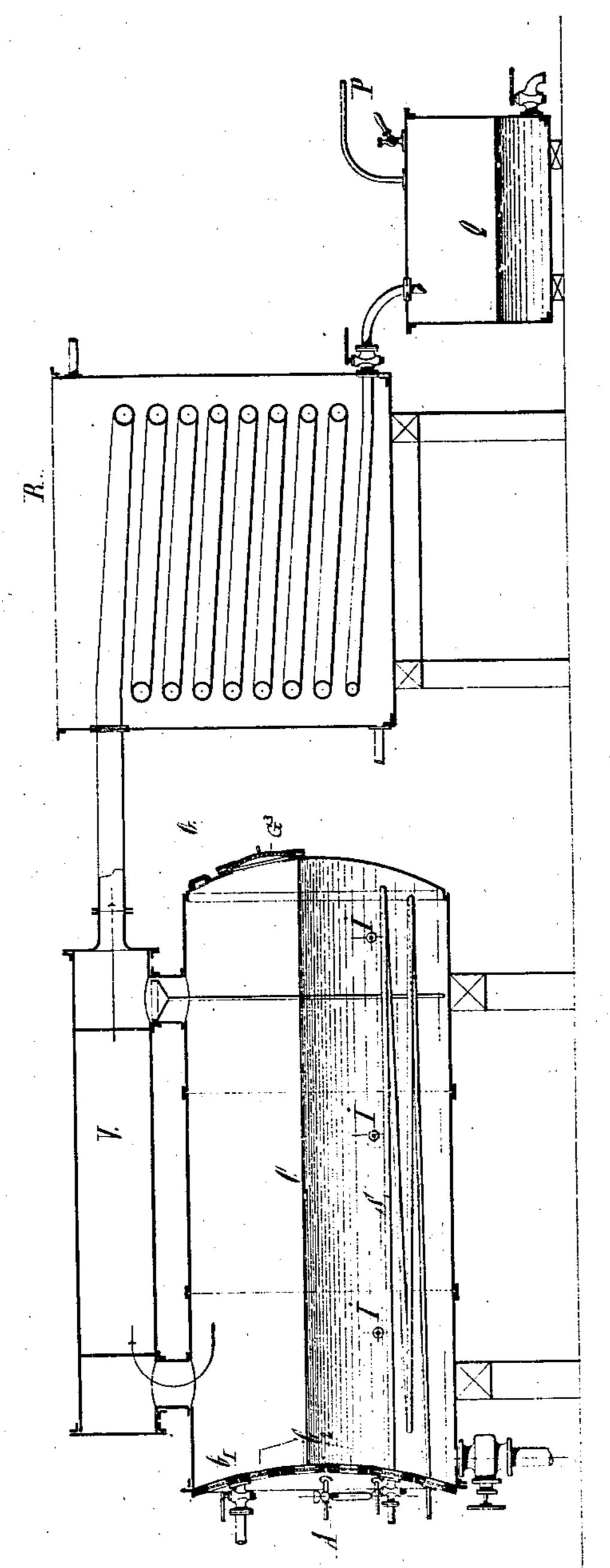
(No Model.)

J. MASSIGNON. PURIFICATION OF FATTY SUBSTANCES.

No. 481,502.

Patented Aug. 23, 1892.



Inventor:

Witnesses & Bolton Jones,

 $\mathcal{B}_{\mathcal{J}}$

Dules Massignon Mewards H his Attorneys.

United States Patent Office.

JULES MASSIGNON, OF PARIS, FRANCE, ASSIGNOR TO THE SOCIÉTÉ ANONYME DES PARFUMS NATURELS DE CANNES, OF SAME PLACE.

PURIFICATION OF FATTY SUBSTANCES.

SPECIFICATION forming part of Letters Patent No. 481,502, dated August 23, 1892.

Application filed June 13, 1891. Serial No. 396,119. (No specimens.)

To all whom it may concern:

Be it known that I, Jules Massignon, a citizen of the Republic of France, residing in the city of Paris, France, have invented certain new and useful Improvements in the Purification of Fatty Substances Extracted by Volatile Solvents, of which I do hereby declare the following is a true, clear, and exact description.

The present invention has for its object the complete separation from fatty substances of the volatile solvents that have been employed

in treating the same.

Heretofore it has been common to drive off 15 the solvent by evaporation, using steam-heat and reduced pressure in the evaporating-vessel, and while these methods result in the removal of the great bulk of the solvent the last traces thereof (whose presence renders the 20 fatty substance of little or no value) are very difficult to remove. Moreover, in treating the fatty substance with jets of steam in the usual way there is more or less condensation, necessitating another operation to remove the wa-25 ter. According to the present invention, the elimination of the solvent and water is accomplished by evaporation in vacuo until ebullition ceases, then injecting dry steam in fine jets, maintaining the temperature of the fatty 30 substance above the boiling-point of water.

