July 24, 1934. W. J. PODBIELNIAK 1,967,258

DISTILLATION CONTROL DEVICE

Filed Oct. 30, 1931 4 Sheets-Sheet 1

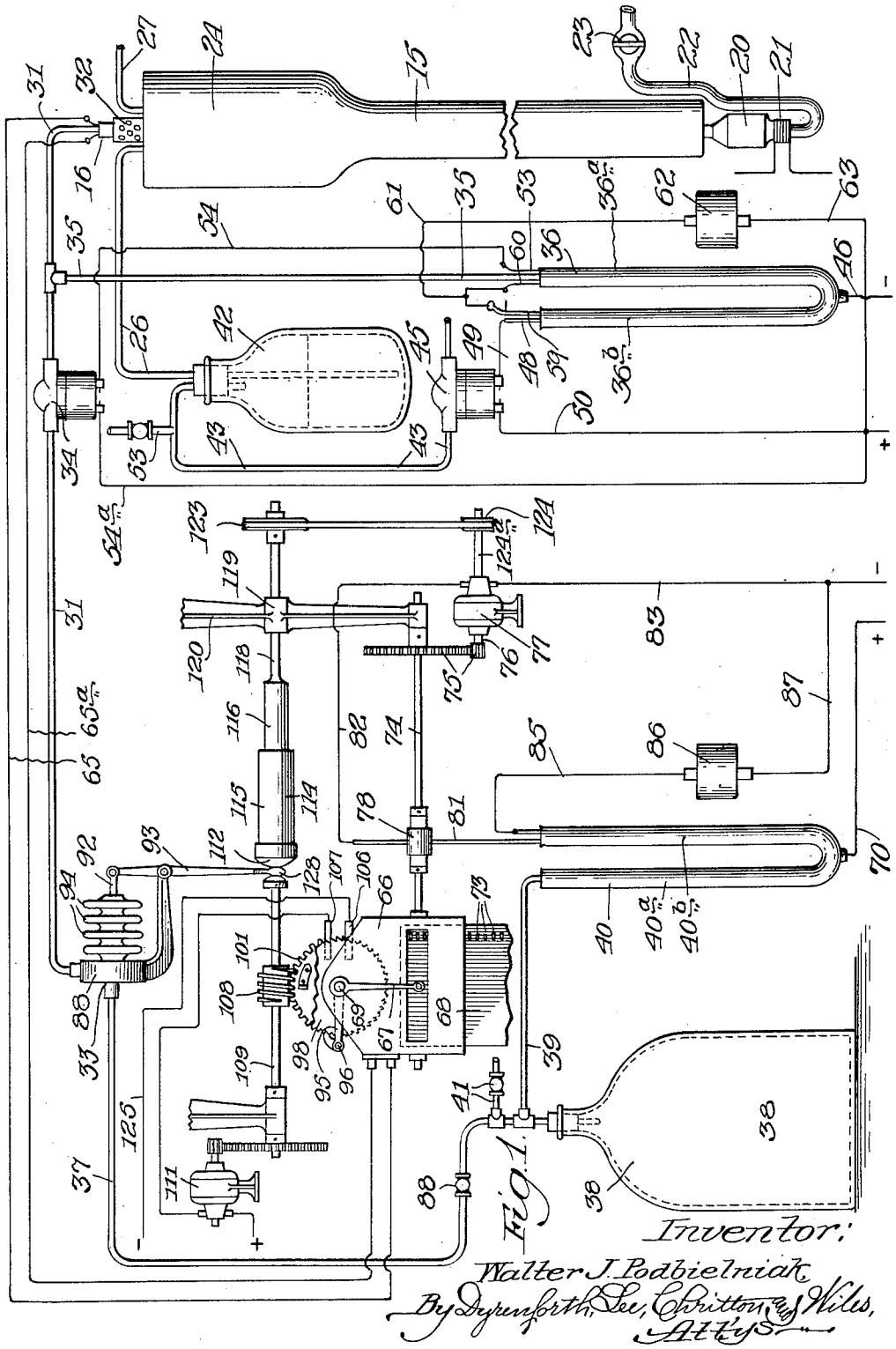


Fig. 1.

Inventor:
Walter J. Podbielniak
By Dyrenforth, See, Christton & Wiles,
Attys.

