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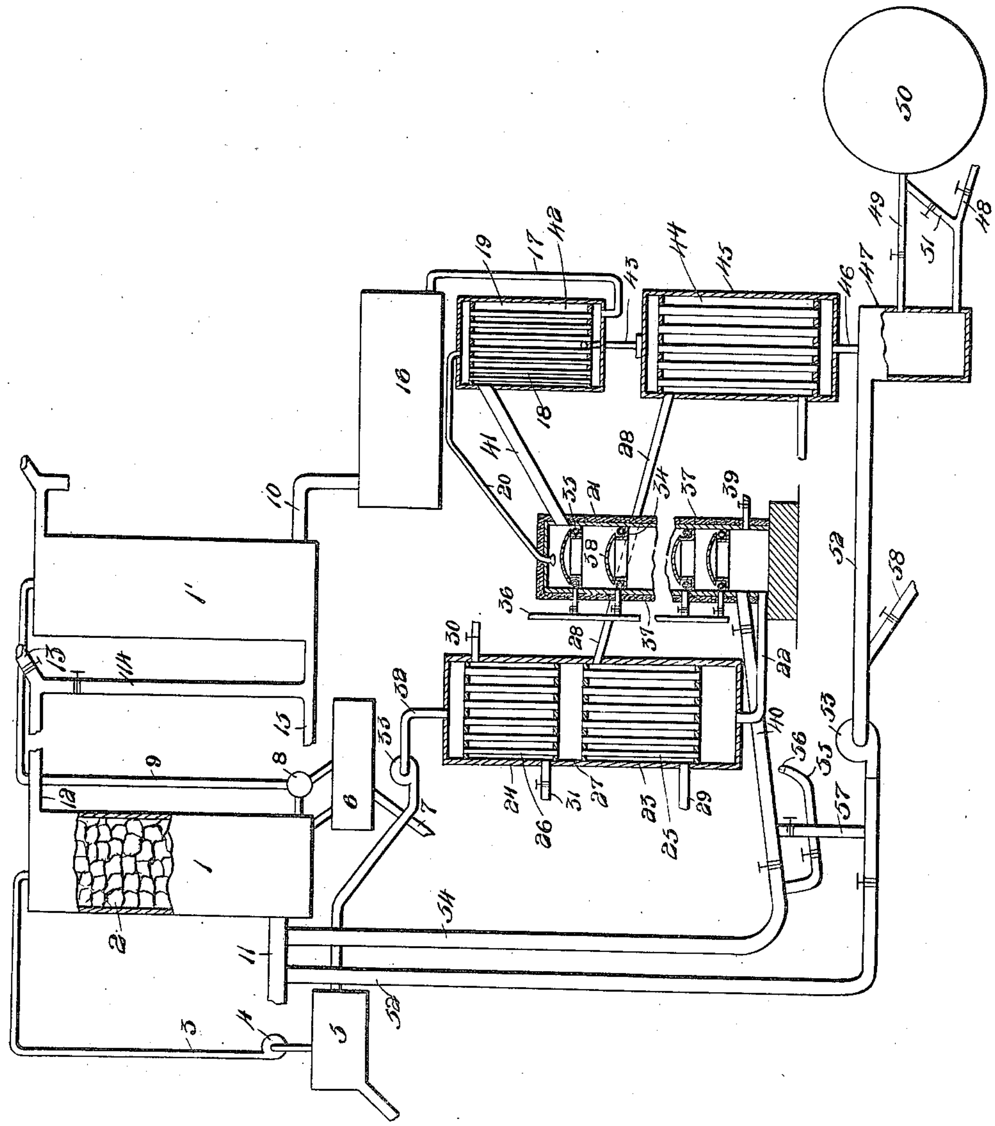
DISTILLING APPARATUS.

APPLICATION FILED MAR. 28, 1907.

991,205. Patented May 2, 1911.



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DISTILLING APPARATUS.

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To all whom it may concern:

Be it known that I, Heinrich Hirzel, a citizen of the German Empire, residing at Leipzig-Plagwitz, Saxony, Germany, have invented certain new and useful Improvements in Distilling Apparatus; 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 invention relates to distilling apparatus and consists in improvements in volatilizing stills and in certain organizations of apparatus comprising such stills; all as more fully hereinafter set forth and claimed.

