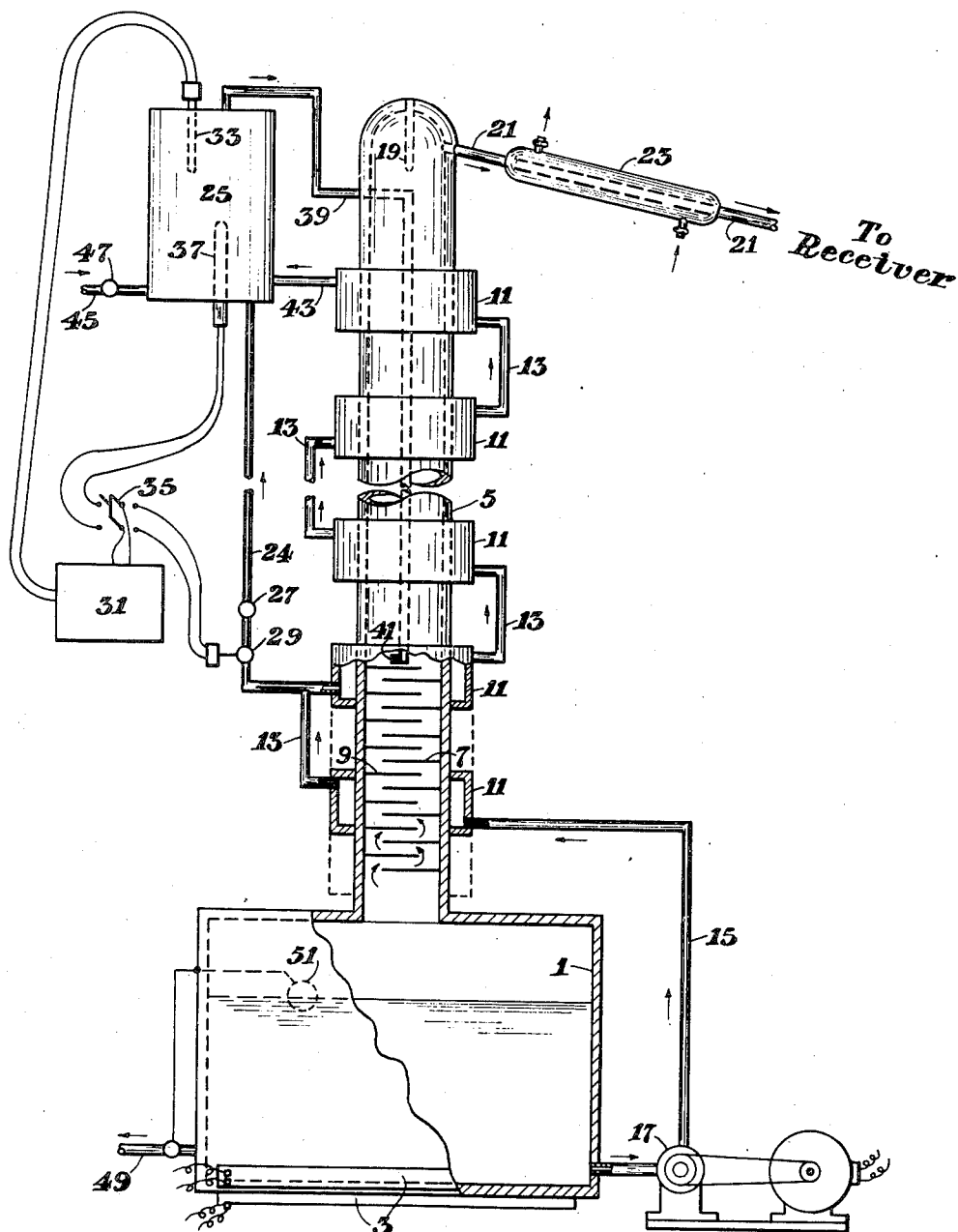


Aug. 2, 1949.

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FRACTIONALLY DISTILLING IN ALTERNATE
HEATING AND COOLING ZONES
Filed Dec. 19, 1945

2,477,595



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2,477,595

FRACTIONALLY DISTILLING IN ALTERNATE HEATING AND COOLING ZONES

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Application December 19, 1945, Serial No. 635,940

10 Claims. (Cl. 202—40)

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This invention relates to method and apparatus for fractionating liquid and is more particularly concerned with method and apparatus for fractionating high-boiling liquids, such as petroleum oils boiling within the lubricating oil range, into close-boiling range fractions.

An object of the invention is to provide apparatus for fractionating liquids containing more than one constituent into fractions of narrow-boiling range.

Another object of the invention is to provide a method for separating liquids of wide boiling range into fractions of narrow boiling range.

Still another object of the invention is to provide method and apparatus for separating high-boiling mineral oils into narrow-boiling hydrocarbon fractions.

