

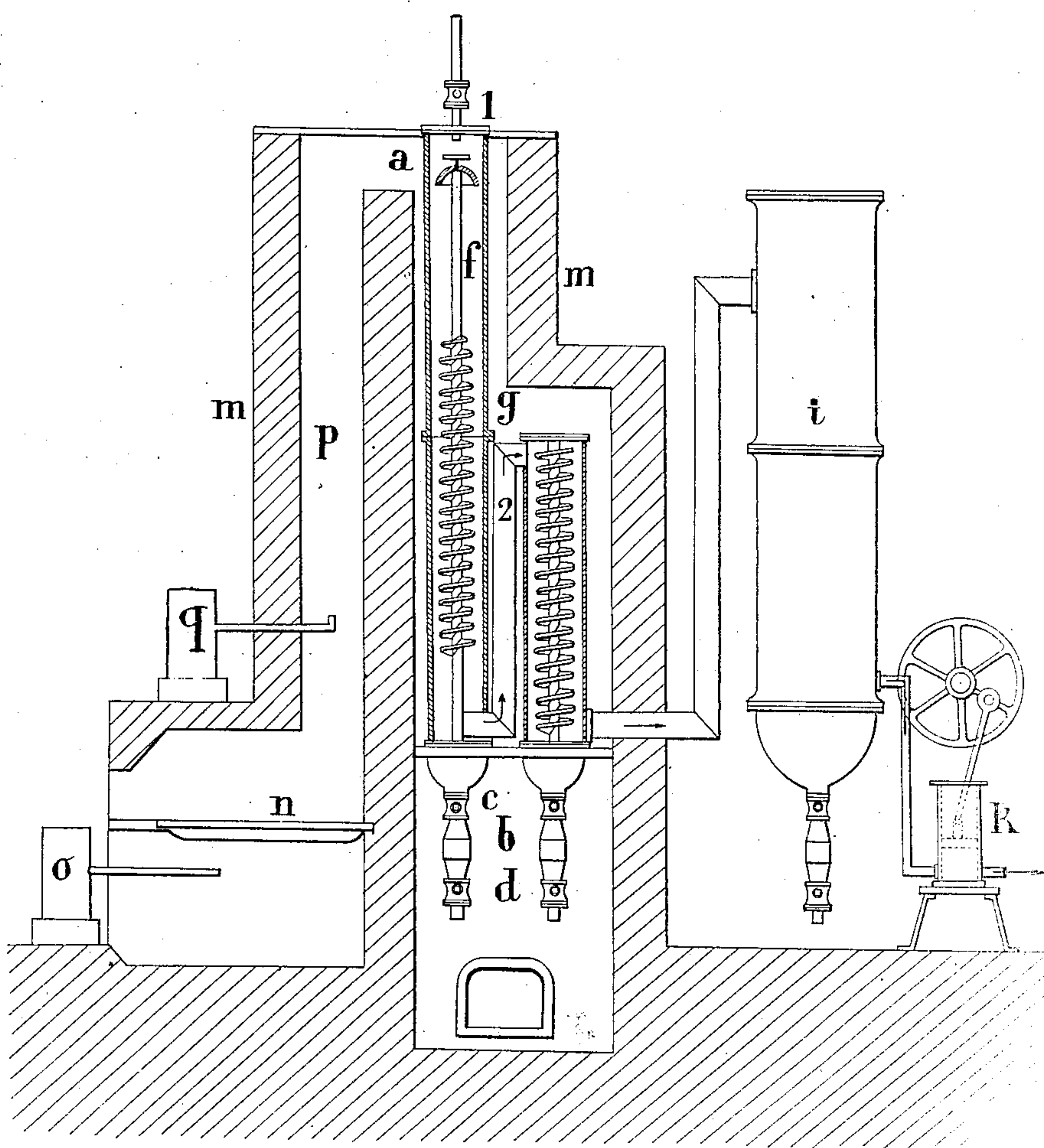
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PATENTED JAN. 23, 1906.

C. SUDRE.

PROCESS OF TREATMENT OF VINASSES.

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WITNESSES

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# UNITED STATES PATENT OFFICE.

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## PROCESS OF TREATMENT OF VINASSES.

