharged from this end of the axis into a suitable receptacle or drain. The portion of the axis passing through the interior of the barrel G is perforated at 6, so as to allow for the escape of vapors formed in said barrel, and a pipe 7 is introduced into the water-delivery end of the axis through which the vapors can be conducted away. A stopper 8 prevents any water from entering the pipe at this end. The water inlet pipe 2 is provided with a stuffing box or gland 9, which permits the axis to revolve around it without leakage, and the stopper 8 is also so constructed as to permit the axis I to revolve around the pipe 7 without permitting any escape of vapors. An opening K is provided for the introduction and removal of material.

I have made 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 a 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.

Referring now to my process, I have found that in order to obtain a perfect granulation, it is necessary as a preliminary step to thoroughly dry the nitro-cellulose, before suspending the same in oil. The form of nitro-cellulose which I preferably use is that known as tri-nitro-cellulose or gun cotton. Gun cotton, as usually prepared, contains a small percentage of water, as dry gun cotton is exceedingly dangerous to handle and store. As in my process, however, I suspend the gun cotton in oil, it is necessary, on account of the refusal of oil and water to mix, to remove this water, in order to obtain an even distribution of the particles of the gun cotton throughout the oil. I dry the gun cotton after the manner described in my application for Letters Patent for an improved process for drying nitro-cellulose, filed June 15, 1893, Serial No. 477,667, by placing the wet gun cotton in a still filled with an oil, such as kerosene oil, and then distilling off the oil and the water together. The distillation may be performed in such a still as that shown in Fig. 3 of the drawings accompanying this application, the action of which will be fully explained when the step of my present process is reached in which it is used. In this manner, all of the water is removed from the gun cotton, and the same is obtained perfectly dry, although impregnated with the oil in which it has been placed. The presence of this oil is, however, not objectionable in my process.

Proceeding now with my process, I fill the churn A to the desired height with oil, and then add thereto nitro-cellulose in the proportion of ten pounds of dry nitro-cellulose to fifty pints of oil. For such oil I preferably use kerosene oil,—free from any heavy oils,—which distills at about 212° Fahrenheit, and which maintains its boiling point pretty constant at that figure. Although the oil may contain lighter products, it must not contain non-volatile matter, as in the latter part of my process it is to be completely removed from the grains formed. The nitro-cellulose is suspended in the oil by the rotation of the blades of the churn, and, when fully suspended, acetone is run into the churn in the proper proportion, and the agitation continued until granulation takes place from the action of the acetone upon the nitro-cellulose. The proper proportion in which to add the acetone depends somewhat upon the grade of oil used. When a light oil is used, it should be added in the proportion of thirty pints of acetone to ten pounds of nitro-cellulose, the latter having been added to the amount of kerosene hereinbefore mentioned. When, however, a heavier oil is used, the same proportion of oil and nitro-cellulose will be required, but only about twenty-five pints of acetone. Of course these proportions can be varied, but those given are well suited for the purpose, and indicate with sufficient definiteness the variation of the quantity of acetone to be added when different grades of suspending oils are used. The reason for such variation is the differing solubility of the acetone in the different grades of oil. The heavier the oil, the less the solubility of the acetone therein, and consequently the less the amount of the same which remains in solution in the suspending oil throughout the granulation process.

When the granulation is complete, the contents of the churn are removed, and placed in the rotating barrel shown in Fig. 2. This barrel is now rotated for a time without the application of any heat, the grains being thus partially compacted and rounded. Water, heated to a temperature a little lower than the boiling point of the solvent for the nitro-cellulose which is used, is then circulated through the jacket surrounding the barrel, and the rotation continued until an entire separation of the excess of acetone from the grains has been accomplished. Acetone, the solvent which I have described as being used, boils at about 165° Fahrenheit. When acetone is used, the proper temperature of the water is about 150° Fahrenheit. The vapors resulting from the application of this heat are conveyed away through pipe 7, a vacuum pump being applied to aid the carrying away of the same as rapidly as formed. The hardening and rounding of the grain thus take place during the removal of the excess of solvent, and while the grain is still immersed in oil, the rounding action appearing to go on better with the oil than without the same. The grain is now completely formed, free from excess of acetone, and in a hardened condition. It is, however, saturated with oil. To remove this oil, the grain is taken from the barrel, the excess of oil strained off, and the oily grain placed in the still shown in Fig. 3, which is partially filled with water. The temperature of the still is now raised, through the admission of steam into the steam jacket surrounding the lower portion of the same, until distillation commences. As the boiling point of the oil and the water is about the same, they pass off together, being condensed on the inner shell or cone R, and from thence falling into the annular trough S, from whence they are conveyed by the pipe T into the vessel U. A separation here takes place, due to the differing specific gravities of the oil and the water, the oil being drawn off through the test cock V, and the water being automatically returned to the still by the siphon W. There is thus no loss of oil, and should any of the solvent have remained dissolved in the oil, it will also be recovered.