July 24, 1934. W. J. PODBIELNIAK 1,967,258

DISTILLATION CONTROL DEVICE

Filed Oct. 30, 1931 4 Sheets-Sheet 2

Fig. 2a.

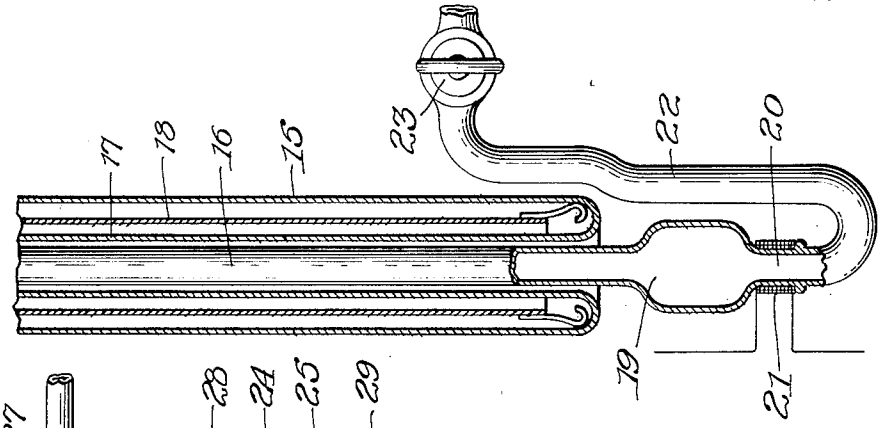


Fig. 2.

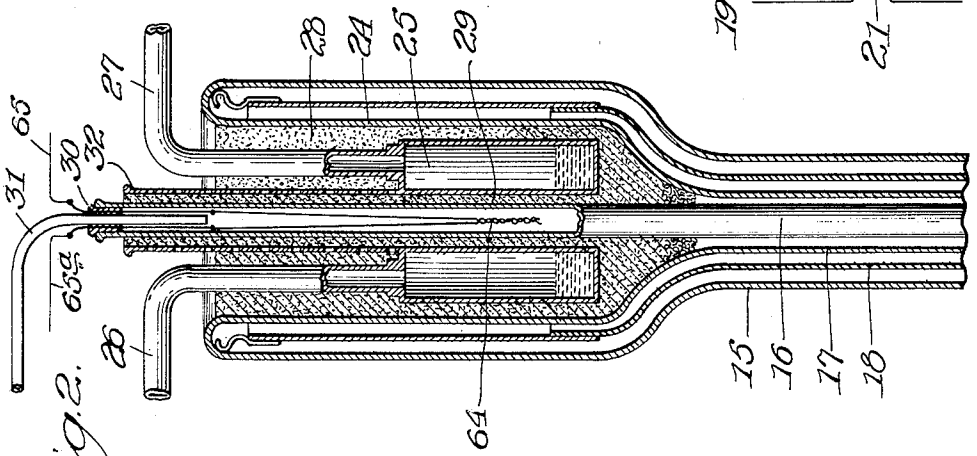


Fig. 3.

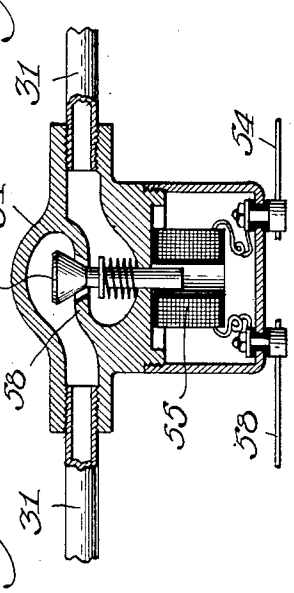
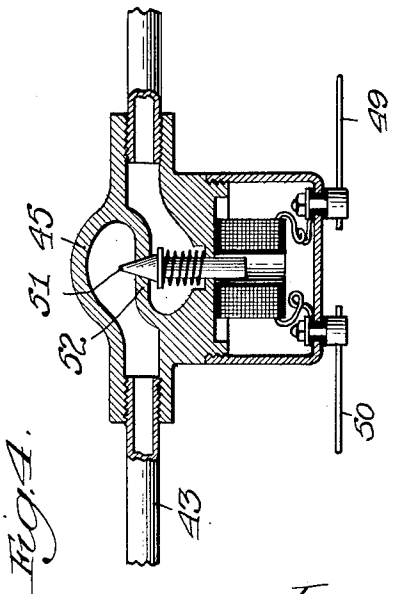


Fig. 4.

