April 24, 1945. W. A. McMILLAN 2,374,549

FRACTIONATING APPARATUS

Original Filed Oct. 5, 1940

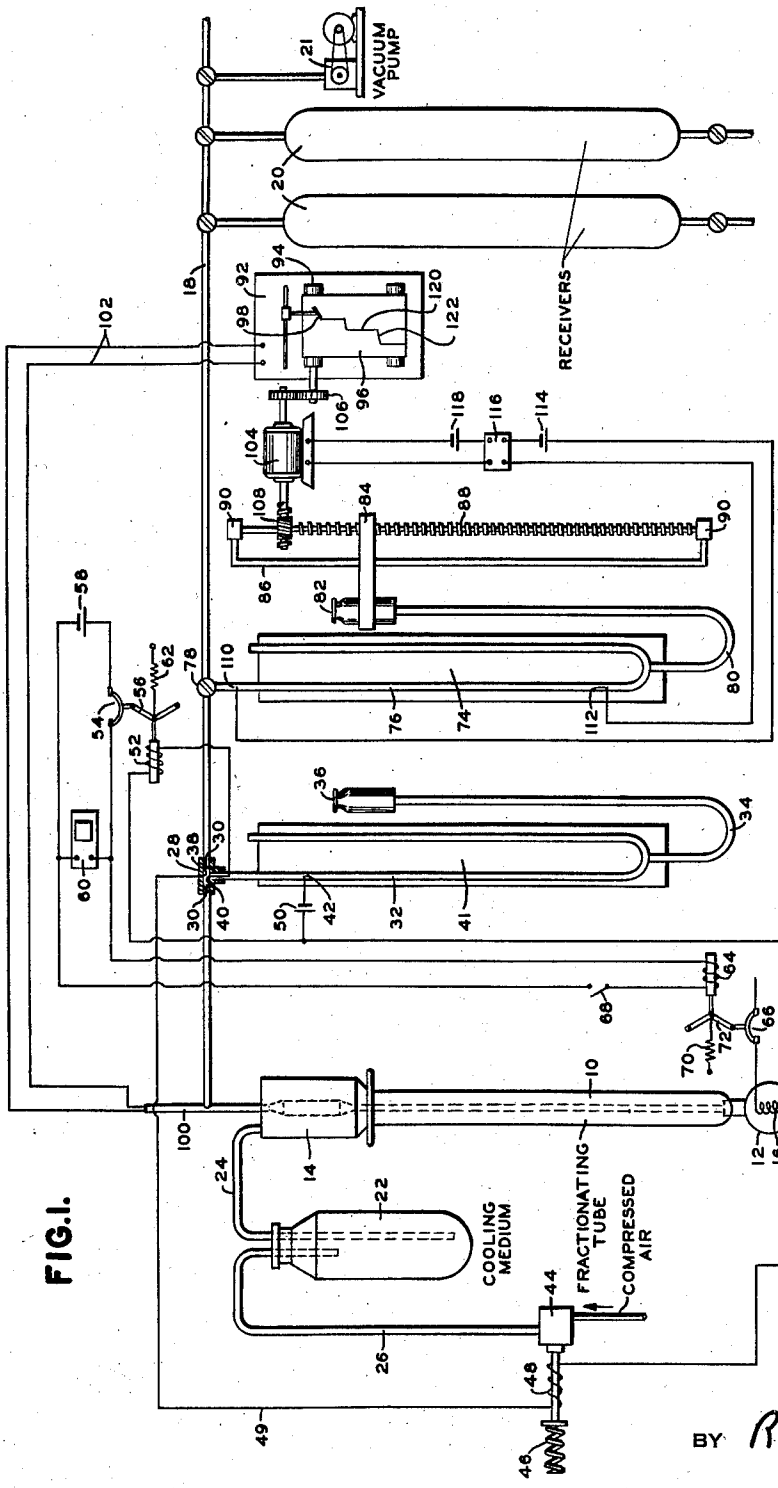


FIG. 1.

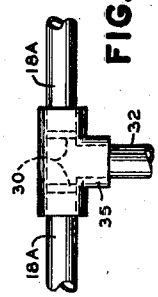


FIG. 3.

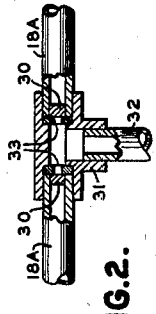


FIG. 2.

