This invention relates to a process and apparatus for distilling liquids and more particularly to an apparatus for distilling a mixture of liquids which contains a large number of constituents having boiling points extending over a wide range of temperatures and varying gradually, or by slight steps, from those of its lowest boiling to those of its highest boiling constituents.

Mixtures of this type occur in crude petroleum in the product resulting from the distillation of heavy hydrocarbon oils, and in other processes in which carbonaceous materials are decomposed by heat. These mixtures do not have a definite, constant boiling point but begin to boil at about the boiling temperature of the lightest, most volatile members and continue to boil with a gradually increasing temperature until the highest boiling members are vaporized. The vapor given off from the boiling mixture at any particular time does not consist of but one constituent but comprises a mixture of vapors which contains at first only the lower boiling members of the mixture, but as the boiling temperature rises contains gradually increasing proportions of heavier and smaller proportions of low boiling constituents until it finally consists principally of only the highest boiling members of the original mixture. Mixtures of this character may be separated into fractions upon distillation by continually condensing the vapors given off by the mixture and separately collecting the condensates obtained within definite boiling points. Thus, in the distillation of petroleum, the vapors may be separated into crude gasoline, naphtha, kerosene, and burning oils or lubricating stock. Each of these fractions will, however, contain members from adjacent groups and will require one or more redistillations to free it from these products and to produce a product of definite boiling point and having a constant composition and definite properties. Each redistillation, also, requires a rehandling of the crude distillates, causing a loss of material through leakage and other wastes and consuming an additional quantity of heat.

The primary object of the present invention is to provide a process by which a mixture of liquids of different boiling points are separated in one operation into a series of fractions having definite narrow boiling point limits and substantially free from substances having boiling points outside these limits.

Another object of the present invention is to provide an apparatus in which a mixture of liquids of different boiling points may be separated in one operation into a series of fractions having definite narrow boiling point limits and substantially free of substances having boiling points outside these limits.

A further object of the invention is to provide a process apparatus in which an accurate and flexible control of the temperature throughout the apparatus may be obtained.

With these and other objects in view, the invention consists in the process and apparatus described in the following specification and defined in the claims.

The various features of the invention illustrated in the accompanying drawing, which shows a vertical section of a still embodying the preferred form of the invention.

The present invention constitutes a continuation in part of my pending application, Serial No. 211,557, filed January 12th, 1918.

In the present invention, oil or other liquid to be distilled, passes downwardly through the still and forms a series of bodies of liquid, arranged alternately with a series of fractional condensers. Each body of liquid is heated to a temperature somewhat higher than that in the body directly above and the downwardly passing stream of oil is heated in this manner to progressively higher temperatures until it reaches a final body of liquid maintained at the highest temperature in the still. The unvaporized oil and residues remaining in the final body of liquid pass through a series of heat interchangers or heat recovery chambers in which they are cooled to progressively lower temperatures by an interchange of heat with a heat-transferring gas passing countercurrent to the passage of the liquid residues. The heat transferring gas is heated by contact with the hot residues and is used to heat the fresh liquid passing downwardly through the
still, thereby returning the waste heat from the liquid residues back to the bodies of liquid undergoing distillation. After being heated by the hot liquid residues, the gas is raised to a still higher temperature by an external source of heat and is brought directly into contact with the lowermost body of liquid and maintains the liquid at the highest temperature in the still. The heavy hydrocarbons of the oil are thereby vaporized and are absorbed by the gas and carried into a condenser immediately above the body of liquid. The condenser is maintained at a temperature slightly less than that maintained in the lowermost body of oil and the vapors carried by the gas are thereupon condensed and removed. The gas continues to pass in this manner alternately through the bodies of oil and through the condensers, being progressively reduced in temperature in its upward passage, contacting each body of oil at successively lower temperatures, absorbing vapors which are progressively more volatile and condensing them in condensers maintained at progressively lower temperatures. The gas passes from the final condenser in which all of the constituents which can be condensed are removed, and is returned to the heat interchangers and recycled through the apparatus.

The condensers arranged alternately between the bodies of oil are maintained at temperatures suitable for fractionally condensing the vapors formed in the next lower vaporizing chamber by means of cooling coils connected in series. Cold liquids, which may consist of cooled residue from the final heat interchanger, is circulated through the cooling coils, passing first into the uppermost, coolest condenser and flowing countercurrent to the heat transferring gases in succession through intermediate condensers at progressively higher temperatures to the final condenser in which it attains a temperature but slightly less than that of the liquid in the hottest vaporizing chamber. From the final condenser the liquid is returned to the heat interchangers in which it imparts its heat to the circulating gas and is cooled to a temperature at which it may be returned to the uppermost condenser.

