No. 848,903. PATENTED APR. 2, 1907.

H. HIRZEL.

ART OF DISTILLING.

APPLICATION FILED FEB. 1, 1900.

2 SHEETS—SHEET 1.

Fig. 1.

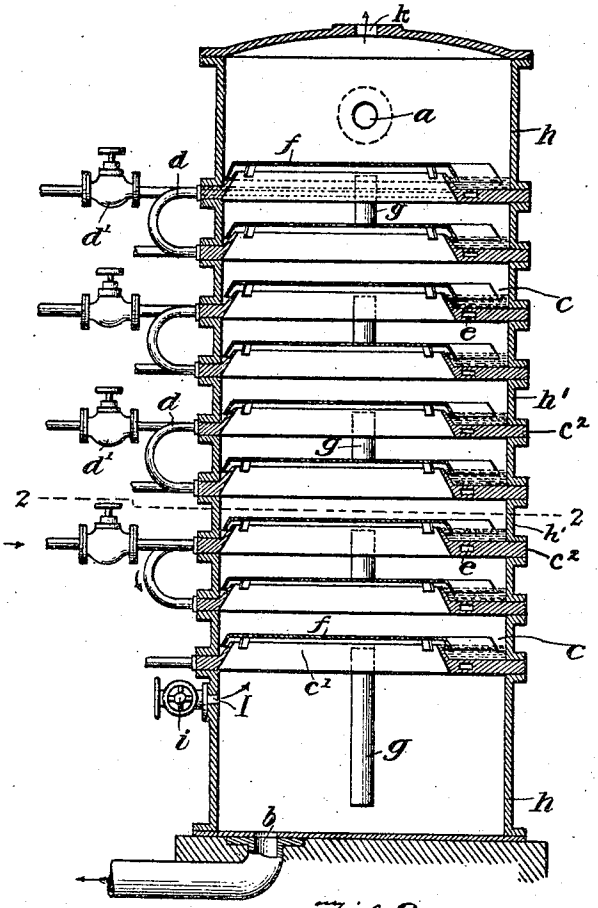
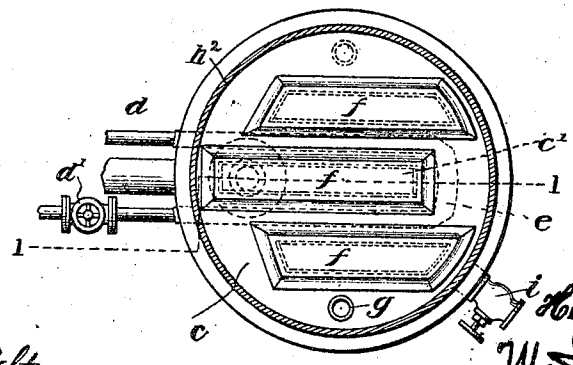


Fig. 2.



Witnesses:
Fenton & Pelt,
Edmund Petersen.

Inventor,
Heinrich Hirzel,
By Wm. H. Pringle
his
Attorney.

No. 848,903. PATENTED APR. 2, 1907.

H. HIRZEL.

ART OF DISTILLING.

APPLICATION FILED FEB. 1, 1900.