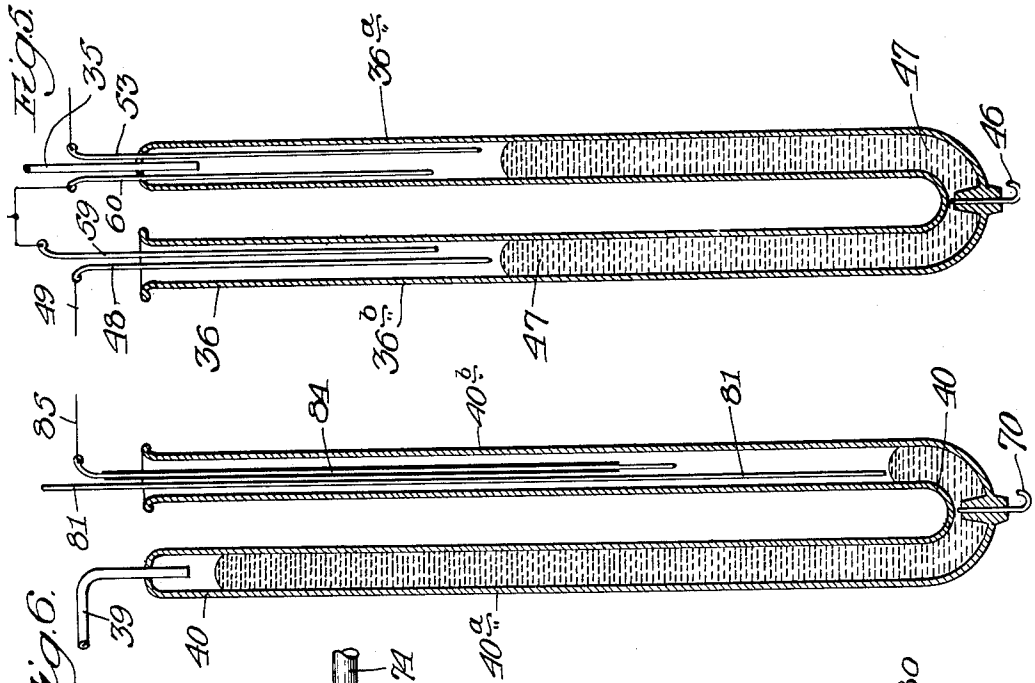


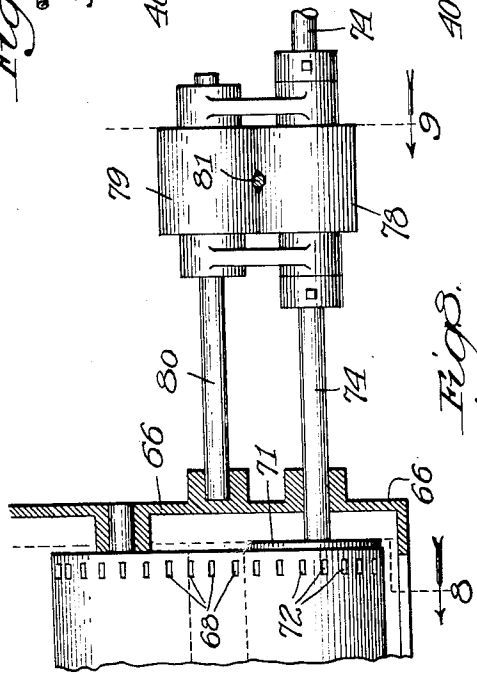
Inventor:
Walter J. Podbielniak,
By Dyrenforth, Lee, Chritton & Wiles,
Attys.

