

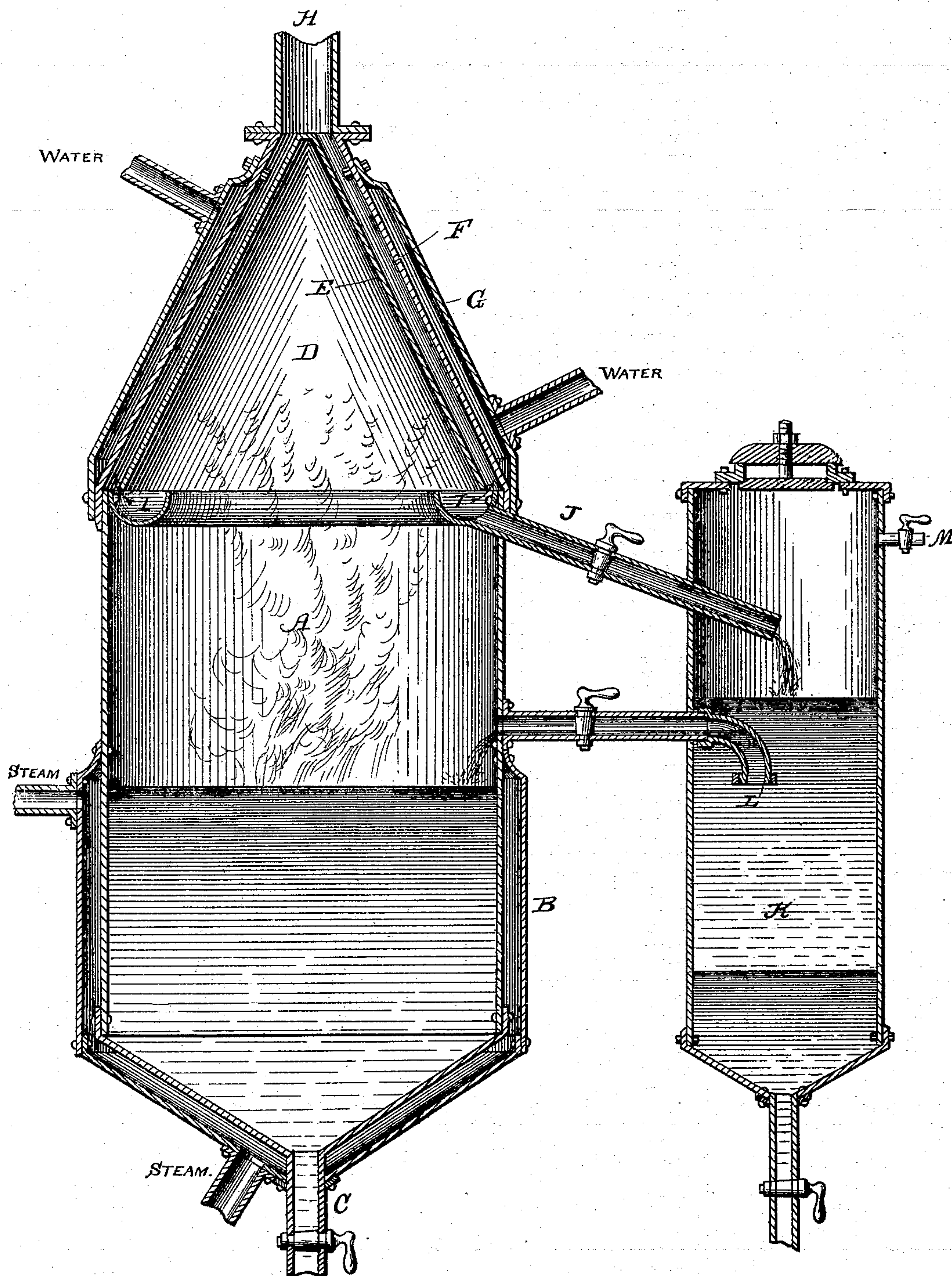
(No Model.)

F. G. DU PONT.

APPARATUS FOR DISTILLING VOLATILE SUBSTANCES.

No. 503,586.

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Witnesses.

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APPARATUS FOR DISTILLING VOLATILE SUBSTANCES.

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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 Apparatus for Distilling Volatile Substances; 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 apparatus for distilling volatile substances, and it consists in an improved form of still, especially designed to be used in the separation of the solvent from the grains of explosive formed by the process of making smokeless powder described in the joint application of myself and Pierre S. du Pont, filed December 21, 1892, Serial No. 455,901, which will be hereinafter fully described, and particularly pointed out in the claims. In said application, the process of making smokeless powder is described, which consists in suspending finely divided nitro-cellulose in a suitable liquid, granulating the same by adding thereto in proper proportions a suitable solvent of nitro-cellulose, and finally removing the excess of solvent, and treating the grains so as to harden and solidify them. As a preliminary or accompanying step to such solidification and hardening of the grains, it is necessary to remove the excess of solvent from the grains, and it is the object of my invention to provide a form of still, which will enable this operation to be carried on easily and rapidly; which will enable the solvent recovered by the distillation to be collected in such a manner as to allow easy measurement of its amount, and therefore of the extent to which the distillation has proceeded; and which will enable the level of the contents of the still to be automatically maintained at or near a certain point, thus facilitating the operation of distillation. This object I attain by the use of a main still, provided with means for condensing and collecting the vapors formed therein, and an auxiliary reservoir or collector used in connection therewith, which is so arranged as to have the distillate from the main still delivered into it, and is further con-

