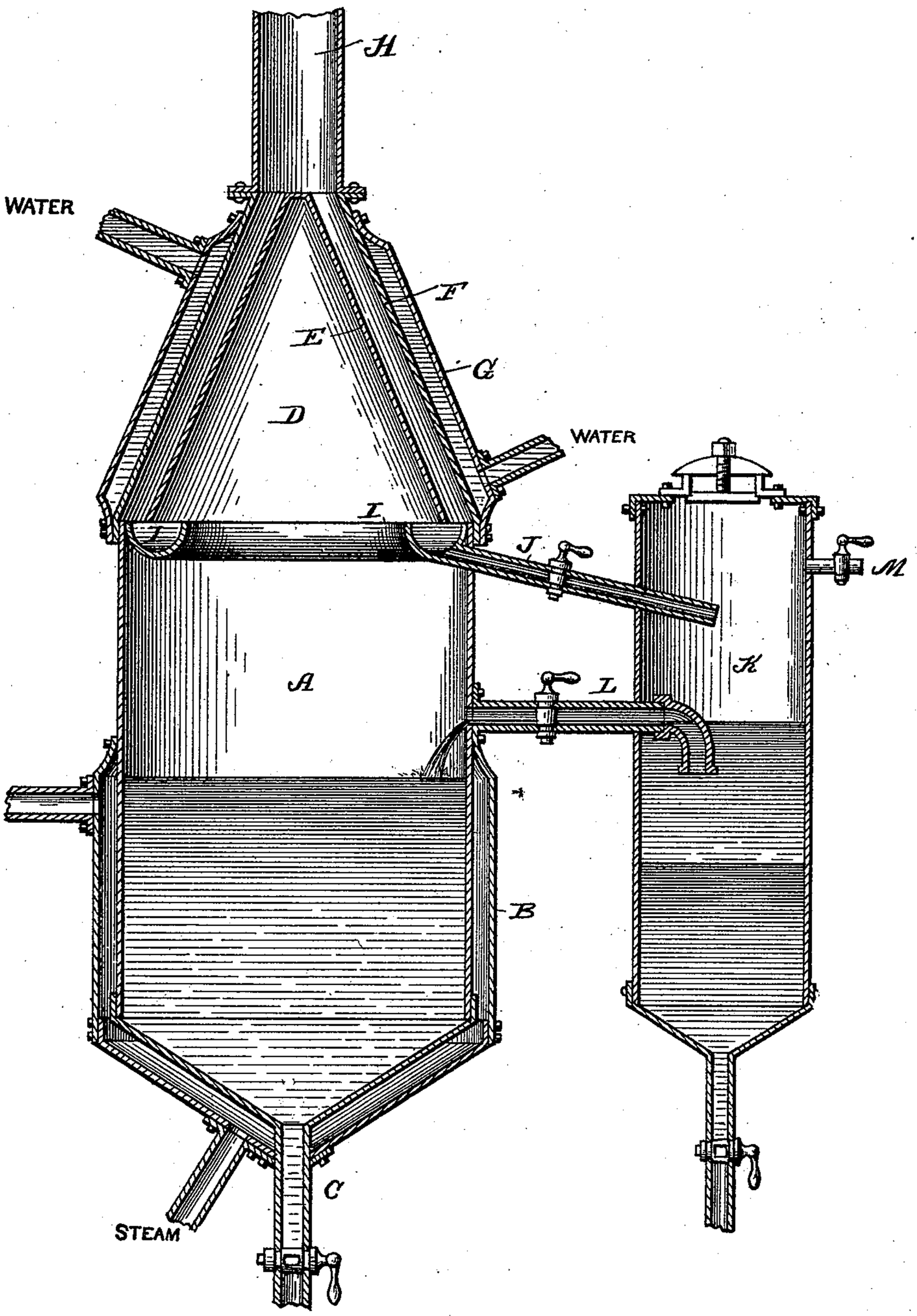
(No Model.)

F. G. DU PONT.

PROCESS OF DRYING NITROCELLULOSE.

No. 516,924. Patented Mar. 20, 1894.



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PROCESS OF DRYING NITROCELLULOSE.

SPECIFICATION forming part of Letters Patent No. 516,924, dated March 20, 1894.

Application filed June 15, 1893. Renewed February 23, 1894. Serial No. 501,288. (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 Processes of Drying Nitrocellulose; 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 processes for drying nitro-cellulose, and particularly that form thereof known as gun cotton, and it consists in an improved process for accomplishing this purpose by placing the wet gun cotton in a still filled with some volatile oil, and then distilling off the oil and the water contained in the fiber of the gun cotton, which will hereinafter be fully described, and particularly pointed out in the claims.