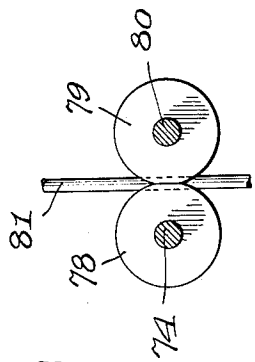
July 24, 1934. W. J. PODBIELNIAK 1,967,258

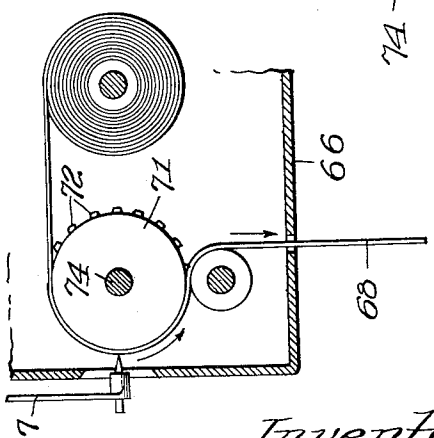
DISTILLATION CONTROL DEVICE

Filed Oct. 30, 1931 4 Sheets-Sheet 3









Inventor:
Walter J. Podbielniak.
By Dyrenforth, Lee, Chritton & Wiles.
Attys.

