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PROCESS OF SEPARATING SOLVENTS FROM NITRO-COMPOUNDS.

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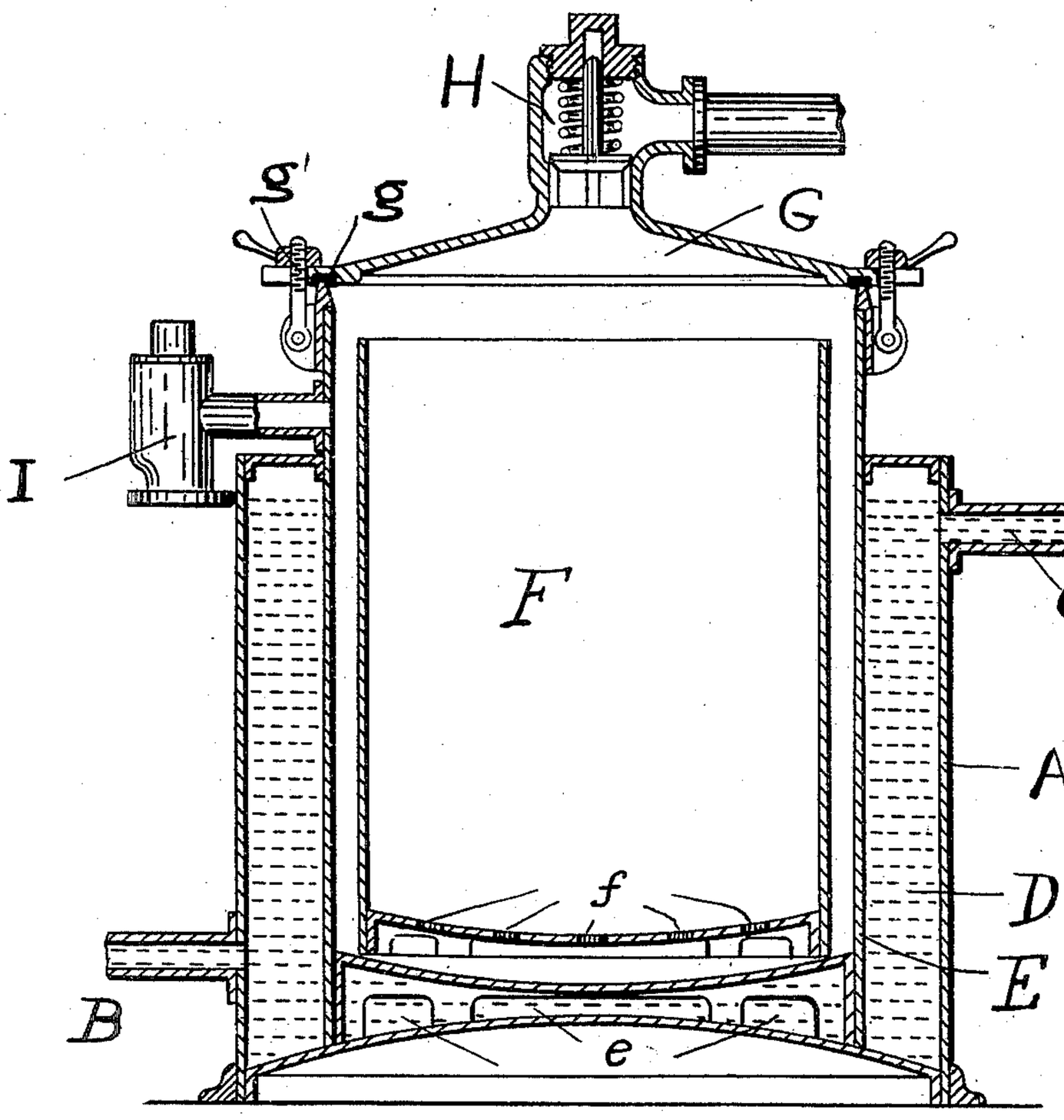
(No Model.)