No. 810,442.

Specification of Letters Patent.

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*To all whom it may concern:*

Be it known that I, CHARLES SUDRE, of No. 33 Boulevard Haussmann, Paris, France, have invented a certain new and useful Process of Treatment of Vinasses; 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.

This application is a division of a prior application filed by me on the 30th day of December, 1902, Serial No. 137,127.

Up to the present in any process for treating the residuary liquors of the distillation of the syrup of beet-roots called "vinasses" in order to extract from them glycerin and other useful products some mechanical apparatus for scraping or cleaning the exhausted products has always had to be employed.

By reason of the very construction of any previous apparatus numerous by-products are produced, which pass into the distilled liquid and render it very impure. It is well known that in order to extract the glycerin without decomposition it is necessary to work in as complete a vacuum as possible. The vinasses under the action of heat and in a vacuum first of all distils water and a little glycerin, when the material becoming anhydrous it distils the rest of the glycerin. During the first part of the operation the steam becoming disengaged produces a froth and carries numerous particles which hinder the operations and in many of the processes used up to now render the working impossible if the vinasses are viscous.

The process which is the subject of the present application for a patent does away with this inconvenience. It is based on the following discoveries and appliances:

First. If the vinasses from a distillery (of treacle, for example) be distilled in a vacuum at a temperature of not more than  $300^{\circ}$ , the residue after the glycerin and the water have been extracted, is liquid at this temperature of  $300^{\circ}$  and flows away of its own accord. The glycerin and water are extracted in a length of time which depends on the thickness of the vinasses. My process consists, in principle, of providing along the walls of an inclosure, suitably arranged, in which there is a vacuum and which is heated by some exterior source of heat in such a way that the temperature of the walls cannot be greater

than  $300^{\circ}$ , a constant stream of the vinasses of regular and regulated thickness. Thus the vinasses enter and pour away in a continuous liquid state without the intervention of any mechanical apparatus and lose in their passage (supposing that this is of a length suitable to the thickness of the liquid film) first of all the water and then the glycerin.

Second. It is proved that if the vinasses are sufficiently acidulated the ammonia escapes only in infinitesimal proportions and remains concentrated in the residue.

Third. By forcing the vapors to take up in the apparatus itself a rapid gyratory helicoidal movement I obtain by centrifugal force the impingement against the heated walls of the particles carried by the vapors, which particles thereby have a fresh opportunity of being distilled.

Fourth. It is admitted that it is necessary to have a current of superheated steam to help the distillation of the glycerin when the vinasses have become anhydrous. In many processes this steam is introduced from without and on condensing produces a dilute mixture of water and glycerin. On the contrary I utilize for this purpose the steam arising from the vinasse itself, which is superheated in the apparatus while taking up its gyratory movement. The superheated steam then bathes and surrounds, before it leaves the apparatus, the glycerin which is given off by the anhydrous substance and draws it out of the distilling apparatus. The glycerin and water vapors exhausted by a pump are condensed into a mixture of glycerin and water, which is concentrated for sale, and the liquid residue made solid by cold and retaining all the ammonia is treated by some known means or sold as it is.

These principles being set forth, I will describe as a representative example a construction for the carrying out of the process shown in section diagrammatically in the accompanying drawing and by which I put these principles into practice; but it is evident that the forms may be infinitely varied.

The apparatus is designed to be heated by coke or charcoal. It consists of a distilling-column *a*, formed of cast-iron tubes with or without exterior ribs, three to six meters high, according to the quantity of water in the vinasses to be treated. Above this column is situated a distributor 1, which spreads