July 24, 1934. W. J. PODBIELNIAK 1,967,258

DISTILLATION CONTROL DEVICE

Filed Oct. 30, 1931 4 Sheets-Sheet 4

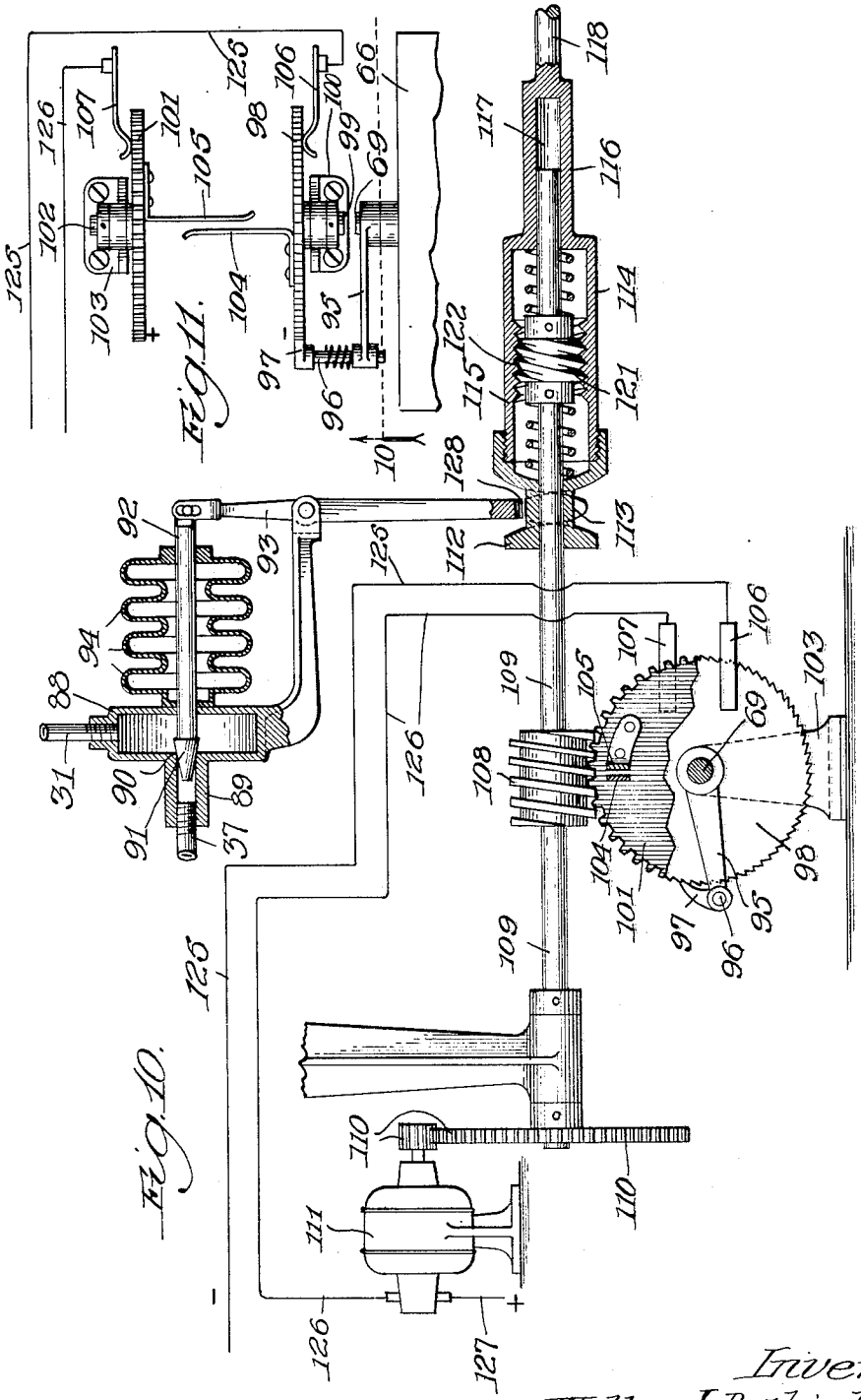


Fig. 11.

Fig. 10.

Inventor:
Walter J. Podbielniak,
By Dyrenforth, Lee, Chritton & Wiles,
Attys.

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UNITED STATES PATENT OFFICE

1,967,258

DISTILLATION CONTROL DEVICE

Walter J. Podbielniak, Tulsa, Okla.

Application October 30, 1931, Serial No. 572,146

53 Claims. (Cl. 202—160)

The present invention relates to improvements in fractional distillation, and more particularly to the control of the progress of more precise fractional distillation, particularly for analytical purposes, as in the analysis of constituents of natural gas, natural gasoline, gasoline and other petroleum distillates and of mixtures of other volatile materials such as chlorinated hydrocarbon products, alcohols, ethers, aromatic hydrocarbons, manufactured gas, coal gas and the like.

In my prior applications Serial No. 351,726, filed April 1, 1929, and Serial Nos. 475,584 and 475,585, filed August 15, 1930, I have described a precision fractionating column, more particularly intended for analytical purposes, and methods of distillation in connection with the operation thereof, whereby the precise analytical distillation of mixed liquids or liquefiable gases may be conveniently and rapidly effected and an accurate determination of the individual constituents of such mixtures made. In accordance with the present invention, in the operation of such a precision distillation column, I secure an automatic control of the distillation operation as a whole, as well as an effective and automatic control of the conditions within the column itself. I am also enabled to secure directly and automatically a complete record of the distillation corresponding to the usual fractional distillation curve and giving directly the relationship between temperature and quantity of material distilled in contradistinction to the usual temperature-time curves ordinarily secured in such operations. By the use of the present invention I am able to make the progress of the distillation, after it has been started, substantially completely automatic.

The invention will be fully understood from the following description thereof, illustrated by the accompanying drawings, in which:

Figure 1 is a general view, partly in elevation and partly in section, and largely diagrammatic, of a layout of apparatus suitable for carrying the invention into effect;

Figs. 2 and 2ª are detail longitudinal sectional views through the upper and lower portions respectively of the fractionating column;

Fig. 3 is a sectional detail view of a solenoid-operated valve for control of the vapor line from the fractionating column;

Fig. 4 is a detail sectional view through a solenoid-operated valve for the control of the supply of cooling medium to the fractionating column;

Fig. 5 is a sectional view through the manometer controlled by conditions in the fractionating column;

Fig. 6 is a sectional view through the manometer controlled by conditions in the vapor receiving container;

Fig. 7 is a detail view, partly in section, of a portion of the apparatus for driving the paper feed of the recording device;

Fig. 8 is a sectional view on the line 8 of Fig. 7; and

Fig. 9 is a detail sectional view on the line 9 of Fig. 7;

Fig. 10 is a detail view, partly in section, of the mechanism provided for control of the rate of distillation; and

Fig. 11 is a detail plan view of a part of the control mechanism shown in Fig. 10.

Referring more particularly to the drawings, the numeral 15 indicates generally a fractional distillation column preferably of the character described in my prior applications 351,726 and 475,585, above referred to. This column structure is provided internally with an elongated distillation column 16 surrounded by an evacuated jacket member 17, suitably constructed of quartz, of a glass of low temperature coefficient, or with its internal wall of quartz and its outer wall of glass. The evacuated jacket is preferably provided internally with a reflector member 18, the transparent evacuated jacket 17 together with the reflector member 18 constituting an exceedingly efficient insulating jacket for the distillation column 16.