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FRACTIONATING APPARATUS

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Original application October 5, 1940, Serial No. 359,853. Divided and this application October 15, 1942, Serial No. 462,113

3 Claims. (Cl. 202—160)

This invention relates to distilling and fractionating apparatus of the type adapted for the analysis of mixtures of volatile liquids, gases of vapors containing condensable liquid fractions, and more particularly to apparatus of this character by means of which efficient control of the fractionation may be obtained while at the same time the temperatures and pressures of the fractionation may be accurately recorded.

The present application is a division of Serial No. 359,853, filed October 5, 1940, for Fractionating apparatus.

The principal object of the invention is to provide an apparatus for precise analytical distillation and fractionation in which the fractionation is regulated automatically in accordance with the pressure of the vapors from a fractionating column and in which the temperatures and pressures of the fractionated vapors are also automatically recorded without the necessity of any manual operation. Another object is to provide means for reducing the amount of heat used for the distillation when the pressure of the fractionated vapors increase to a predetermined value. Still another object is to provide means for regulating the passage of fractionated vapors from the fractionating column to a receiver in accordance with the pressure of said vapors.

Apparatus for analytical distillation and fractionating comprising essentially a distilling bulb, an elongated fractionating tube or column surmounting the bulb and a condenser section at the upper end of the fractionating tube with equipment for collecting and measuring the distilled vapors is known; for example, see Podbielniak Patent No. 1,917,272, granted July 11, 1933. Apparatus of this type operates on the principle of sharp, relatively adiabatic fractionation to separate constituents of the mixture being analyzed in the order of their boiling points. Apparatus of this type which is available on the market has so far sufficed to provide only a proximate analysis of the mixtures and has proved incapable of giving accurate quantitative results, or even reproducible results with different operators.

An apparatus for accomplishing these results, and by means of which accurate and reproducible results may be obtained, is described in the McMillan Patent No. 2,005,323 granted June 18, 1935. The proper operation of that equipment necessitates the constant attendance of at least one operator since it is necessary to make various manual adjustments to the apparatus during the distilling and fractionation of a given mixture. With the apparatus to be described the operation can be conducted without the frequent attendance of any operator since the fractionation and the recording of the temperatures and pressures are automatic. Apparatus which is now on the market also necessitates several complicated elements which are fragile and very expensive and also involves a complicated measuring and recording system which requires the use of precision equipment which is difficult to obtain. Much of this precision equipment is obtainable only from foreign countries. Moreover, with the apparatus now on the market there is considerable dead space in the pipe connections through the receivers and this, of course, interferes with the accuracy of the determinations.

In accordance with the present invention, an apparatus comprising a distilling bulb, an elongated adiabatic fractionating tube disposed thereabove and surmounted by a condenser section is provided together with means for automatically supplying a cooling liquid to this condenser in accordance with the pressure of the vapors leaving the condenser section. Means are also provided for decreasing the heat applied to the distilling bulb when or if the pressure of the vapors leaving the condenser section reaches a predetermined point. The invention also contemplates the use of a pressure-temperature recorder of the "recording potentiometer type," this instrument being operated automatically by and in accordance with variations in pressure in the vapors passing from the condenser section. A pen is adapted to trace a record on a chart roll in accordance with variations in temperature of the vapors in the condenser section while at the same time the chart roll is advanced so that the chart travel is directly proportional to increases in pressure of the vapors leaving the condenser section.

For a further understanding of the invention reference may be had to the accompanying drawing in which:

Figure 1 is an elevational view generally diagrammatic of the apparatus embodying the improvements of the present invention;

Figure 2 is a vertical, sectional elevation through the liquid valve, and

Figure 3 is a vertical elevation showing a modified form of valve.