2 SHEETS—SHEET 2.

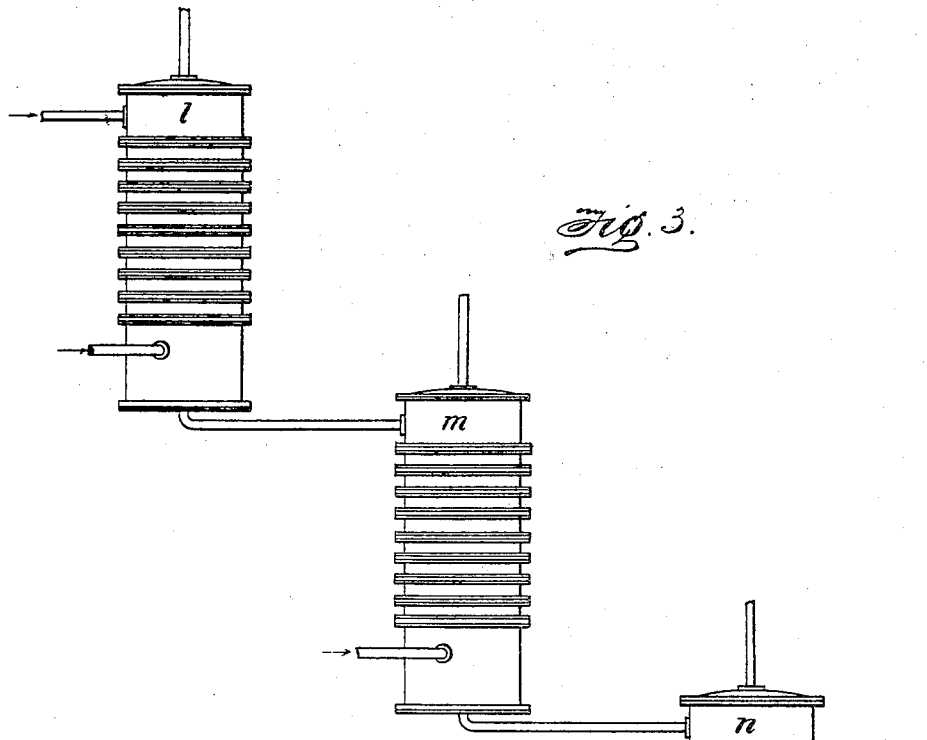


Fig. 3.

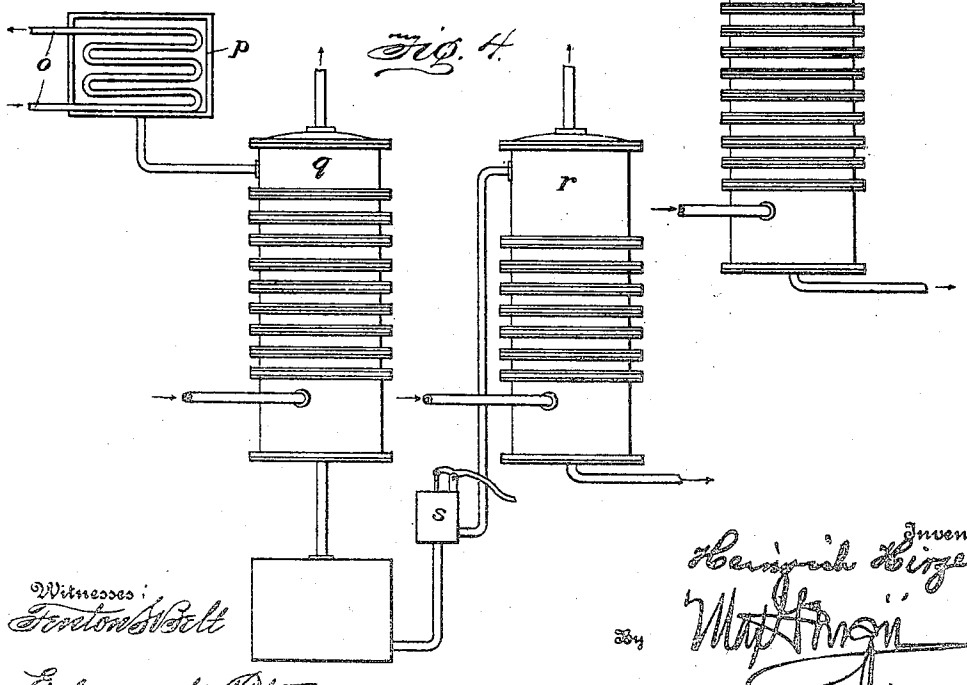


Fig. 4.

Witnesses:
Fenton & Bell
Edmund Pierson.

Inventor:
Heinrich Hirzel,
By
his Attorneys.

UNITED STATES PATENT OFFICE.

HEINRICH HIRZEL, OF LEIPZIG-PLAGWITZ, GERMANY.

ART OF DISTILLING.

No. 848,903. Specification of Letters Patent. Patented April 2, 1907.

Application filed February 1, 1900. Serial No. 3,610.

To all whom it may concern:

Be it known that I, HEINRICH HIRZEL, a citizen of the Republic of Switzerland, residing at Leipzig-Plagwitz, in the Empire of Germany, have invented certain new and useful Improvements in the Art of Distilling; 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.

The present invention relates to the art of distilling mineral oils and products—such, for example, as petroleum, tar, and the like; and the object of the same is to carry out this distilling process free from danger and in a continuous manner, the mineral product being treated in column apparatus and being exposed at all points to the action of heated air or indifferent gases or vapors.