The distillation column 16 is of small diameter, preferably in the order of 3.4 to 5 mm. internal diameter for use in the analytical distillation of materials boiling at low temperatures, such as constituents of natural gas, natural gasoline, coal gas and the like, although it may be found convenient to employ a tube of larger diameter, say of 9 to 11 mm. internal diameter for the analysis of higher boiling constituents, such as gasoline, mixtures of lower alcohols and the like.

At its lower end the distilling column 16 projects beyond the insulating jacket 17, as shown in Fig. 2ª, and is enlarged to form the distilling bulb 19, from the lower end of which there projects downwardly a reduced tubular displacement section 20 surrounded by the heating coil 21, by which heat may be applied to material contained in the distilling bulb 19. The displacement tube 20 is connected by the line 22 provided with a valve or stop-cock 23, by which

the material to be analyzed may be introduced into the column or a displacement liquid, such as mercury may be introduced to enter and fill the displacement tube 20 below the distillation bulb to control the quantity of the sample subjected to distillation and to serve for imparting heat thereto.

At the upper end of the column, the evacuated jacket 17 is enlarged, as at 24, to provide for reflux cooling at the upper portion of the distilling column, as shown in detail in Fig. 2. Within the enlarged space around the distilling tube 16 provided by the enlargement 24, there is disposed an annular cooling chamber 25, suitably of metal, provided with an inlet pipe 26 for the supply of a suitable cooling medium, such as liquid air, liquid carbon dioxide, liquid ammonia, or, for higher temperature use, brine or the like. The cooling chamber 25 is provided with an outlet line 27. A protecting sleeve 28, suitably of metal, perforated to reduce its heat conductivity, extends from the cooling chamber 25 upwardly to or beyond the top of the evacuated jacket. The space within the enlarged portion 24 of the insulating jacket 17 is filled with a suitable dry insulating material, such as comminuted asbestos, kieselguhr or the like, except for the space occupied by the cooling vessel and that between the cooling vessel 25 and the distilling tube 16, which is either left empty or filled with a material having a high heat conductivity, such as the finely divided copper or iron particles 29.

The top of the distilling column 16 is closed by a seal or plug 30, through which passes the vapor outlet line 31. The vapor line 31 extends to the control valve 33, the construction and operation of which will be more fully hereinafter set forth. Between the control valve 33 and the column there is provided in line 31, a suitable shut-off valve 34, preferably automatically operated; and also a connection 35 leading to the manometer 36, which indicates and controls the pressure conditions prevailing in the column.

From the control valve 33, vapors from the distilling column are conducted through the line 37 to the receiving bottle or container 38, in which, as described in my prior applications hereinbefore referred to, a reduced pressure is maintained and a temperature such that the contents thereof are kept in vapor form. The receiving container 38 is of fixed volume and the increase in pressure therein resulting from the receipt therein of vapors evolved from the distillation column is indicated by providing from the line 37 a connection 39 leading to a manometer 40, by which the pressure in the receiving container 38 is indicated and a further control of the operation secured, as more fully hereinafter set forth.

In beginning distillation of a sample to be subjected to analysis, for example, natural gasoline, the entire system is evacuated, suitably through a valved line 41, with the valves 33 and 34 open and the stop-cock 23 controlling the opening to the lower portion of the distilling column closed. When evacuated, the column is isolated by closing the valves 33 and 38 in the lines leading from it and the column is cooled by supplying cooling medium to the cooling chamber 25. For this purpose, the inlet line 26 to the cooling chamber 25 may be connected with a suitable source of supply of the cooling medium, for example, the vacuum bottle 42 containing liquid air, the supply of the liquid air to the cooling chamber being controlled by a compressed air line 43 provided with the control valve 45. When the desired cooling has been effected, the sample to be subjected to distillation introduced into the distilling bulb 19 in the lower portion of the column through the line 22, the sample being followed, if desired, by a sufficient amount of mercury to displace the sample from the displacement section 20 surrounded by the heating coil 21. The pressure in the column is still somewhat below atmospheric, and heat is supplied to effect vaporization and build up pressure within the column to approximately atmospheric or somewhat above, as found desirable for the particular material under treatment. Distillation is then permitted to proceed by opening valve 88 in line 37, to permit uncondensed vapor to pass from the column through the lines 31 and 37 to the evacuated, fixed volume receiver, which is under a lower pressure than that prevailing in the column. The operations of initiating distillation are fully set forth in my prior applications above referred to.