The grains formed by this process have the same general character as those produced when nitro-benzole is used to effect granulation, but have a tendency to be harder and more compact, and perhaps to shrink a little tighter, thus making a denser powder, capable of occupying rather less space than the grains formed by nitro-benzole. The evaporation of the solvent in the manner described has the effect of leaving the grains with less channels or pores in it than that produced by the other process.

The detailed steps of my process will vary somewhat with the solvent of nitro-cellulose used, on account of the change in boiling point and specific gravity of the same. Thus when acetone is used, since its boiling point is much less than that of the oil, it will pass off first in the freeing of the solvent from the excess of solvent and the oil, while if a solvent is used which has a boiling point higher than that of the oil used, the oil will pass off first. If an oil is used which is heavier than water, the end of the siphon pipe extending down in the reservoir will have to be cut off so as only to return the lighter portion of the distillate, and the oil can be drawn off from the bottom of the reservoir. These variations, however, do not affect the essential feature of my process, which consists in suspending the nitro-cellulose in an oil, and then granulating the same by agitating therewith a solvent of the nitro-cellulose which, although soluble in the suspending oil, will leave the same to act upon the nitro-cellulose.

Among the high boiling point ketones that can be used in carrying out my process, I may mention methyl tri-methyl carbon ketone, which boils at 105° centigrade; dipropyl ketone, which boils at 145° centigrade; methyl propyl ketone, which boils at 100° centigrade; and a ketone formed by the dry distillation of calcic butyrate, thought to be ethyl-propyl ketone, which boils at 128° centigrade.

The explosive force of the grains produced, can of course be lessened, if desired, by the addition of any of the ordinary moderating agents. Such operation can be performed, for instance, in the manner described in the patent of myself and Pierre S. du Pont, No. 503,583, above referred to, and the moderating agents therein specifically mentioned may be used.

I do not confine myself to the particular apparatus described, nor to the particular proportions of ingredients mentioned, nor to the use of the particular oil and solvents stated, as these may be varied without affecting the merits of my invention.

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

1. The process of manufacturing a smokeless explosive, which consists in suspending nitro-cellulose in a suitable liquid not a solvent of the nitro-cellulose, granulating the nitro-cellulose by agitating therewith in suitable proportions a solvent of the same which, though soluble in the suspending liquid is still capable of exerting a solvent action upon the nitro-cellulose, and subsequently hardening the grains thus produced, substantially as described.

2. The process of manufacturing a smokeless explosive, which consists in suspending nitro-cellulose in oil, granulating the same by agitating therewith in suitable proportions a solvent of the nitro-cellulose which, though soluble in the suspending oil, is still capable of exerting a solvent action upon the nitro-cellulose, and subsequently hardening the grains thus produced, substantially as described.

3. The process of manufacturing a smokeless explosive, which consists in suspending nitro-cellulose in a hydro-carbon oil, granulating the same by agitating therewith in suitable proportions a solvent of the nitro-cellulose which, though soluble in the suspending oil, has a stronger affinity for nitro-cellulose, and subsequently hardening the grains thus produced, substantially as described.

4. The process of manufacturing a smokeless explosive, which consists in suspending nito-cellulose in a hydro-carbon oil, granulating the same by agitating acetone therewith in suitable proportions, and subsequently hardening the grains thus produced, substantially as described.

5. The process of manufacturing a smokeless explosive, which consists in suspending nitro-cellulose in oil, granulating the same by agitating therewith in suitable proportions a solvent of the same which, though soluble in the suspending oil, is still capable of exerting a solvent action upon the nitro-cellulose, hardening the grains thus produced by agitation, accompanied by removal of the excess of solvent, and finally removing the oil from the grains, substantially as described.

6. The process of manufacturing a smokeless explosive, which consists in suspending nitro-cellulose in oil, granulating the same by agitating therewith in suitable proportions a solvent of the same, which though soluble in the suspending oil, is still capable of exerting a solvent action upon the nitro-cellulose, hardening the grains thus produced by rotation, accompanied by removal of the excess of solvent, and finally removing the oil from the grains, substantially as described.

7. The process of manufacturing a smokeless explosive, which consists in suspending nitro-cellulose in oil, granulating the same by agitating therewith in suitable proportions a solvent of the same which, though soluble in the suspending oil, is still capable of exerting a solvent action upon the nitro-cellulose, hardening the grains thus produced by rotation while immersed in the oil, accompanied by a removal of the excess of solvent, and finally removing the oil from the grains, substantially as described.

8. The process of manufacturing a smokeless explosive, which consists in suspending nitro-cellulose in a hydro-carbon oil, granu-

lating the same by agitating therewith in suitable proportions a solvent of the nitro-cellulose, which, though soluble in the suspending oil, is still capable of exerting a solvent action upon the nitro-cellulose, hardening the grains thus produced by rotation while immersed in the oil, and then by continued rotation accompanied by the removal of the excess of solvent, and finally removing the oil from the grains, 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.