In the process and apparatus covered in my application for Letters Patent of the United States, Serial No. 637,226, filed May 19, 1897, the gases or liquids are caused to pass through a column apparatus, while at the same time dry steam is carried from the bottom to the top of the column and brought into direct contact with the gases or liquids to be treated. Since under this treatment it is unavoidable that water of condensation is formed and caused to mix with the petroleum or tar distillation products or other distillates, by this means the distillates cannot be obtained free from water, so that it is often very difficult, particularly in the case of petroleum lubricating-oils, heavy tar-oils, &c., to separate these distillates from the water which they contain. Another disturbing factor is the fact that the residual tar products, which flow out of the column, are always more or less mixed with water in spite of their high temperature, for the reason that fine bubbles or globules of water are forced out of the steam into the thick tar and having once penetrated the same cannot be eliminated. Such water-containing tar is unsuitable for many uses. The same difficulty takes place in the distillation of petroleum, in which after distilling off the heavier oils a residue containing water is left, such residue not being capable of being further utilized and treated on account of its percentage of water.

The present invention, which is designed to remove the above disadvantages, consists in passing heated air into the heating-columns in lieu of steam in those cases where no danger of ignition or explosion exists and in other cases in passing heated indifferent inert gases (that is, gases which have no chemical combining action on the material being treated) through such columns, such heated air or gases acting similarly to the steam without, however, producing a residue from the distillation containing water. The air or gases simply escape from the distilling apparatus after the distillate has been condensed and collected by the cooler and, if desired, pass to a gasometer or tank, so as to be available for repeated use.

My invention, moreover, consists in such other features as will be hereinafter described, and pointed out in the claims.

In carrying my invention into effect a column apparatus is employed which is distinguished from the apparatus disclosed in my former application by the fact that the tray-bottoms themselves are provided with heating channels or passages instead of arranging heating-coils in the trays. This arrangement enables me to heat the column more uniformly, since through it the tray-bottoms, and therefore the liquids flowing over the same, are more rapidly and uniformly heated, the heating-surface of such tray-bottoms being considerably larger than that of a steam-coil and the absence of the coil permitting the liquids to pass through the trays in much thinner sheets, because it is not necessary to cover any steam-coils. This is a matter of considerable importance and advantage in many cases where it is desired to heat rapidly and to a high temperature. Moreover, since the several column-trays may be made more shallow more of such trays may be arranged in a column apparatus of the same height, hence making the same far more effective than the former forms of column apparatus.

In the accompanying drawings I have represented a column apparatus embodying the preferred form of carrying out my invention.

In the drawings, Figure 1 represents a vertical central section of such column apparatus, and Fig. 2 a horizontal section of the same. Fig. 3 is a diagrammatical view of a petroleum-distilling apparatus, illustrating one application of my invention; and Fig. 4, a similar view of an appartaus embodying my invention as applied to the distillation of tar.

In the drawings, *a* is the feed-orifice for the mineral substance to be distilled, and I is the

inlet for the heated gases to be used in the distillation, while *i* is a valve or cock which regulates the flow of the said heating-gases and by which the same may be admitted to or cut off from the apparatus.

The column apparatus *h* is provided at suitable intervals with the trays or troughs *c*, formed of a bottom piece c^2, and the corresponding ring *h'* of the column, which troughs are in turn provided with heating channels or chambers *e*, (shown in section in Fig. 2,) which communicate with the steam-pipes *d*, which pipes are controlled by the cut-off valve *d'*, by which the steam may be admitted to or cut off from the heating chambers or channels *e*. The trays *c* are each provided with openings or throats *c'*, as illustrated by Figs. 1 and 2, the edges of the trays being flanged upward around said openings, as will be clear from the drawings. Over these openings are placed hoods *f* in the nature of baffle-plates, whereby the vapors, gases, or air may pass upward through the respective openings or throats in the trays and be deflected downward into intimate contact with or through the liquid in the corresponding trays. The liquid proceeds from one tray to the other step by step by means of the overflow-pipes *g*, as shown, and the column residue passes out of the column through the exit-pipe *b* in the bottom of the same, while the hot air or indifferent gases introduced at I escape through the opening *k* in the roof or dome of the column, carrying with them the distillates.