Ordinary methods of fractional distillation, or the separation of the components of mixing liquids, are based on the differences in the boiling points of such components, and usually consist of a series of regulated and methodical boilings of the mixed liquids at different temperatures, whereby the lower boiling component is boiled away from the higher boiling. The well known column still, used for separating mixtures of alcohol and water, is constructed on this principle and, broadly stated, consists of an air cooled metal shell having heating means at its base and a series of liquid holding devices along its height. The balancing effect between the base heating and the cooling due to the skin cooling of the shell and the introduction of relatively cool liquid at the top causes a regular diminution of heat upward, and by introducing a mixed liquid at the top of the column and causing it to travel successively through the liquid holding devices, it is boiled at a series of successively increasing temperatures. The vapors from the boiling liquid in each liquid holding device serve to boil the liquid in the device next above, condensing therein and thereby most of their high-boiling constituent, and adding the vapor of their low boiling constituent to that which is there evolved. This method and apparatus while sufficiently successful with two-component liquids, like alcohol and water, where the boiling points are neither too far apart nor too near each other, are not well adapted to complex mixtures like petroleum, containing many components.

The boiling point of a mixture of mutually soluble liquids while perfectly definite with a given mixture under given conditions, is nevertheless a very complex phenomenon, depending not only upon the respective boiling points of its components and their relative proportions, but also upon many other factors, such as their vapor tension at different temperatures, their mutual affinities and solubilities in liquid and vaporized states, the pressure and composition of the atmosphere in the apparatus and so on. As conditions change, these factors change quite independently of each other and with complex mixtures there is no simple relation between the compositions of the vapors evolved in boiling at different temperatures; or, in other words, there is no certainty as to the result which will be obtained in running such a mixture through a column still. Using such a simple still upon petroleum, for instance, the fraction boiled off at a temperature which should be that of boiling gasolene will be found to contain the very high boiling lubricating oils. In one experiment, beginning with a number of barrels of petroleum and fractionating by boiling and re-fractionating the fractions in the same manner a number of times, it has been found that not even a pint of liquid of constant boiling point and composition could be attained.

In the present invention, distillation conditions are simplified and results correspondingly improved by departing altogether from the common practice and relying solely upon differences in vapor tension instead of differences in boiling points; in other words, upon one factor in lieu of upon a complex of factors. The liquid to be treated is converted into a large area film, and by suitable arrangement of heating elements, the film thus formed is uniformly heated to a temperature approaching but below the boiling point of the component to be removed; that is, near its point of maximum vapor tension. This film is treated with a heated current of gas or vapor into which said component volatilizes and from which it may be again removed by condensation. The operation is, so to speak, an evaporation in lieu of the boiling of ordinary fractional distillation methods, and with the type of apparatus employed in the present invention, the temperature throughout the distilling device is substantially uniform. This uniformity of temperature is in marked contrast to the conditions existing in the ordinary column still where the temperature varies from a minimum at the top to a maximum at the base. The gas used to sweep out the component volatilized from the liquid undergoing distillation is suitably treated in a condenser to recover the valuable volatile product and is then returned to the distilling device and the process repeated. Apparatus working upon this principle may be advantageously used in a variety of arts, but the type herein described is particularly adapted to the manufacture of benzol.

Crude benzol is a trade term for a mixture of bodies contained in and removed from coal gas, this mixture as it is recovered and brought upon the market containing not only the chemical individual known as "benzene" or "benzol," (C_6H_6) but also a wide variety of other substances, mostly of the aromatic series but partly also of the aliphatic series, having very different boiling points, ranging from that of the chemical "benzol", which is about 80° C., to that of naphthalene, 218° C. There is even some anthracene, boiling point 360° C. The boiling point of the commercial mixture, while varying with its source, composition and method of extraction, generally ranges, with the grades hereinafter contemplated, above 140° C. But though having this relatively high boiling point, the crude benzol is nevertheless very volatile, all its components having the common characteristic of a very high vapor tension; a characteristic to which is due their presence in the vapor state in coal gas. Crude benzol is valuable commercially as a source of the true benzol, of xylol, toluol and other cyclic or aromatic hydrocarbons used in making anilin dyes, synthetic medicines and other products.

In ordinary gas making methods, the gas is cooled and whatever will condense is removed with the coal tar. But because of the stated high vapor tension of its components, much or most of the crude benzol passes the cooling condensers and remains dissolved in or admixed with the gas in vapor form. It is estimated that of the C_6H_6 component, often ten times as much remains with the gas as is removed in the condensers. In the manufacture and use of ordinary lighting gas, it is generally deemed desirable to allow the crude benzol passing the condensers to remain in the gas as it contributes materially to luminosity, though it is sometimes removed and the commercially less valuable petroleum hydrocarbons substituted. There is, however, an abundance of gas of the same character made as a by-product in coking coal, in which luminosity is of no importance, and the extraction of crude benzol from this is desirable. It is this gas which is particularly contemplated in the hereinafter described apparatus for obtaining crude benzol.