Referring to the accompanying drawings, crude petroleum or other liquid to be treated is supplied to a distilling apparatus 10 through a supply pipe 12 and enters a distilling chamber 14 near the upper end of the apparatus. Upon passing into the chamber 14, the oil spreads out over the bottom of the chamber and is heated by contact with a heat-transferring gas passing upwardly through the oil in a number of small bubbles uniformly distributed through the area of the chamber. From the chamber 14 the oil flows through an orifice pipe 16 to a lower vaporizing chamber 18 in which it is heated to a somewhat higher temperature by contact with hot gas. In this manner the oil flows in succession through vaporizing chambers 20, 22 and 24, passing from one chamber to the next through overflow pipes 26, 28 and 30. In the lowest vaporizing chamber 24, the oil contacts with gas at the highest temperature, is itself raised to the highest temperature in the still, and the highest boiling constituents are vaporized and absorbed in the circulating gas. From the final vaporizing chamber 24 the oil flows through an outlet pipe 31 to a series of heat interchangers or heat recovery chambers 32, 33, 34 and 35 in which it is cooled to progressively lower temperatures by countercurrent passing current of heat-absorbing gas, the oil passing from one heat interchanger to the next lower one through a series of connecting pipes 36, 37 and 38.

The oil in the final vaporizing chamber 24 is heated by means of hot gas supplied from a chamber 39 directly beneath the chamber 24. The gas is delivered from the chamber 39 to the chamber 24 through an opening 40 in a partition 41 dividing the chambers and is deflected by means of a dome 42 directly above the opening 40 into a space 44 formed between the partition 41 and a perforated plate 46, spaced a short distance above the partition. From the space 44 the gas passes upwardly into the body of liquid in the chamber 24 through a number of openings 48 uniformly distributed throughout the plate 46. In passing upwardly through the oil in the vaporizer 24, the heated gas is brought directly into intimate contact with the oil to be heated and thereby imparts its heat to the oil without establishing a temperature gradient through the liquid such as is required for the transmission of heat by conduction from an outside heating source. This method of heat transmission therefore provides a very efficient and uniform heating of the oil. The temperature of the oil is dependent upon the temperature of the gas and upon the quantity of gas supplied for a given quantity of oil, and may therefore be quickly and uniformly varied by varying the rate of supply of gas. From the vaporizing chamber 24 the gas and the vapors taken up by the gas in passing through the oil pass through an opening 50 into a condensing chamber 52 arranged directly thereabove.

The gases entering the condensing chamber 52 are uniformly distributed throughout the chamber by means of a gas-distributing plate 54, similar to the plate 46, and pass directly into contact with a cooling coil 56 extending throughout the chamber at a short distance above the distributing plate. The temperature of the cooling coil 56 is maintained slightly below that of the vaporizing chamber 24 so that the greater part of the
vapors formed in the chamber 24 will be condensed. Owing to the low heat conductivity of gases and vapors it is difficult to obtain a sufficiently rapid interchange of heat directly between the upwardly passing gases and the cooling coil to obtain a thorough condensation of the vapors carried by a gas. To facilitate the transfer of heat between the cooling medium and the upwardly passing gases, a body of liquid condensate is maintained in contact with the cooling coil and the gas is bubbled through the body of condensate in such a manner as to agitate it and circulate it continuously against the coil.

Through this arrangement a very rapid transfer of heat from the gas to the body of condensates and from the condensate to the cooling coil is obtained. To maintain a body of condensate in contact with the cooling coils, the condenser chamber 52 is provided with an outlet pipe 58 which may be positioned at any desired distance above the bottom of the chamber.