Referring to the drawing, an elongated, jacketed, fractionating tube or column 10 is provided at its lower end with a distillation vessel or bulb 12 and a jacketed condenser section 14 at its upper end. The distillation bulb is provided with a suitable heating element such as the electrical

resistance coil 16. A conduit 18 comprising a vapor passage leads from the condenser section to one or more receivers 20 in which a reduced pressure is normally maintained as by means of a suitable vacuum pump 21. A container 22 of a cooling medium such as liquid nitrogen is connected to the condenser section 14 by means of pipe 24 and a pipe 26 leading from a source of compressed air, not shown, is connected to the top of the vessel 22, the arrangement being such, as will be described hereinafter, that the compressed air forces the liquid nitrogen into the condenser section 14 at the proper time, thereby reducing the pressure of the vapors taken off through the conduit 18 and thus regulating the fractionation by control of reflux and distillation rate. Disposed in the conduit 18 is a valve device 28 comprising a pair of porous discs 30 preferably formed of sintered glass filter plates and extending across the vapor passage. One leg of a mercury manometer 32 is connected to the conduit 18 between the disc 30 and the other leg of the manometer is open to atmosphere as usual. A flexible tube 34 connects the lower end of the manometer 32 with a mercury bulb or reservoir 36 which can be adjusted up or down to vary the height of the mercury in the manometer. The porous discs 30 may be formed of glass ground to a powder, burned to a sintering point and formed into discs or plates of a convenient size and are of such construction that they are pervious to the passage of gas or vapor while impervious to the passage of mercury therethrough. Thus, when the pressure in the offtake 18 drops below a certain value the mercury will rise in the manometer leg connected to the offtake and will fill the space between the two sintered discs 30, thus preventing vapor from the fractionator 10 from passing to the receivers 20. When the pressure in the offtake 18 rises above the predetermined value or normal the mercury is depressed within the manometer leg so as to open the gas passage through the sintered discs 30.

The general construction of the valve device 28 comprising the pair of sintered glass discs 30, is described in the aforementioned McMillan Patent 2,005,323. The glass discs have formerly been sealed in parallel in opposite sides of a glass T and this construction has been obtainable only from certain foreign countries. In accordance with the present invention, a somewhat different mounting of the discs has been provided as shown in Figures 2 and 3 and has proved to be very satisfactory. As shown in Figure 2, the discs 30 are sealed in the ends of pipes 18A, these pipes comprising a portion of the offtake 18 previously described. The ends of the pipes 18A containing the discs 30 are placed in a T 31 which may be of metal or other suitable material and the ends of the pipes are preferably seated against suitable gaskets 33. A sealing material is placed so as to seal the space around the ends of the pipes 18A and between the pipes and the T 31. It has been found that this construction is simple to effect and it renders unnecessary the importing of expensive equipment. Although the T 31 is shown as constructed of metal, this member may, of course, be made of glass which will provide suitable insulating means for the electrical contacts to be placed within the valve and which will be described hereinafter. Another method of mounting the glass discs 30 is shown in Figure 3 in which the discs are sealed in opposite legs of a T 35 formed of a plastic, preferably of acrylic or styrene resin types. The ends of the pipes 18A can be sealed into the opposite legs of the T after the T has been constructed, or the entire T may be cast around the ends of the pipes and the discs 30. In either case a construction is provided which is transparent so that mercury within the valve can be observed and which will provide an insulating medium in which electrical contacts and wires may be mounted.

Within the valve 28 are arranged upper and lower electrical contacts 38 and 40, respectively, and at a lower point in the manometer 32 is disposed a third contact 42. Disposed in the pipe 26 leading to the vessel of cooling medium 22 is a valve 44 normally held in an open position by means of a compression spring 46. The valve 44 is provided with a solenoid 48 and the solenoid winding is connected in circuit with a suitable source of electricity 50, the upper contact 38 and the third contact 42 in the manometer 32. In the operation of this portion of the apparatus when the mercury within the manometer 32 fills the space between the discs 30 so as to shut off the gas or vapor passage, the electrical circuit will be completed between contacts 38 and 42 and the solenoid 48 will be actuated to hold the valve 44 in its closed position against the compression of the spring 46. When the pressure increases in the offtake 18, the mercury will be depressed in the manometer leg and the electrical circuit will be broken between contacts 38 and 42, whereupon the solenoid 48 will be deenergized and the spring 46 will open the valve 44 allowing compressed air to pass into the vessel 22 and force part of the liquid nitrogen into the condenser 14 to cool the vapors and thus reduce the pressure. When the pressure has been sufficiently reduced, the mercury will rise in the manometer leg, thus again closing the electrical circuit just described, so that the valve 44 will be closed to stop the flow of cooling medium into the condenser, thereby allowing the pressure to increase again. It will be seen that an apparatus has thus been provided whereby the fractionation will be controlled automatically in accordance with the pressure of the fractionated vapors.