My new process offers peculiar advantages when employing a system of columns heated to different temperatures for the distillation of petroleum, as shown in Fig. 3. Thereby a continuous distillation and one which is entirely free from danger is obtained. The petroleum to be treated under this process is first led into a distilling-column 1, which is not directly heated by flame, but indirectly by steam led through suitable coils, whereby the volatile constituents of the same—the "benzin," so called—are driven off. Thereafter the petroleum, which has been freed of its most dangerous and explosive constituent—the benzin—is carried into a distilling-column *m* of the type shown in Fig. 1, with separate heating-trays, which will permit a heating of the oil to a temperature of from 150° to 160° centigrade, while at the same time a moderate amount of heated air or gases pass through the column from below—that is to say, an amount of air or gases sufficient to produce enough air-current in the column to enable the said air or gases to pass from the bottom to the top of the column. The temperature of the said heated air or gas is preferably about the same temperature as the column. The passage of these gases through this column causes that fraction to be distilled off, which under ordinary distilling processes over a free flame or fire will only boil at a much higher temperature, (between 150° and 300° centigrade.) This step furnishes illuminating petroleum or kerosene, while out of the bottom of the column there passes off a residue containing the heavy oils and the paraffins. The lamp petroleum or kerosene driven from the column in this manner is distinguished by the fact that it requires much less sulfuric acid for refining than the product hitherto distilled from the alembic, and in some cases it even enables one to dispense with the use of sulfuric acid entirely for this purpose. The distillate is, moreover, distinguished by its feebler and mild odor, its light color, and particularly by the fact that it is entirely free from paraffin, a result which has hitherto been impossible to attain in the distillation of paraffin-containing petroleums. The residue which, as already stated, contained the heavy oils (lubricating-oils) and some paraffin after it drains from the column, heated to about 150° to 160° centigrade, is now carried into a second column *n*, of the type shown in Fig. 1, heated to from 175° to 180° centigrade. Under these conditions those oils and products are driven off which under ordinary distilling operations from alembics and kettles do not distil off from below 300° to 360° centigrade. These distillates contain the lubricating-oils and paraffins, and these products when obtained from my column under my process are distinguished by much greater purity than those obtained heretofore and may therefore be manufactured into products of a very high grade. Moreover, greater yield of these products is obtained and also a better, completely-undecomposed petroleum residue which is exceedingly well adapted for heating purposes and for the manufacture of oil or naphtha gas. For the distillation of tar I also make use of several column apparatus of the kind hereinbefore described, as shown in Fig. 4—that is, a column apparatus having heated tray-bottoms through which heated air or gases are carried in the amounts required. The tar for this purpose is preferably subjected to a preliminary heating or warming—as, for example, by a steam-coil *o*, placed in a reservoir *p*, containing said tar—and is then passed through a distilling-column *q*, like that shown in Fig. 1, which is substantially the same as used for the distillation of crude benzene or benzol out of the wash-oil which has been saturated with the same. In this column apparatus *q* the tar is heated to from 108° to 115° centigrade, and from it is obtained as distillates the fraction usually designated as "light oils" in good condition. The residual tar which drains out of this column and which has been deprived of its light oils in the above manner is now forced into a second column *s*, which is also like that shown in Fig. 1, by means of a

pump *s* and while in a heated condition. In this second column apparatus the trays are heated to a higher temperature. Here also the heated air or gases circulate through the column apparatus from the bottom to the top, their heating capacity being such that the tar which circulates from the top to the bottom is heated to from 150° to 160° centigrade. The fraction distilled from this column contains the so-called "middle" oils—that is to say, those ingredients which in ordinary distillation will not pass off at a temperature lower than from 200° to 300° centigrade. This fraction is distilled off in a perfectly pure condition and without any admixture of oils having a high boiling-point, such as anthracene and the like. The separation of the middle oils from the heavy oils, which, as indicated above, do not distil off at a column temperature of from 150° to 160° centigrade, is surprisingly sharp and well defined. Moreover, no products of decomposition are formed, so that from this distillate the various ingredients—such as, for example, carbolic acid and the same—may be obtained in a pure condition far more readily than with a corresponding distillate of a distilling alembic heated over a fire or by direct heat. By my invention one is also enabled to obtain the light oils and middle oils together as one fraction if the tar is distilled from a column heated to from 150° to 160° centigrade. After the tar has passed through such a column and the residue, which has been deprived of the light and middle oils, is drained the same contains only the heavy oils, together with pitch. The heavy oils may also be driven off by means of a column having heating-coils of sufficient heating effect in the trays. For this purpose the tar as it passes is heated to from 175° to 180° centigrade, steam of about ten atmospheres pressure being employed for heating the column-trays. The distillate from a column heated in this manner will contain such oils and products as would only be distilled off at temperatures between 300° and 360° centigrade under ordinary forms of distillation over a direct fire or flame. This column distillate is again free from products of decomposition, and therefore much richer in valuable ingredients—such, for example, as anthracene and the like.

The employment of a combination of individually-heated column-trays and air or indifferent gases in contact with the substance to be distilled is entirely new in its application to column apparatus for tar distillation. Under this process every variety of air may be distilled by the continuous process without the employment of direct fire or flame, and it enables one to separate different fractions of distillation for a further treatment and to collect the same.