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PROCESS OF SEPARATING SOLVENTS FROM NITRO COMPOUNDS.

SPECIFICATION forming part of Letters Patent No. 704,628, dated July 15, 1902.

Application filed April 12, 1902. Serial No. 102,496. (No specimens.)

To all whom it may concern:

Be it known that I, EMIL GATHMANN, a citizen of the United States, residing at Washington, in the District of Columbia, have invented a new and useful Improvement in Processes of Separating the Solvents from Nitro Compounds, of which the following is a specification.

My invention relates to an improved process for extracting the solvent material from nitro compounds. In commercial terms it might be expressed as "an improved process for drying smokeless powder." In preparing such nitro compounds it is necessary to use a volatile solvent or solvents, and after the process of manufacture such solvents must be separated from the solid mass as completely as possible. In usual methods there is a rapid escape of the solvent vapor and a rapid contraction of the solid body very soon after the process of manufacture has been completed, so that with ordinary smokeless powders much the larger proportion of the solvent passes away as vapor within two or three days; but the result is that the surface hardens, the outer pores of the powder grains or rods close up, and a percentage of the solvent becomes virtually trapped or sealed within the body of the grain. It requires six to eight weeks to dry the usual section, and even then an undesirable percentage of the solvent still remains. Powder on shipboard actually dries for a year or so and the solvent never seems totally absent. About four-fifths of the total original quantity of solvent passes off in a comparatively few hours, while it requires weeks to reduce the balance to the stipulated minimum of three or four per cent. The reason for this I have found to be that the exterior surface of the powder-grain hardens by rapid drying and contracting, seals up a quantity of solvent in the interior, and prevents its escape. My invention prevents this undesirable result in a simple and efficacious way. I subject the powder or nitro compound to the action of solvent vapors on the exterior, and thus maintain its porosity and keep open the ways of escape for the vapors from the interior of the grain. As long as the compound is acted upon by these vapors its surface will be prevented from the sealing-up process, and by subjecting it to a very moderate heat the volatile vapors will rapidly pass off and the mass will be dried uniformly and to practical perfection in a brief period. I find that I can dry smokeless-powder sections by this means in much shorter time and much better than the same compounds can be dried by the usual means and now known methods.

In a broad sense my invention is therefore a process or method of drying nitro compounds by maintaining the same in the presence of solvent vapors or a bath of solvent vapor, thereby keeping the nitro compounds porous to allow the free escape of the solvent vapors from all parts of such compound.

In the accompanying drawing, which is illustrative merely, the figure is a vertical section of a type of evaporating apparatus with which the process can be carried out.

The outer vessel A is provided with induction-pipe B and eduction-pipe C, said induction and eduction pipes supplying and giving vent for the circulation of the heating medium D, conveniently hot water. Sitting within the outer vessel A is a second vessel E, which is provided with a closed bottom *e*. In the construction shown this vessel is centrally placed, and its side walls form an annular space, in conjunction with the side walls of the outer vessel A, for the heating medium D. A third receptacle or tank F, conveniently, but not necessarily, made of a material of poor heat-conducting power, (or lined therewith,) is placed within the vessel E. The bottom of vessel F is preferably provided with perforations *f*, or it, as well as the sides, may be made of a porous or perforated material. Cover-plate G is provided for the vessel E, which is fitted with gaskets *g* and securing bolts and nuts *g'*. The cover-plate G is adapted to seat on the vessel E and is provided with a spring-actuated pressure pop-valve H at its apex. Vacuum or inward-acting pop-valve I is provided, which opens into vessel E.