The electrical contact 40 is connected in another circuit with the coil 52 of a break relay 54, the source of electricity 50 and contact 42. The armature 56 of the relay 54 is adapted to close a circuit containing a source of electricity 58 and a suitable alarm device such as a buzzer 60. Thus, should the pressure in the offtake 18 rise to an abnormal degree, the circuit between the contacts 40 and 42 will be broken, thus de-energizing the coil 52 and permitting a spring 62 to pull the armature 56 to the right to close the circuit to energize the alarm 60. In addition the circuit containing the electrical source 58 is preferably connected to the coil 64 of a relay 66 and a suitable switch 68 through wires 69. The relay 66 is connected in circuit with the electrical heating coil 16 of the distillation vessel 12 so that in the event of an abnormal increase in the pressure of the vapors in offtake 18, the relay 54 will be closed not only to actuate the alarm 60 but also to energize the relay coil 64, thus pulling the armature 72 to the right against the tension of spring 70 to open the circuit containing the heating element 16 of the distillation vessel 12. The reduction of heat in the vessel 12 will, of course, slow up the distillation to reduce the pressure of the vapors in conduit 18.

In accordance with the invention, means are also provided for automatically recording the pressures and temperatures of the vapors leav-

ing the condenser section 14 and passing through the conduit 18 to the receivers 20. To this end a second mercury manometer 74 has one leg 76 communicating with the conduit 18 through a valve 78. The other leg of the manometer is open to the atmosphere as usual and a flexible tube 80 connects the lower end of the manometer with a mercury bulb or reservoir 82. The reservoir 82 is mounted in a bracket member 84 slidable vertically on a guide rod 86 and having threaded connection with a rotatable threaded rod 88, the latter supported in suitable bearings 90. As the rod 88 is rotated the bracket 84 and thus the mercury reservoir 82 will be moved up or down so as to control the level of the mercury in the manometer leg 74. A recording instrument 92, preferably of the recording potentiometer type, is provided with a chart roll 94 over which a chart 96 passes as the roll is rotated. The instrument is also provided with a recording pen 98 which is moved laterally of the chart 96 in accordance with variations in temperature in the fractionating column 10, which temperature variations actuate a thermocouple 100 connected to the instrument 92 by means of suitable wires 102. The instrument 92 may be of any suitable type as long as it includes the elements so far mentioned. A reversible electric motor 104 is arranged to drive the chart roll 94 through suitable gearing 106 and this motor is also connected through suitable reduction gearing which may comprise a worm and wheel 108, to drive the threaded rod 88. Thus, when the motor 104 is energized it will rotate the chart roll 94 to advance the chart 96 while at the same time the rod 88 will be rotated to raise the bracket 84 and thereby the mercury reservoir 82.

In order to control automatically the motor 104, an electrical circuit is provided comprising a contact 110 mounted within and near the upper end of the manometer leg 76, and another contact 112 located within and adjacent the bottom of the manometer 74. In addition to the contacts 110 and 112 this circuit also includes a source of electricity 114 and a break relay 116. The contacts, not shown, of the relay 116 are connected in another circuit with the motor 104 and a source of electricity 118. As the receiver pressure rises during distillation the mercury level in the manometer leg 76 will fall below the contact 110, which comprises a fixed reference point, and the circuit to the relay 116 will thereby be broken and the actuation of the relay will then complete the circuit to energize the motor 104. As stated hereinbefore, when the motor 104 is energized it will drive the chart roll 94 to advance the chart 96 and simultaneously therewith it will rotate the threaded rod 88 so as to lift the mercury reservoir 82 to bring the mercury level in the manometer leg 76 back until electrical contact is remade at 110. The completing of the circuit between contacts 110 and 112 will thus actuate the relay 116 to stop the motor 104 and the chart 96 and the mercury reservoir 82 will remain in the positions then obtained until the receiver pressure again depresses the mercury in the manometer leg 76 sufficiently to open the circuit to the relay 116 when the cycle will be repeated.