Other objects of the invention will become apparent from the following description and the accompanying drawing, in which the single figure is an elevational view, partly diagrammatic and partly in cross-section, illustrating the invention.

Referring to the drawing numeral 1 indicates a still which may be heated in any suitable manner, as for example by means of an electric heater 3. It will be understood that any conventional type of still and heater may be used, as for example a tube and drum still in which the oil is heated in the tubes by means of combustion gases to the desired temperature and flashed into the drum. The heater 3 may be automatically controlled to maintain a predetermined temperature within the still.

Mounted on the top of the still is a fractionating column 5 the interior of which is equipped with a series of horizontal, imperforate, vertically spaced baffles 7 and 9. The baffles 7 and 9 are alternately fastened to opposite sides of the column 5 and do not occupy the entire cross-sectional area of the column, thereby permitting space between the edge of the baffle and the inner wall of the column for vapors to pass upwardly and liquid to flow downwardly through the column in a zigzag path. It will be understood that instead of baffles other liquid vapor contact means such as bubble plates may be used in the column.

Column 5 is made of metal or other material of high heat conductivity and is surrounded by a series of spaced annular hollow jackets 11 through which oil or other heating medium may be circulated. The jackets 11 are serially connected by means of pipes 13. The lowest jacket 11 is connected by line 15 and pump 17 to the bottom of still 1.

As shown in the drawing the jackets 11 are all

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substantially of the same size and are spaced from each other a distance approximately equal to the height of the jacket. It may be desirable, however, to make the lowermost jacket of greater height than the remaining jackets in order to supply sufficient heat for vaporization at the bottom of the column 5. An enlarged jacket is indicated by dotted lines on the drawing.

The top of the column 5 is equipped with a thermometer well 19 in which a thermometer is adapted to be inserted in order to permit observation of the temperature at the top of the fractionating column.

Vapors leave the top of the fractionating column through line 21 and pass through condenser 23. Line 21 is connected to a receiver (not shown) which, in turn, may be connected to a vacuum pump or other pressure reducing device for maintaining reduced pressure in the still and fractionating tower.

A by-pass line 24 connects the lowermost jacket 11 to a tank 25 which is adapted to be maintained full of oil or other liquid undergoing fractionation. Line 24 contains a manually operated by-pass valve 27, and an automatically operated by-pass valve 29 which is adapted to be electrically operated through the control element 31 in accordance with the temperature in tank 25. A thermocouple 33 is inserted in the tank 25 and connected to the control element 31. The numeral 35 indicates a manual bi-polar throw switch which may be left in open position as shown in the drawing, or may be thrown in closed position to close the circuit connecting the heater 37 in tank 25 to the control element 31, or may be thrown in position to close the circuit connecting the automatic by-pass valve 29 to the automatic control 31.

A line 39 is provided for returning liquid from the top of tank 25 to the lower portion of the column 5 through outlet 41. A line 43 is provided to connect the uppermost jacket 11 to the bottom of tank 25. Fresh oil is charged to the bottom of tank 25 through inlet pipe 45 controlled by valve 47.

The still 1 may be provided with an automatically controlled liquid draw-off 49 adjacent the bottom thereof controlled by float 51 in order to keep a constant level of liquid in the still.

The operation of the apparatus just described is as follows: Fresh oil is charged through line 45 to the still 1 until the desired level of oil in the still is reached. The oil in the still 1 is heated to the desired temperature, which will depend upon

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the liquid undergoing distillation. In the event the oil undergoing distillation is a mineral lubricating oil temperatures should be below 600° F. and distillation should be conducted under reduced pressure in order to avoid cracking. Pump 17 is operated in order to withdraw oil from the still and circulate it through the jackets 11 then to tank 25 and back through line 39 to the lower portion of column 5. As the oil vapors ascend the column 5 they are alternately subjected to heating and cooling effects. In the zones surrounded by the jackets, descending liquid composed of circulated oil plus condensate is partially vaporized. In the cooling zones between the jackets a portion of the vapor is condensed. Thus by a series of partial vaporization and condensation steps the vapor ascending towards the top of the column becomes enriched with respect to more volatile constituents, and depleted with respect to the less volatile constituents. In the top of the column a final condensation of the less volatile fractions takes place since the top of the column is subjected to cooling effect of the surrounding atmosphere. By passing the hot oil serially from bottom to top, a temperature gradient is established in the jackets decreasing from bottom to top.

In the event the temperature in the still 1 or in the column 5 exceeds the desired temperature, the liquid undergoing distillation may be withdrawn by pump 17 and by-passed through bypass line 24 directly to tank 25 where it may be mixed with fresh cool oil to lower the temperature. The liquid may be by-passed manually by opening valve 27, or may be by-passed automatically by opening valve 29 and closing the bypass valve circuit so that the amount of oil by-passed will be regulated by the temperature in the tank 25.

Where the temperature in the still and/or the temperature in the fractionating column drops too low, additional heating may be imparted to the oil in chamber 25 by means of element 37. By closing the heater circuit heater 37 will function automatically to maintain any desired temperature.