Gun cotton has always been an exceedingly difficult material to dry, for the reason that the process of drying by the mere application of heat is very dangerous, and accidents constantly take place when it is attempted. Gun cotton when wet is absolutely safe. It can only be exploded by specially prepared primers. It can be burned without danger, and no special care need be taken in storing it. But when gun cotton is dry, the case is entirely different. Dry gun cotton is very dusty, and the particles of it flying through the air are apt to explode from very slight, and apparently insignificant causes, such as an electric spark caused by the hand touching a piece of metal. These particles lodge at every point and crevice, and render necessary the greatest care to avoid accidents, which yet frequently happen.

I have discovered that gun cotton can be dried by placing the wet material in a still filled with volatile oil, such as kerosene or benzine, and then vaporizing the oil. If the boiling point of the oil used be lower than that of water, the water in the fiber of the gun cotton will be carried off mechanically by the particles of vaporized oil; but if the boiling point of the oil be about that of water, the two will be vaporized together. It is preferable to have the boiling point of the oil at about 212° Fahrenheit, as the distillation then takes place more uniformly and rapidly than when the oil has a lower boiling point. The dry gun cotton obtained is impregnated with the oil in which it has been placed, but this does not prevent the use of the same for many important purposes, such as for the manufacture of blasting gelatine by admixture with nitro-glycerine, or the manufacture of collodion varnish by mixture with ether, or for use in the manufacture of celluloid articles, and for treatment with solvents in all processes in which dry gun cotton is necessary, though I do not restrict myself to these uses. By this process of drying gun cotton, all dust is avoided, and the product is collected in a compact form, and in a condition which renders it less liable to cause accidents than the common dry gun cotton. By observing the amount of water driven off in the course of distillation, it is easy to determine when all of the water has been driven off; or the gun cotton may be left with any desired degree of moisture therein, if it is wished to leave the same slightly wet.

In the drawings accompanying and forming a part of this application, I have represented a form of still that is especially fitted for use in this process, although I do not limit myself to carrying out my process with this particular form of still, mentioning it merely as a convenient form. I have made separate application for Letters Patent of the United States for this still, said application having been filed April 13, 1893, and serially numbered 470,347. The feature of the same which renders it especially applicable to the use suggested is that it permits an automatic return to the main still of the oil driven off from the same, while enabling an accurate determination of the amount of water distilled off to be made. The use of large quantities of oil is thus rendered unnecessary, and the care which has to be taken with the apparatus is much lessened.

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

Referring to the drawings, A represents the still, which is of any convenient size and construction, and has around its lower portion, B, an encircling steam jacket, provided with steam inlet and outlet pipes. The pipe C, 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, D, around the outer shell, F, of which is placed the jacket G, which is provided with water inlet and outlet pipes, and between which, and the said shell F, a stream of cold water is constantly flowing when the still is in operation. The outlet H is for the conduction of vapors escaping from the still to a suitable condenser, which 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 shell F, and of a form corresponding to that of said shell, is suspended the inner shell or cone E, the function of which is to prevent the distillate coming from the condenser from falling again 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 I, which surrounds the interior of the still at the base of the still-head. The pipe J affords an exit from this trough into the outer vessel K, which answers as a reservoir for the distillate. This reservoir has a conical bottom, and a pipe at the apex of the same, from which may be drawn from time to time the water which collects in the same, the water being heavier than the oil. The said reservoir is also provided with an air-tight closure at its top, and a stop cock M for the admission of air. Besides the connection afforded by pipe J, the reservoir is also connected with the still by the overflow pipe L, which is joined to the still at a point a little above the level of the contents of the same, and enters the reservoir at a point below the level of discharge of the distillate, thus allowing the oil, which in this case is the lighter product of distillation, to flow back into the main still as rapidly as formed. Cocks are provided in the two pipes connecting the still and the reservoir, so that in case a vacuum is used in connection with the outlet pipe of the still, they may be closed, and air admitted to the reservoir through the cock M, restoring atmospheric pressure, and enabling the heavy liquid at the bottom of the reservoir to be drawn off from time to time as needed.