Heretofore in the art it has been proposed to remove the crude benzol by washing the gas with high boiling tar oils, or similar oils and recover the benzol by a subsequent boiling of the charged wash oil. These methods have not been particularly successful, for a number of reasons. As is usual in all washing operations the amount of solvent employed is disproportionately great compared with the amount of dissolved matter extracted; *i. e.*, the charged wash oil is rather a weak solution of the crude benzol. Its initial boiling point is far above that of the crude benzol itself and of course steadily increases on boiling, so that in such a boiling distillation the final temperature is generally high enough to damage the wash oil and contaminate the benzol recovered both with wash oil constituents and with products of destructive change of the wash oil.

By using volatilizing stills of the character described the charged wash oil may be very conveniently treated for the removal of its contained crude benzol. Maintained at a temperature near but below the boiling point of crude benzol, say eight or ten centigrade degrees below, it is not injured in character and may be re-used indefinitely for extracting more benzol. It is in fact better upon such re-use than when fresh since whatever volatile impurities it may have originally contained are removed in the first distillation or so, and it thereafter yields nothing to contaminate the benzol. With the volatilizing still employed for this treatment may be usefully combined apparatus for recharging the wash oil treated in it and returning the same for renewed distillation, making a convenient organization of apparatus for the cyclic treatment of such wash oil.

In the accompanying drawings are illustrated sundry of the many types of apparatus within the limits of my broad invention.

In this illustration: Figure 1 shows, partly in elevation and partly in vertical section, the apparatus as applied to the extraction and recovery of crude benzol; and Fig. 2 shows a similar but modified apparatus applied to the treatment of mixed volatile liquids, like petroleum.

In Fig. 1 the reference characters 1 and 1′ designate two similar gas washing towers, intermediate connections being shown broken away to indicate that intermediate like towers may be intercalated if desired. In practice, the series will frequently have more than two towers. Internally the towers are provided with any suitable means for filming wash-oil and securing intimate contact with the gas to be washed. As shown, the filming means is the crushed coke 2, but plates and trays or other means may be substituted if desired. Down and over the coke, wash oil is showered, it being led to the top of the tower (1) by pipe 3, provided with pump 4, from the storage tank 5. From the base of the tower the charged wash oil flows into tank 6, whence it may be led by outlet pipe 7 for distillation, or, preferably, as shown, by pump 8 and pipe 9 it may be taken to the top of the next succeeding tower (1′), sent therethrough and drawn off by pipe 10.

Gas is passed through the first tower in the opposed direction to the flow of oil by means of inlet pipe 11 and outlet pipe 12, thoroughly contacting with and being washed by the downflowing oil and giving up its contained benzol thereto. The outlet pipe is provided with a valved connection 13 by which the gas may be led off to any gas using or consuming device (not shown). It is also provided with another connection 14, leading into the base of the next tower in series. This connection is further provided with an inlet 15, by which fresh gas may be directly introduced into the second tower. By this arrangement, it is obvious that the successive towers may be used in parallel or in series, either for wash oil or for gas. Preferably the wash oil connections are used in series and the gas connections in parallel; fresh gas being introduced into each tower and the wash oil going through two or more towers in succession. As many towers may be used as are deemed necessary, though for the sake of simplicity of illustration but two are shown.

From the end tower of the series, where the wash oil is sent through a plurality of towers successively, the charged oil flows into a storage tank 16. From this it flows through pipe 17 to and through the pipes 18 of the heat interchanger 19, where it is heated by vapors from the still, and thence through 20 to still 21. In the still it is stripped of its volatile constituents, or crude benzol, which it has taken up from the gas in the towers. From the still it is taken at the base by pipe 22 in a heated condition. As it must be cool or cold for reuse in the towers, this pipe communicates with a cooler 23, and chiller 24, provided respectively with tube nests 25 and 26. As shown, cooler and chiller are in one structure, being separated by diaphragm 27. The cooler is provided with an inlet 28 and an outlet 29 for cooling fluid; the chiller has similar elements, 30 and 31, for a chilling fluid. From the chiller, the wash oil is taken by pipe 32 and pump 33 back to the wash oil tank of the first tower.

The still is of peculiar construction, not being intended as is customary in a column still to be of differential heat from bottom to top, but uniformly heated throughout. This is for the reason that it is not desired to fractionate the liquid treated; as is usually done, by a series of successive boilings, but to remove a single desired constituent by exposing such liquid in a large area film heated throughout to a temperature approaching but below that of the constituent in question to a circulating gas current. For this purpose it is evident that any device capable of producing such a heated large area film could be used, but an apparatus built generally like a column still is convenient. Since, however, differential heating of different portions of the liquid is not required, substantial uniformity in temperature throughout the extent of the film being desired, the still should be correspondingly modified.