The colloided nitro compounds, shaped into any desired forms of grain or masses, are placed in the receptacle F as they come from the dies or mold. In this condition the colloid material or nitrocelluoid is still impregnated with a large proportion of its total

weight of volatile solvents or emulsion. Taking the apparatus as shown, the method or process of expelling the solvent or drying the colloid and indurating it is as follows: The green grains or masses of material being placed in the vessel or receptacle F, the cover-plate G is placed on the upper end of the vessel E and firmly secured by the bolts g'. Water heated to from 130° to 150° Fahrenheit is forced into the vessel A and surrounds vessel E at its side and bottom. The height to which the water will rise in vessel A is regulated by the position of the eduction-pipe C. By this means a circulation of hot water can be maintained. As the contents of the vessel becomes heated the vapors of the solvent will fill the vessel, which being sealed the compound will be retained in a vapor-bath. As soon as the pressure in vessel E is sufficient to overcome the spring-pressure on valve H the said valve will be opened and a portion of the vapor permitted to escape. Thus the percentage of solvent in the vessel E and that within the colloid material is reduced, and the body of the compound becomes indurated and denser. The exterior of the grains or masses, however, remains "comparatively soft," so to say, by reason of the action of the vapors of the solvent by which they are surrounded until practically all the solvent, or at any rate a high percentage of the same, has been expelled from the grain or masses of colloid material, and the volatile solvent vapors are permitted to escape against a predetermined tension or pressure on valve H.

The keeping of colloid materials surrounded by a vapor bath of solvent, thereby keeping the exterior or surface portion of the grain or mass of such material soft and penetrable, and thus freely allowing the escape of and conversion into vapor of the contained liquid solvent, is the essential feature of my invention. Any suitable method and construction of apparatus for carrying out this process may be employed, and the type I have shown is illustrative merely.

In place of the heated water D any other liquid might be employed, or steam, heated air, or any other form of imparting heat to the nitro compound might be employed, without departing from the spirit of my invention.

My system or process may be employed to further dry or expel remaining volatile solvent from powder-grains or colloid masses that have been previously dried by other methods. In such cases I prefer to add a certain percentage of solvent contained in the form of vapors through the check or vacuum valve I, or the solvent may be placed directly in bottom of tank or vessel E or F and allowed to vaporize and pass through the mass or, rather, surface of the colloid material which has been placed in the vessel F in a similar manner, as through the green masses of material previously described. Powder rods or sections already dried by usual process may have remaining solvent removed by putting them in with green grains, which will furnish sufficient solvent to soften their hardened surface and permit the trapped solvent from the interior to escape.

The surface of colloids being softened, the process of lowering the percentage of solvents in the same occurs in a similar manner as when the green grains alone were treated.

I am aware that powder sections or rods have been boiled or steamed to effect the removal of the solvent. This process is unsatisfactory, because while quite efficient in removing the solvent the action of the applied heat affects injuriously the physical qualities or characteristics of the colloid material.

Apparatus for recovery of volatile solvents, such as distilling and condensing appliances, may obviously be used in conjunction with my process.

It is to be understood that the invention comprehends, first, the utilization of the solvent contained in the nitro compound; second, the utilization of a solvent other than that contained in the compound, and, third, the utilization of the solvent contained in the compound with the addition of other solvent, either of which steps may be resorted to to keep the compound in the bath of solvent vapor sufficient to soften or maintain in a soft condition the outer portions of the compound.

Having thus described my invention, I claim as follows:

1. The method of drying nitro compounds consisting in placing the compounds in a sealed vessel, heating and retaining the compound in the vessel in the presence of its solvent vapor until the solvent of the compound is practically completely liberated therefrom.

2. The method of drying nitro compounds consisting in subjecting the compound in a sealed vessel to the action of heat in the presence of a solvent, maintaining the compound in the presence of sufficient solvent vapor to keep it porous, and continuing the treatment until the compound is substantially dry.

3. The method of drying nitro compounds consisting in maintaining the compound in a bath of solvent vapor, applying heat, permitting portions of the vapor to escape, and adding additional solvent to maintain the porosity of the compound.

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