Proceeding now with my process, I place in the still about two hundred gallons of a suitable volatile oil, such as kerosene or benzine, and I prefer to have the boiling point of this oil at about 212° Fahrenheit, as thereby the water in the fiber of the gun cotton is more uniformly and more rapidly removed. I now place in the still about two hundred pounds of gun cotton, in a state of pulp, which, having been drained of its surplus water, contains about fifty **per** cent. of its weight of water. A vacuum pump is preferably connected to the pipe H, in order to keep the contents of the still at a low temperature, which is an advantage. Steam being now circulated through the steam jacket, distillation commences. The vapors produced rise to the conical top of the still, and a portion of the same will be condensed on the inner side of shell E, and will by said shell be delivered into the annular trough I, from whence it will be conveyed to the reservoir K by pipe J. The uncondensed portion of the vapors passes under the edge of the inner cone, and up between the outer and inner cones. Since the outer cone is surrounded by cold water, a further and much greater condensation takes place here, this portion of the distillate, like the preceding, being delivered into the reservoir K by pipe J. If any uncondensed vapor remains, it passes upward through pipe H into the condenser, and is there condensed, whence it finds its way into the annular trough I, and from thence, by pipe J, into the reservoir K. All of the distillate is thus condensed, collected in the annular trough, and delivered into the reservoir. When the distillate reaches the reservoir, a separation of the products forming the same takes place, the heavier product of the same, water, falling to the bottom of the reservoir, and the lighter product, oil, (after the level of the contents of the reservoir has reached that of the pipe L,) passing over into the main still. The quantity of water collected is a constant index of the amount dried out of the gun cotton, because it is easy to ascertain the average amount of water that is contained in the original gun cotton. This water should be drawn off from time to time, in order that its level may not rise to that of the pipe L, and thus water be returned to the still. While this operation is being performed, the cocks to pipes J and L should be closed, and the cock M opened, so that the ordinary atmospheric pressure may exist in the reservoir. When no more water collects in the reservoir, the process is finished, and the contents of the still are drawn off, the surplus oil being drained off from the gun cotton. The gun cotton is now in condition to be used for any process in which the presence of oil is not objectionable.

The process may be carried on with a lighter oil in the still, but in such case the operation of removing the water in the still is necessarily slower, as the temperature is not so high, and the water comes over more slowly, most of it being carried mechanically by the vapoized oil, so that if it is desired that the fiber of the gun cotton should be impregnated with a lighter oil, it is preferable to accomplish this by percolation, after the process of drying the gun cotton has been finished. If such lighter oil is used, the process of carrying off the water is first one of evaporation, and then of mechanically carrying over the watery vapor with the vapor of the low boiling oil. For instance, take an oil which boils at 75° centigrade. This is considerably below the boiling point of water, and yet when water, mixed with gun cotton, is placed in such an oil, the vapor of the water is carried off rapidly by the vapor

of the oil. A very volatile oil will of course carry off the oil too slowly, and there is a limit at which the process will go on too slowly for practice. This limit may be set at about 50° centigrade, although the process may possibly be carried on at a lower temperature. The quantity of oil evaporated has nothing to do with the economy of the process, except in loss of time, because the oil is constantly returned to the still, separate from its water, and the process can go on in a continuous cycle. While it is thus true that when oils are used whose boiling point is lower than that of water, the removal of the water takes place very slowly, the reverse is the case when oils are used whose boiling points are higher than that of water. In this case, the process will go on very rapidly, but here arises a consideration which does not hold with oils of low boiling point. Suppose for instance that I use an oil whose boiling point is 120° centigrade. When the still is heated, the water in the gun cotton will keep the boiling point down as long as it is coming off, but as the quantity of water in the gun cotton becomes less and less, the boiling point will continually rise; so that when the cotton is dry, it will be heated up to the temperature of the oil, a temperature which is detrimental to its stability. This does not, however, prevent the drying in oils of even a very high boiling point.

In case an oil is used which has a greater specific gravity than water, it will sink to the bottom of the reservoir, and the water will rise to the top of the same. The oil is returned in such case by extending the branch of the pipe L which extends into the reservoir to the bottom of the same.

I do not confine myself to the use of kerosene, or benzine as the oil in which to place the gun cotton for removal of the water contained in its fiber, but use for that purpose any volatile oil that is found to be suitable; nor do I confine myself to the use of my process in drying gun cotton, for I may dry by it other forms of nitro-cellulose.

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

1. The process of drying nitro-cellulose which consists in placing the wet nitro-cellulose in a volatile oil, vaporizing the oil, and thereby removing the water in the fiber of the nitro-cellulose, substantially as described.

2. The process of drying nitro-cellulose which consists in placing the wet nitro-cellulose in a hydro-carbon oil, vaporizing the oil, and thereby removing the water in the fiber of the nitro-cellulose, substantially as described.

3. The process of drying nitro-cellulose which consists in placing the wet nitro-cellulose in kerosene, vaporizing the oil, and thereby removing the water in the fiber of the nitro-cellulose, substantially as described.

4. The process of drying nitro-cellulose which consists in placing the wet nitro-cellulose in kerosene oil having a boiling point of 212° Fahrenheit, vaporizing the oil and the water in the fiber of the nitro-cellulose, and thereby drying the nitro-cellulose, substantially as described.

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

FRANCIS G. DU PONT.

Witnesses:
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