As shown the still is internally provided with a series of annular trays, 34, each provided with a comparatively large steam coil 35. All the coils communicate with a valved header 36, outside the still, so that they may be independently supplied. The specific heat of the wash oil, however, being low, the coils may be arranged to act successively, though not so shown, passing the same portion of steam through all. Since skin cooling and differential heating are not desirable here, as in the ordinary type of column stills, the still is preferably lagged by heat insulating material 37 to assist in maintaining uniformity of temperature. Depending into each annular tray is a cap 38, spaced a little above to allow flow. Through the still, upwardly against the descending filmed liquid is sent a current of heated indifferent gas to strip it of volatile constituents. This gas may be steam from inlet 39, or another indifferent gas from inlet 40. At the temperature at which the benzol still is ordinarily run, about 130° C., steam is a gas.

The gas, carrying the volatilized benzol, leaves the still through vapor pipe 41, circulates in the space 42, around the tube nest of the heat interchanger, thereby warming inflowing wash oil and itself being cooled, descends thence through pipe 43 to and through the tube nest 44 of condenser 45, where it is cooled by circulating water. The water leaving the condenser as shown, is used for the wash oil cooler (23), a pipe connection (28) being provided for that purpose. From the condenser, condensed liquids flow through pipe 46 into tank 47. If steam has been used as the gas, condensed water accumulates in the bottom of this tank and is removed from time to time through valved outlet pipe 48, benzol being drawn off through valved pipe 49 and sent to a storage tank 50. If another gas has been used, water of course does not accumulate in material amount, and the benzol may be tapped off through the water pipe, a valved connection 51 being provided for that purpose. Using such other gas, it is withdrawn from the benzol tank by means of pipe 52 and fan 53.

Lighting gas, such as is washed in the

towers, is a convenient carrier. In its employment, the inlet pipe of the first tower may be tapped by valved pipe 54 and gas sent direct into the bottom of the still, being heated on the way, as by the branch pipe 55 ending in gas jet 56. The gas fed to the washing towers is of course saturated with crude benzol at the temperature at which it enters, but by heating, as in the manner shown, it is enabled to take up more benzol and thereby serve to remove the same from the wash oil in the still. This excess it again deposits when passed through the condenser. The fan (53) serves to maintain circulation through the system. The gas withdrawn by it from the benzol tank may be returned through 52 to the tower inlet, as shown. In case other gas is desirable, such as air or products of combustion, producer gas, etc., it may be used with the same apparatus and kept circulating cyclically. For this purpose the two gas pipes (52 and 54) are provided with a valved bridging pipe 57, located on the pressure side of the fan. Auxiliary inlet pipe 58 provides for introducing a supply of such gas and for regulation of pressures within the system.

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

1. In a benzol recovery plant, gas washing means for passing wash oil filmwise against a current of gas, means for transmitting effluent wash oil in a uniformly heated film past a current of a heated gaseous carrier, means for condensing out benzol absorbed by the carrier and returning said carrier to serve anew, means for chilling said oil, and means for returning the chilled oil to the original gas washing means.

2. In a benzol recovery plant, means for cyclically passing wash oil in a chilled film through a gas washer, in a reheated film through a uniformly heated still past a gaseous carrier cyclically circulating over said film and through a condenser, and back through chilling means to the gas washer.

3. In a benzol recovery plant, means for cyclically passing wash oil, alternately chilled and heated, through a gas washer and a still, said still comprising means for distributing said wash oil into a large area film of uniform heat and being provided with means for circulating through and over said film a heated gaseous carrier.

4. In a benzol recovery plant, means for cyclically passing wash oil, alternately chilled and heated, through a gas washer and a still, said still being a heat insulated column still provided with internal film-forcing devices in liquid communication and with heating means throughout its height and also provided with means for cyclically circulating a gaseous carrier through the still, a condenser, a reheater and back to the still.

5. In a benzol recovery plant, means for cyclically passing wash oil, alternately chilled and heated, through a gas washer and a still, said still being provided with film forming means and uniformly heated throughout, and means for circulating a portion of gas abstracted from the inlet to the gas washer over the so-formed film, through a condenser and back to said inlet.

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

HEINRICH HIRZEL.

Witnesses:
RUDOLPH FRICKE,
BRUNO RIMPLER.