From the condenser 52 the partly cooled gases pass through an opening 60 into the vaporizing chamber 22 and serve to heat the body of liquid in the chamber to a temperature somewhat less than that maintained in the vaporizing chamber 24 and condenser 52. In the vaporizing chamber 22 the gas vaporizes and absorbs constituents of the oil which volatilize at a lower temperature than those produced in the vaporization chamber 24 and at the same time any heavy vapors which may have passed through the condenser 52 are condensed by contact with the liquid in the chamber 22 and subsequently returned to the vaporizing chamber 24. The gas with the absorbed vapors passes from the vaporizing chamber 22 through an opening 62 into a condenser 64 positioned immediately above the chamber. This application has matured into Patent No. 1,472,116 on October 30, 1923. The condenser 64 is similar in construction to the condenser 52 and is provided with a cooling coil 66 and overflow pipe 68. In a similar manner the gas passes from the condenser 64 through an opening 70 into the vaporizing chamber 20 immediately above and through a condenser 74, vaporizing chamber 18, condenser 76, and vaporizing chamber 14 to a final condenser 78. The condensers 74, 76 and 78 are provided with cooling coils 50, 82 and 84 respectively and with overflow pipes 86, 88 and 90 arranged similarly to the condensing coils and overflow pipe in the condenser 52. In the final condenser 78, the gases are cooled to the lowest temperature obtainable and all of the volatile constituents which can be condensed are removed from the circulating gas.

The cooled gas is withdrawn from the chamber 78 through an outlet pipe 92 and is forced by means of a fan 94 to a pipe 96 leading to the heat interchanger 35 at the bottom of the still. The gas entering the heat interchanger 35 spreads out under a perforated plate 98 and passes upwardly through the liquid in the heat interchanger 35 in a series of small uniformly distributed bubbles. In a similar manner the gas from the heat interchanger 35 passes upwardly through a series of openings 100, 102 and 104 connecting the interchangers 35, 34, 33 and 32, to the uppermost heat interchanger 32, being continually heated by successive contacts with progressively warmer liquid. From the uppermost heat interchanger 32, the heated gas passes through an opening 106 into the chamber 39 and is raised to a temperature above that of the lowermost vaporizing chamber 24 by the admission of hot products of combustion from a furnace 108 communicating with the chamber 39 through a flue 110. From the chamber 39, the heated gas is circulated through the vaporizing chambers 24, 22, 20, 18 and 14 in the manner described above. The excess of gas caused by the continuous admission of hot products of combustion to the chamber 36 is permitted to escape from the outlet pipe 93 through an exhaust outlet 112.

The quantity of fuel required to operate the process is reduced to a minimum by the transference of all of the heat of combustion of the fuel burned in the furnace 108 to the oil undergoing treatment and the abstraction of substantially all of this heat by the oil before the excess gas is released to the atmosphere. The fuel, which may be fuel oil or gas, is introduced into the furnace 108 through burners 113 and burned with just sufficient air to supply complete combustion. The heat of combustion of the fuel is set free as sensible heat in the products of combustion and is transferred directly with the hot products of combustion to the gas mixing chamber 39. The products of combustion from the furnace may be incandescent or at a temperature far above that to which the oil undergoing treatment may be heated, but the temperature is reduced by the dissemination of heat throughout the comparatively larger body of gas in the chamber 39 to a temperature suitable for vaporizing oil in the chamber 24. The furnace 108 is placed very close to and in direct communication with the chamber 39, forming, in effect, a part of the chamber.

The oil residues are cooled in the final heat interchanger 35 to approximately the temperature of the cooled gas flowing through the gas inlet pipe 96 and are therefore suitable for use as a cooling medium in the uppermost condenser 78. The cooled residue is therefore withdrawn from the lowermost heat interchanger 35 through an outlet pipe 114 and forced by means of a pump 116 through an inlet pipe 118 to the coil 84 in the uppermost condenser 78, and passes
successively through cooling coils 84, 82, 80, 66 and 56 by means of connecting pipes 120, 122, 124 and 126 respectively. In passing through the cooling coils 84 to 56, the cooling liquid is heated to progressively higher temperatures and finally reaches a temperature but slightly less than that of the oil in the vaporizing chamber 24. From the final cooling coil 56 the heated liquid passes through an outlet pipe 128 to the branch pipe 30 and joins with the liquid passing to the final vaporizing chamber 24. The heated liquid, together with the hot oil from the vaporizing chamber 24, thereupon passes through the vaporizing chamber 24 and enters the heat interchangers 32 to 35 from which it is recirculated through the cooling coils. The volume of liquid passing through the heat interchangers is continuously increased by the addition of fresh oil residues produced from the liquid supplied to the still for treatment. This excess liquid is removed from the circuit through a draw-off pipe 130 connected to the outlet pipe 114.