As long as a certain component of a gaseous mixture is being distilled from the column 10 the temperature will, of course, remain substantially constant. The pressure in the receiver 20 and the offtake 18 will, however, increase during this period and the pen 98 will, therefore, draw a straight, vertical line such as 120 on the chart 96. When the distillation of one component of the gaseous mixture has been completed the temperature will, of course, rise until the boiling point of the next component or the next constant boiling mixture is reached. The pen 98 will thereupon move horizontally a certain distance across the chart 96 as indicated at 122 with very little vertical movement of the chart during this time so that the distillation curve will be a series of slopes and plateaus in the usual manner. The proportion of any ingredient in a gaseous mixture can be readily computed from the chart by calculation of the ratio of the length of the vertical line representing the distillation of that component, to the total vertical length of the chart during the entire distillation. When the mercury reservoir 82 has been lifted to the top of the rod 88, or whenever it is desired to change a chart, to adjust the amount of mercury in the reservoir or to change distillate receivers it is merely necessary to close the valve 78 and to energize the motor 104 so that it will be operated in the reverse direction. The rod 88 will then rotate to lower the bracket 84 and the reservoir 82 to a point near the bottom of the rod after which the valve 78 can be opened and a new recording started.

It will thus be seen that by a comparatively simple construction the apparatus of the present invention accomplishes the following operations entirely automatically: controls the supply of cooling medium to the condenser jacket of the distillation column, in accordance with the distillation pressure at the outlet of the column; controls the supply of heating fluid to the still, in accordance with the distillation pressure, and actuates the chart of the recording instrument so that the travel of the chart is made proportional to the pressure increase within the vapor receiver.

Obviously many modifications and variations of the invention, as hereinbefore set forth, may be made without departing from the spirit and scope thereof and therefore only such limitations should be imposed as are indicated in the appended claims.

I claim:

1. In apparatus for distillation and fractionation, comprising in combination a distilling means, a fractionating column communicating with said distilling means, a partial condenser for condensing vapors passing from said fractionating column and for supplying reflux to the fractionating column, means for supplying a refrigerant to said condenser, a receiver, a vapor offtake leading from said condenser to said receiver, a liquid valve in said offtake including a member porous to vapor but impervious to liquids and a mercury manometer communicating with said vapor offtake at the downstream side of said porous member and responsive to pressure in said offtake, and means associated with said liquid valve and actuated on a predetermined movement of the mercury in said liquid valve for actuating said refrigerant supplying means, said last named means comprising an electrical circuit including an electrical contact in said liquid valve, a second electrical contact in said manometer, a source of electricity, and a device for actuating said refrigerant supplying means when the electrical circuit is open between said two contacts.

2. In apparatus for analytical distillation and fractionation having a distilling section and an

elongated fractionating column communicating with said distilling section, means for cooling the upper end of said fractionating column, means for actuating said cooling means, a vapor offtake from said column, a liquid valve in said offtake comprising a pair of discs porous to vapor but impervious to liquid and a mercury manometer having one leg communicating with said valve between said discs and responsive to pressure in said offtake, an electrical contact within said valve normally covered by said mercury, a source of electricity, and an electrical circuit including said contact, the mercury in said manometer, said source of electricity and said actuating means, the arrangement being such that on a predetermined increase in pressure in said offtake the mercury in said valve and said manometer leg will be depressed to uncover said contact and open said electrical circuit thereby actuating said cooling means to cool said column and reduce the pressure in said offtake.

3. In apparatus for distillation and fractionation, comprising in combination a distilling means, a fractionating column communicating with said distilling means, a partial condenser for condensing vapors passing from said fractionating column and for supplying reflux to the fractionating column, a supply of compressed gas connected to force a refrigerant into said condenser, a receiver, a vapor offtake leading from said condenser to said receiver, a valve in said offtake responsive to the pressure therein, said valve comprising a pair of vertically disposed flat members porous to vapor but impervious to liquid, said members being arranged in separated relation to form a liquid chamber therebetween, a mercury manometer communicating with said chamber, and means associated with said valve for operating said refrigerant supplying means, said operating means comprising a source of electricity, an electrical contact in the upper part of said chamber, a second contact in the lower portion of said manometer, a solenoid operated valve in said compressed gas connection, and an electrical circuit including said contacts, said solenoid and said source of electricity, the arrangement being such that on a predetermined increase in pressure in said offtake, the mercury in said chamber will be depressed to open said electrical circuit to actuate said solenoid to allow compressed gas to force said refrigerant into said condenser.

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