In order that the invention may be more fully understood, I will describe the operation in detail, reference being had to the accompanying drawing, which illustrates in longitudinal sectional elevation apparatus suitable

for use in practicing the processs.

The said apparatus composed of a cylindrical boiler C contains a steam coil-pipe S to heat the interior thereof, and at its side said to boiler is provided with vapor-injectors I for the purpose of introducing jets of steam. At one end the boiler is provided with a series of valves A, enabling the material under treatment to be sampled from time to time. Glass-tovered inspection-openings G' G² permit the condition of the material to be observed from the outside. A dome V, mounted upon the body of the boiler, forms a safety vessel and shows any priming which might be caused in 50 case of the emulsion of the material. A pipe

leads from the dome to the coil contained in the refrigerator R, the other end of said coil discharging into a receptacle Q, with which a vacuum-pump P communicates. The fatty mass containing the water and solvent is in- 55 troduced into the boiler through the manhole G³, beneath the opening G, and the heat exerted by the coil S will vaporize the solvent and water and drive off the same to the dome and condenser for separation. The vaporad mitted 6c through the injectors I effects the evaporation. of a large portion of the solvent and its separation from the fatty body. The condenser R concentrates the solvent, after which it is passed into the receptacle Q for further treat- 65 ment. Samples are taken from time to time by means of the valves A, and when it is seen that the solvent has been completely eliminated the operation is at an end. The vacuumpump P and the injectors I maintain the nec- 70 essary relative temperature throughout the operation. Evaporation of the solution being completed in the vacuum there is at a given moment no more ebullition of the solvent, although the matter still contains an appreci- 75 able quantity. This period is readily determined, first, by the appearance of the material as seen through the inspection-openings and also because as the heating continues the temperature rises, while at the same time the 80 degree of pressure diminishes. It is at this time that I apply my method of purification to completely eliminate all traces of the solvent without introducing water into the fatty substances, and it is for this purpose that the 85 temperature of the fatty body to be purified in the apparatus is maintained above the boiling-point of the water, while steam as dry as possible is introduced into the mass by one or several streams, this steam being under a 90 high pressure. (Thus steam escaping from the steam-motor can easily be utilized for this purpose.) It is useful, however, that the steam jet or jets be applied in a manner to produce energetic stirring in the fatty mass, so as to 95 bring all particles of the mass into contact with the steam. This result can be easily obtained by the use of injectors. The steam thus injected into the apparatus not only does not condense, but the particles of water which 100 it may contain will be vaporized by the heat of the fatty matter. The matter, in fact, is constantly maintained during the operation at a temperature above that corresponding to 5 the boiling-point of water in the vacuum, as already explained. This condition is essential, because without it the mass would cool off—first, by the volatilization of the solvent; second, by the evaporation of the drops of water always borne by the steam whatever care be taken to free it before its entry into the apparatus and which results, also, from the relaxation of pressure of the steam at the moment of introduction into the vessel C.

The operation is governed by the following considerations: First, the temperature of the fatty mass in the interior of the apparatus is in no case to be higher than the point at which the fatty body is susceptible to change; sec-20 ond, this temperature being determined regulates, also, that of the steam, which must be some degrees lower than that of the fatty mass; third, the temperature of the steam fixes the degree of the vacuum at which the operation is 25 to be conducted by virtue of the law of variation of the boiling-point of any liquid with the variation of the pressure to which the same is subjected; fourth, the difference of temperature above indicated between the fatty 30 body and the steam in the interior of the apparatus must be so much larger and the vacuum so much the greater as the boiling-point of the solvent is raised. This difference must also he increased in inverse proportion to the

dryness of the steam. It will also be advisable, if the fatty body under treatment is susceptible to change at a temperature lower than 100° centigrade, to expand the steam entering into the double bottom by making therein a vacuum sufficient to insure that in 40 no case will the temperature of the side heated by this double attain the temperature at which that fatty body would undergo an alteration. Under these conditions the steam acts on the fatty body in the apparatus like a gas, but a 45 gas which condenses afterward in a condenser at the same time as the vapors of the solvent and does not hinder in any manner the condensation of this latter.

The described process of separating volatile solvents from fatty substances by evaporating the solvent in a vacuum or rarefied medium until ebullition ceases, then injecting dry steam in jets into the body of the 55 substance and maintaining the latter during the operation at a temperature above the boiling-point of water under the conditions present and above the temperature of the injected steam, as set forth.

In testimony whereof I have signed this specification in the presence of two subscribing witnesses.

JULES MASSIGNON.

Witnesses:
ROBERT M. HOOPER,
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