The rise in temperature of the liquid from its entrance through the pipe 12 until it reaches the final vaporizing chamber 24 is approximately equal to the drop in temperature of the liquid passing from the uppermost heat recovery chamber 32 to the outlet pipe 130. The amount of heat recovered from the residues of distillation alone will, therefore, only suffice to heat a portion of the incoming oil equal to the amount of residue remaining after distillation. The heat required to heat and evaporate the portion of the liquid which is vaporized and condensed in the vaporizing and condensing chambers of the column is supplied to the heating gas partly by abstraction of heat from the cooling liquid passing from the final cooling coil 56 and flowing through the heat recovery chambers with the hot residue, and partly by the heat introduced into the chamber 39 from the furnace 108. To bring about an approximately complete heat balance between the vaporizing and condensing portion of the column and heat recovery compartment of the column, the amount of liquid circulated through the coils 84 to 56 should have a heat capacity equal to the heat of the liquid and the heat of vaporization of the total amount of liquid vaporized and condensed in the column. The proportion of cooling fluid to hot residue will, therefore, vary with the proportionate amount of residue left after distillation and will be smaller as the quantity of residue increases. The quantity of residue withdrawn through the outlet pipe 130 is regulated by means of a control valve 131 until a heat balance is obtained for the particular oil undergoing treatment.

In the above described invention, the exhaust gases and the oil residue leave the apparatus at nearly the same temperature at which they enter. The heat absorbed to supply the latent heat of vaporization in the vaporizing chamber is given up by the condensation of the vapors in the condensing chamber or vaporizing chamber immediately above, and returned to the apparatus. Consequently, there is but little loss of heat outside of radiation losses, which are reduced by suitable insulation, and accordingly but a very small amount of heat need be supplied by the furnace 108. Since the entire heat of combustion of the fuel burned in the furnace is supplied to the apparatus, the heating efficiency and fuel economy are very high.

By dividing the apparatus into a large number of vaporizing and condensing chambers, the number of fractions which may be obtained may be increased and the oil may be divided into any number of fractions of narrow boiling point limits and free from constituents having boiling points outside of these limits. By a proper mixing or blending of the various fractions, a product of any desired boiling point limit and of any desired composition may be obtained.

Having described the invention, what is claimed as new is:

1. A distilling apparatus which comprises a series of alternate vaporizing and condensing chambers, means for passing liquid to be treated successively through said vaporizing chambers cooling means within said condensing chambers, means for circulating a cooling fluid successively through said cooling means in a direction parallel to the flow of said liquid to be treated, a series of heat recovery chambers arranged to receive the liquid from said vaporizing chambers, means for circulating a heat transferring gas successively through said heat recovery chambers, and said series of vaporizing and condensing chambers and means for introducing heated gas into said circulating gas between said heat recovery chambers and said series of vaporizing chambers.

2. A distilling apparatus which comprises a series of alternate vaporizing and condensing chambers, means for passing liquid to be treated successively through said vaporizing chambers, cooling means in said condensing chambers, means for circulating a cooling liquid successively through said cooling means in a direction parallel to the flow of liquid to be treated, a series of heat recovery chambers, means for passing residuum liquid from a vaporizing chamber successively through the heat recovery chambers, means for circulating a heat transferring gas successively through said heat recovery chambers and said series of vaporizing and condensing chambers, a furnace, and means for introducing hot products of combustion from said furnace into said circulating gas between said series.
of heat recovery chambers and said vaporizing chambers.

3. A distilling apparatus which comprises a series of alternate vaporizing and condensing chambers, means for passing a liquid to be treated successively through said vaporizing chambers, cooling means within said condensing chambers, means for circulating a cooling liquid successively through said cooling means at progressively higher temperatures, means for maintaining a body of condensate in said condensing chambers in contact with said cooling means and for withdrawing a portion of said condensates and means for circulating a heated gas successively through the liquid and condensates in said vaporizing and condensing chambers.

4. A distilling apparatus which comprises a series of alternate vaporizing and condensing chambers, means for passing a liquid to be treated successively through said vaporizing chambers, cooling means within said condensing chambers, means for circulating a cooling fluid successively through said cooling means in the same general direction as the flow of liquid through said vaporizing chambers, means for maintaining a body of condensate in said condensing chambers in contact with said cooling means and for withdrawing a portion of said condensates, a series of heat recovery chambers, means for passing residuum liquid from the last vaporizing chamber of the series successively through the heat recovery chambers, means for circulating a heat transferring gas successively through said heat recovery chambers and through said series of vaporizing and condensing chambers, and means for introducing heated gas into said circulating gas between said series of cooling chambers and said vaporizing chambers.