through an annular passage onto the interior surface of the column a continuous stream of the vinasses to be treated. At the foot of this column is placed a pipe 2 for the escape of the gas and vapors which are driven off by the vinasses under treatment. Below this the column is constricted and is terminated by a chamber *b*, provided with stop-cocks *c* and *d* and in which the residue collects and which is arranged in such a way that it can be drained off at regular intervals. Inside the column *a* is suspended or fastened in any convenient way a rod *f*, round which is coiled a helix *g*, of which the outside surface is very close to the walls of the cast-iron column. The gas and vapors escape into a shorter accessory column *h*, called the "purifying-column," smaller than the principal column, but also containing a fixed helix. The solid substances drawn in impinge onto the walls and, distilling glycerin while descending the whole length of the walls, enter a chamber like the first, while the gas and purified vapor are directed into a condenser *i* by a suction-pump *k*. The whole is inclosed within a casing of refractory material *m*, leaving sufficient space between it and the column for the passage of the gases for heating the apparatus. These gases are produced in a furnace *n*, placed outside the column. This furnace is fed by coke or charcoal and is supplied with air by a blower *o*. The hot gases proceed by a vertical flue *p* to the top of the casing inclosing the column. In their passage a second blower *q* injects cold air into them in a regulated quantity, so that their temperature at the top of the column is from 600° to 700° centigrade. The gases redescend in contact with the cast-iron column and produce the evaporation of the water, whereby the greater portion of their heat is abstracted. About the middle of the column the gases are not more than 300° centigrade. The small column *h* annexed should not be higher than this point. As from other reasons the distillation of the rest of the glycerin, which is in very small quantities, requires very little heat, the temperature remains practically the same till it escapes by the lower flue.

The vinasses being watery and admitted to the vacuum, the water contained is distilled at 40° centigrade, and in consequence until all the water is distilled the temperature of the vinasses scarcely rises above this, in spite of the gases outside being at a temperature of 600° or 700° centigrade, all the heat of these being used up to distil the water. When the water is evaporated, the vinasses commence gradually to grow hotter, and after this time the gases must not rise beyond 300° centigrade, or, as has been explained before, the fluidity of the residue of the vinasses will be altered. The quantity and the temperature of the hot gases must, therefore, be regulated in such a way that their temperature is lowered

to 300° centigrade when the vinasses have lost all its water. This is why two blowers are used, of which the discharge can be regulated each independently of the other.

The steam produced inside and at the top of the column exhausted by the suction-pump is required to descend, which it does by following the path of the helix, taking up a gyratory movement, which by the centrifugal force developed rids it of all the particles which are thrown against the wall where the distillation is completed, while descending the steam becomes superheated and helps in the distillation of the glycerin in the anhydrous portions.

Let us now follow an operation. The vinasses previously concentrated has some acid added to it, so as to have an acidity corresponding to 4° or 5° of  $H_2SO_4$ . Then it is introduced into the top of the column, which has first of all been brought up to the proper temperature by heating. The vinasses spread in a film and submitted to heat in a vacuum undergoes a violent ebullition. It descends the length of the walls and loses first of all, as has been explained, its water, and then, becoming superheated, its glycerin. The steam in the upper portion, which contains particles in suspension, enters the interior helix, the particles under the action of centrifugal force are projected against the wall and further distilled, and, becoming heated, surround and draw the escaped glycerin first into the accessory column *h*, where it undergoes a fresh purification, then into the condenser, where it again becomes liquid. The residuary gases after having been drawn through the suction-pump are washed to rid them of any nitrogenous material they may contain, and then they escape into the atmosphere. The substances deposited in the accessory column undergo, as I have already described, a fresh distillation, and the glycerin they distil merges into the glycerin-vapor from the principal column, which vapor passes through the accessory column. The substances deprived of glycerin flow into the bottom chamber, from whence by means of successive movements of the stop-cocks they are withdrawn at intervals. Thus by this apparatus is obtained, first, a mixture of glycerin and water of great purity, which has only to be concentrated to obtain glycerin ready for sale; second, a residuary liquid very hygrometric which solidifies in air, retaining all potash and nitrogen, which can be sold as it is for manure, or be redistilled with lime to produce ammonia, methylamin, pyridic tars, or be treated in any other suitable manner.

I claim—

The herein-described process of extracting from vinasses the glycerin contained therein in the form of a mixture of glycerin and water to be concentrated and for producing a resi-



due containing all the ammonia which consists in first rendering the vinasses acid and then causing it to flow in a vacuum along a surface heated to a maximum temperature  
5 of 300° centigrade and causing the steam produced to have a gyratory movement to free it of particles carried with it.

In witness whereof I have hereunto set my hand in presence of two witnesses.

CHARLES SUDRE.

Witnesses:

LOUIS GARDET,  
HERNANDO DE SOTO.