A very important advantage flowing from the application of this new invention to the distillation of tar resides in the fact that from fifteen to twenty per cent. more distillate is obtained from the same than in the ordinary distilling processes or with a free fire or flame and that the yield of the different products is therefore much higher. Moreover, these products, as already stated, may separate much more readily and in a purer condition, for the reason that no decomposition occurs by this mode of distillation. Therefore these products are obtained in the best condition possible with less difficulty and expenditure of time and purifying means than hitherto. Where the tar distillation is carried on in connection with coke-furnaces, the gases from the coke-furnaces may be directly employed as the inert gases to be passed upward through the column. This involves considerable economy, and, moreover, these gases are particularly adapted to the distillation of tar.

A further advantage of the new distilling process for mineral oils, such as petroleum, as well as for tar in the same, is that it may be carried on with perfect safety, there being no danger of combustion or explosion.

It is to be understood that the two examples of distillation of petroleum and tar are recited merely as examples of the application of the new invention and that the same is not by any means confined to these substances; but it may also be employed for the distillation of the most varied kinds of liquids and distillable substances—such, for example, for the regeneration of the wash-oil from the benzene or benzol plants which have become unfit for use.

Having thus described my invention, what I claim as new, and desire to secure by Letters Patent, is—

1. The process of continuous distillation, which consists in separating the material to be distilled into a plurality of portions located one above the other, heating the portions to a temperature approaching but below the boiling-point of the product which is to be obtained, and permitting a heated gas to flow upward into contact with the different portions *seriatim*.

2. The process of continuous distillation, which consists in separating the material to be distilled into portions located one above the other, heating said portions to a temperature approaching but below the boiling-point of the product which is to be obtained, leading a heated gas through the various portions *seriatim*, whereby the fraction is carried off with the gas, and then cooling said gas and fraction, whereby the two are separated.

3. The process of continuously distilling petroleum and the like which consists in passing such petroleum through a heated column apparatus and concurrently passing a heated gas through the apparatus in the

opposite direction to remove and to recover benzin; and passing the treated petroleum through a second and uniformly-heated column apparatus of a temperature in the neighborhood of 150° to 160° centigrade against an opposed current of heated gas to remove and recover kerosene.

4. The process of continuously distilling petroleum and the like which consists in passing such petroleum through a heated column apparatus and concurrently passing a heated gas in the opposed direction to remove benzin; passing the treated petroleum through a second and uniformly-heated column apparatus of a temperature in the neighborhood of 150° to 160° centigrade against an opposed current of heated gas to remove and recover kerosene, and finally passing the treated petroleum through a third and uniformly-heated column apparatus of a temperature in the neighborhood of 175° to 180° centigrade against an opposed current of heated gas to remove and recover lubricating-oils.

5. The process of continuously distilling oily bodies which consists in passing such oily bodies consecutively through a series of column apparatus, each uniformly heated and certain members of the series being respectively of a temperature in the neighborhood of 108° to 115° centigrade, in the neighborhood of 150° to 160° and in the neighborhood of 175° to 180° centigrade and at the same time bringing into contact with the oily body in each apparatus an opposed current of heated gas to remove and recover the bodies volatile at the temperature prevailing therein.

6. The process of continuously distilling oily bodies which consists in preliminarily heating such oily body, and passing the same consecutively through a series of column apparatus, each uniformly heated, and certain members of the series being respectively of a temperature in the neighborhood of 108° to 115° centigrade, in the neighborhood of 150° to 160° centigrade and in the neighborhood of 175° to 180° centigrade and at the same time bring into contact with the oily body in each such apparatus an opposed current of heated gas to remove and recover the bodies volatile at the temperature prevailing therein.

7. The process of recovering fractions from a composite oily body which consists in heating it in films to a temperature approaching, but below its boiling-point, stripping it of constituents of high vapor tension at such temperature by means of a gas-current, and repeating the treatment at a series of successively-higher temperatures.

8. The process of recovering fractions from petroleum which consists in heating films of petroleum to a temperature near but below the boiling-point of such petroleum, and stripping it of constituents having a high vapor tension at such temperature by means of a gas-current.

9. The process of recovering fractions from petroleum which consists in heating petroleum in films to a temperature near but below the boiling-point of gasolene; stripping it of gasolene by means of a gas-current; again heating it in films to a temperature near but below the boiling-point of kerosene, stripping it of kerosene by means of a gas-current; and again heating it in films to a temperature near but below the boiling-point of lubricating-oils, and stripping it of such lubricating-oils by means of a gas-current.

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

HEINRICH HIRZEL.

Witnesses:
OSWIN HELBING,
RUDOLPH FRICKE.