The distillation operation may be conducted as a batch, semi-continuous or continuous operation. Where continuous operation is practiced a withdrawal line from still 1 is provided, which may be automatically controlled in accordance with the level of liquid in the still. Likewise, the oil inlet valve 47 may be automatically controlled in accordance with the liquid level in still 1.

Instead of returning liquid oil from tank 25 to the lower portion of column 5 it may be returned directly to the still 1.

Instead of maintaining alternate series of hot and cold zones in the fractionating column 5 by means of a series of jackets through which liquid undergoing distillation is circulated, and a series of intervening spaces open to the air, an independent heating medium, such as Dow-Therm, may be circulated through the jackets 11 and jackets may be provided in the intervening spaces through which a cooling fluid such as water may be circulated.

Control of or maintenance of heat balances or heat ratios may be effected in the following ways: (1) by varying the rate of flow of hot liquid through the heating jackets 11; (2) by varying the amount of heat which is removed in the cool zones between the heating jackets 11; (3) by varying the heat gradient established in the heat-

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ed zones 11; (4) by varying the distillation rate as expressed in volume throughput per hour; (5) by varying the mode of reintroducing hot oil into the still; (6) by reconditioning the returning oil before introducing it to the still, that is—by regulating its temperature and/or composition; (7) by maintaining on the distilling system a reduced pressure, the magnitude of which is controlled by the desired rate of distillation; and (8) by the regulation of the amount and composition of reflux created or introduced into the column.

The invention is applicable to straight distillation and fractionation of liquids as well as to distillation and fractionation of liquids carrying in suspension solid comminuted catalysts, refining agents, or other comminuted substances which can be circulated with the oil. The invention may also be used for solvent extraction, extractive distillation, and azeotropic distillation processes.

Although the apparatus shown and described is designed primarily for laboratory distillation, it should be understood that the invention is applicable to large scale operation.

It is claimed:

1. A fractionating device comprising a still, a fractionating column made of heat conducting material connected thereto by means of a vapor conduit in order to receive vapors therefrom, said column being provided with liquid-vapor contact means, a series of vertically spaced jackets surrounding said column, means for passing liquid from said still serially through said jackets and returning it to said still, bypass means for circulating liquid to and from said still without passing it through at least most of said jackets and means for heating and cooling liquid circulated from and to said still.

2. A fractionating device in accordance with claim 1 in which means is provided for returning liquid to the lower portion of the fractionating column and then to the still.

3. A fractionating device in accordance with claim 1 including a tank, means for passing liquid from said jackets to said tank, means in said tank for automatically regulating the temperature of the liquid therein and means for returning liquid from said tank to said still.

4. A method for fractionating liquid of wider boiling range into fractions of narrower boiling range comprising maintaining a body of said liquid at vaporizing temperature in a vaporizing zone, passing resulting vapors upwardly through a fractionating zone, subjecting said vapors and condensate therefrom in said fractionating zone to a plurality of heating steps by indirect heat exchange with liquid from said vaporizing zone, alternately subjecting said vapors and condensate in said fractionating zone to a series of cooling steps, returning liquid after use as heating medium as aforesaid to said vaporizing zone and withdrawing vapors from said fractionating zone and condensing them.

5. Method in accordance with claim 4 in which the liquid used for heat exchange is maintained at a decreasing temperature gradient in each successive heat step in the direction of flow of vapors through the fractionating zone.

6. Method in accordance with claim 4 in which the temperature of the liquid used for heat exchange is adjusted before returning it to said vaporizing zone.

7. Method in accordance with claim 4 in which the liquid used for heat exchange is returned to

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the lower portion of the fractionating zone and then to said vaporizing zone.

8. A fractionating device comprising a still, a fractionating column connected thereto, said column being provided with liquid-vapor contact means, a series of vertically spaced indirect heating zones along the length of said column, a series of cooling zones along said column alternating with said heating zones, means for passing liquid from said still serially through said heating zones to accomplish heating, means for returning liquid to said still, and bypass means for circulating liquid to and from said still without passing it through at least most of said heating zones.

9. A fractionating device comprising, a vertical column connected to a still by means of a vapor conduit in order to receive vapor therefrom, means in said column to provide for liquid-vapor contact and means for repeatedly and alternately heating and cooling said column at spaced vertical intervals, said alternate heating means comprising a series of serially connected jackets through which heating medium liquid withdrawn from the still can be circulated from bottom to top and back to said still.

10. A fractionating device comprising, a ver-

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tical column connected to a still by means of a vapor conduit, means in said column to provide for liquid-vapor contact, a plurality of spaced means along said column for heating said column, means alternating with said heating means for cooling said column, means for circulating liquid from said still serially upwardly through said heating means and means for returning liquid leaving said heating means to said still.

ALBERT W. GOLDSBARRY.

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