5. An apparatus for treating liquids which comprises a column, a series of horizontal partitions dividing the upper portion of said column into a vertical series of adjacent alternate vaporizing and condensing chambers, and the lower portion of said column into a series of heat recovery chambers, a series of connected cooling coils in said condensing chambers, means for conducting the cooling fluid from said coils to said heat recovery chambers, means for circulating liquid from said heat recovery chambers to the uppermost of said cooling coils, means for removing condensates from said condensing chambers, means for passing liquid to be treated downwardly through successive vaporizing chambers and through said heat recovery chambers, means for circulating a heat transferring gas upwardly successively through said series of heat recovery chambers and through said series of alternate vaporizing and condensing chambers, a furnace adjacent said column and means for introducing products of combustion from said furnace into said circulating gas between said heat recovery chambers and said series of vaporizing and condensing coils.

6. An apparatus for treating liquids which comprises a column, a series of horizontal partitions dividing the upper portion of said column into a vertical series of adjacent alternate vaporizing and condensing chambers and the lower portion of said column into a series of heat recovery chambers, a series of connected cooling coils in said condensing chambers, means for conducting the cooling fluid from said coils to said heat recovery chambers, means for circulating a heat transferring gas between said heat recovery chambers and said series of vaporizing chambers.

7. In an apparatus for distilling liquids, a condensing chamber which comprises a foraminous partition arranged to receive gasses and vapors and to distribute them throughout said chamber, a cooling coil positioned a short distance above said partition and an overflow outlet for condensate from said chamber, so positioned as to maintain the body of condensate in contact with said cooling coil.

8. The process of distilling liquid mixtures which comprises passing a liquid mixture through a series of bodies of such liquid maintained at successively higher temperatures, circulating a cooling fluid through a series of bodies having successively higher temperatures, circulating a current of heat transferring gas countercurrent to the flow of said liquid, and alternately into contact with the bodies of liquid and into heat transferring relation to the cooling fluid and maintaining said cooling fluid at such a temperature as to condense constituents condensable at approximately the temperature of the next preceding body of liquid.

9. The process of distilling liquid mixtures which comprises passing a liquid mixture through a series of bodies of such liquid maintained at successively higher temperatures, circulating a cooling fluid in a direction parallel to the flow of said liquid, circulating a current of heat transferring gas in a continuous cycle through said bodies of
liquid countercurrent to the flow of said liquid, and alternately into contact with said bodies of liquid and into a heat transferring relation to said cooling fluid and subsequently circulating said gas through the liquid residues remaining after the distillation of said liquid.

10. A process of distilling oil which comprises circulating the oil downwardly through a still as a series of connected bodies, passing a heated gas countercurrent to said oil and condensing oil vapors in said gas in separate bodies as it passes from one body to the next by cooling the condensate bodies with bodies of liquid circulating through said still separately from said oil.

11. A process of distilling oil comprising passing oil as a series of connected bodies downwardly through a still, progressively heating the oil to evaporate a series of fractions thereof, the boiling points of the fractions being progressively higher as the oil flows downwardly and removing the fractions as vapors into condensed bodies of oil cooled independently of the oil being evaporated.

12. A process of distilling oil comprising passing oil in a continuous circuit downwardly through a still, progressively heating the oil to evaporate a series of fractions thereof, removing the fractions as vapors into condensing bodies of oil cooled independently of the oil being evaporated and returning to the oil being evaporated vapors not condensed.

13. A process of distilling oil comprising passing oil in a continuous circuit through a still, maintaining a hot mid-portion in the still, progressively heating the oil as it advances toward said mid-portion, separating a series of fractional distillates from the oil as it is heated, cooling the oil after it has been passed through said mid-portion and circulating the cooled oil through the heated portion of the still for condensing said fractional distillates.

14. A process of distilling oil comprising passing oil in a continuous circuit through a still, conducting a heated vaporizing gas countercurrent through said oil to evaporate the same, removing said gas and vapors from the oil circuit and passing them through a condensing body of oil cooled independently of the oil being evaporated.

15. A process of distilling oil comprising passing oil in a continuous circuit through a still, conducting a heated vaporizing gas countercurrent through said oil to evaporate the same, removing said gas and vapor from the oil circuit and passing them through a condensing body of oil cooled independently of the oil being evaporated and returning to the oil circuit the uncondensed gas and vapors.

In testimony whereof I affix my signature.

HENRY L. DOHERTY.