nected to the still by a pipe jointed to the still at a point slightly above the level of the liquids in the same, and to the reservoir at a point below the level of discharge of the distillate, the connection being such that after the level of the contents of the reservoir reaches that of the said overflow pipe, any further addition to its contents is siphoned over to the main still. The distillate obtained by the special use of the still herein described consists of the solvent vaporized from the grains of explosives which are placed in the tank, and the water which is added to the grains. Of the two-fold distillate thus obtained, the distilled solvent, being heavier than the condensed water, settles to the bottom of the reservoir, and can be drawn off and measured at any time, thus permitting an accurate quantitative determination of such distillate to be obtained. The distilled water is, in the manner above stated, automatically returned to the still, and as the amount of distilled solvent is much less than that of the distilled water, and as nearly all of such water is returned to the still, the level of the contents of the same is maintained practically constant.

The apparatus forming my invention is fully represented in the drawings accompanying and forming a part of this application, which is a sectional view of the still, the reservoir, and the parts used in connection therewith.

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 any 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 solvent which collects in said bottom, the solvent being heavier than water. 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 water, which is the lighter product of the 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.

The operation of my still is as follows:—A portion of the grain produced by the process described in the application above referred to, and the solvent found in connection therewith, is placed in the still, and water is added about to the level of the jacket. A vacuum pump is then connected to pipe H, and steam circulated through the jacket surrounding the bottom of the still. The object of using the vacuum is to decrease the atmospheric pressure in the still, thus hastening the ebullition of the contents of the same, and preventing the temperature of the same rising above the desired point, which is, when treating the grains of explosive, about 140° Fahrenheit. The vacuum is not necessary, however, in the treatment of all classes of volatile substances, and its use does not form an essential portion of my process. The vapors thus 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 deliv-

ered 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, and delivered on the outer surface of the inner cone, 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 solvent falling to the bottom of the reservoir, and the water, after the level of the contents of the reservoir has reached that of the pipe L, passing over into the main still. From time to time, the solvent should be drawn off from the reservoir, in order that too much of the solvent may not gather therein, and 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. In this manner, the exact quantity of solvent distilled from the grain of the still may be ascertained, and thus it can be determined when the distilling operation has proceeded far enough. The solvent recovered is perfectly pure, and is in condition to be used over again in the production of a fresh quantity of explosive grain. As the amount of solvent recovered is small in proportion to that of the water distilled over into the reservoir, and as the amount of such solvent is small in comparison with the amount of water in the still, the automatic return of the water to the still maintains the level of the contents of the same practically constant. It is thus unnecessary to at times interrupt the operation of the still, and introduce a fresh supply of water into the same. After the siphoning action has commenced, the then level of the contents of the still is maintained almost absolutely constant, a slight deviation being due to the removal of the distilled solvent. The time of commencement of such siphoning action depends largely upon the relative proportions of the still and reservoir, and the position of the overflow pipe in said reservoir.

It is evident that by prolonging the siphon pipe L to the bottom of the reservoir, and by providing a side outlet for the reservoir, I may use my apparatus for the separation and recovery of the lighter of the two products of distillation, returning the heavier product through pipe L to the still, and drawing off the lighter product through the side outlet of the reservoir, and I do not confine myself to the separation and recovery of the heavier product of distillation.

I do not confine myself to the exact form and proportions of the apparatus described herein, or to the use of the apparatus in the process of manufacture of smokeless powder.

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

1. In a still, the combination with the coni-

cal top of the same provided with refrigerating means, of an inner conical shell suspended within the top, an annular trough arranged so as to collect the distillate, and a delivery pipe for said trough, substantially as described.

2. In an apparatus for distilling volatile substances, the combination with a main still provided with a conical top having refrigerating means, an inner conical shell suspended within said top, and an annular trough arranged so as to collect the distillate, of a reservoir for receiving the distillate, provided with an outlet for one of the condensed products, means for automatically returning the other condensed product to the main still, and a pipe leading from the annular trough of the still to the reservoir for conducting the distillate to the same, substantially as described.

3. In an apparatus for distilling volatile substances, the combination with a main still provided with a conical top having refrigerating means an inner conical shell suspended within said top, and an annular trough arranged so as to collect the distillate, of a reservoir provided with an outlet at its bottom, a pipe conducting the distillate to the reservoir, and a pipe connecting the reservoir with

the still at a point above the level of the liquids in the still, substantially as described.

4. In an apparatus for distilling volatile substances, the combination with a main still provided with a conical top having refrigerating means, an inner conical shell suspended within said top, and an annular trough arranged so as to collect the distillate, a reservoir having an outlet opening at its bottom, a pipe conducting the distillate to the reservoir, and a pipe connecting the still with the reservoir at a point above the level of the liquid in the still, and below the point of entrance of the distillate, substantially as described.

5. In an apparatus for distilling volatile substances, the combination with a main still A, provided with the inner shell E and annular trough I, of a reservoir K, having an outlet opening at its bottom for the heavier condensed product, and connected to said still by the distillate-discharge pipe J, and the overflow pipe L, 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.