The distillation having been initiated, its progress is automatically controlled in accordance with the present invention as fully set forth hereinafter.

As stated above, the manometer 36, shown on enlarged scale in Fig. 5, is provided with a connection 35 to the vapor line 31 leading from the distillation column and in consequence thereof, is sensitive to pressure conditions in the column. The manometer 36 is suitably of the U-tube type with a closed leg 36^a with which the connection 35 communicates, and an open leg 36^b. The pressure-sensitive liquid in the manometer 36 is preferably a liquid of high electrical conductivity, such as mercury, and is connected by a suitable conductive wire 46 passing through the glass of the tube with one pole of a suitable source of electricity, for example, the negative pole. As will be readily apparent, an increase in pressure in the distillation column will be communicated through the vapor line 31 and connection 35 to the closed leg 36^a of the manometer, forcing the mercury column 47 upwardly in the open leg. A metal contact rod 48 is provided which extends into this leg and ends only a short distance above the desired normal level of the mercury column therein. The contact rod 48 is provided with an electrical connection 49 leading to the solenoid-operated valve 45 in the compressed air supply line 43 leading to the liquid air container 42 or other container for the cooling medium. The solenoid-operated valve 45 is connected to the opposite pole of the source of electricity through the line 50. As will be apparent, on rise in pressure in the vapor line, contact is made between the mercury column 47 in the manometer 36 and the contact rod 48, resulting in a series connection through the control circuit of the solenoid-operated valve 45, which is shown in section on an enlarged scale in Fig. 4. Although in practice it is preferred to operate this and other control circuits through relay circuits controlled by the manometer contacts, for simplicity in illustration, direct control circuits are shown herein. The solenoid operates the conical valve member 51 which is normally held against a valve seat 52 by spring pressure, opening the valve and permitting a flow of compressed air, which forces additional liquid air or other cool-

ing medium out of the container 42 into the cooling vessel 25 surrounding the upper portion of the column, thereby effecting additional cooling and restoring the pressure to normal. The mercury column 47 in the manometer 36 responds to this decrease in pressure, its level in the leg 36b of the manometer is lowered, contact with the contact rod 48 is broken and the valve controlling the supply of compressed air to the vessel 42 is returned to its normal closed position. I have found it convenient to provide a minute continuous vent or leak of air or gas from the vaporizable cooling medium in the chamber 42 for example, through vent line 53, provided with an adjustable valve whereby excess vaporized air or other cooling medium may be vented. The operation of the valve member 51 in the manner hereinbefore described causes the supply of a substantial additional quantity of compressed air and a corresponding additional supply of cooling medium to the reflux column.

Provision is also made in connection with the pressure indicating device and manometer 36 for completely closing the vapor line 31 by means of the shut-off valve 34 in the event that there is an undesired decrease in pressure in the distillation column, such as may occur with an improper supply of heat to the column or near the end of a distillation. For this purpose, a metal contact rod 53 is provided in the closed leg 36a of the manometer, so adjusted in position as to be contacted by the mercury column 47 when the pressure in the distillation column and vapor line drops to a predetermined amount below normal, say 8 to 10 mm. When contact is made by the mercury column 47 with the contact rod 35, a circuit is closed through the connector 54, the operating solenoid 55 of the shut-off valve 34 (shown in section in Fig. 3) and connector 54a, leading to the opposite pole of the source of electricity. When this circuit is established, solenoid 55 is operated and the valve member 57, normally held open, by spring pressure, for example, is drawn against its seat 58, and the vapor line 31 is thereby shut off. Pressure then begins building up in the distillation column, and as soon as the increase in pressure is communicated to the manometer 36, the mercury column is forced out of contact with the contact rod 53, the circuit through the valve 34 is broken, and distillation is again permitted to proceed.

If desired, additional provision may be made in connection with the pressure responsive device or manometer 36 to operate emergency signals in the event of an excessive rise or drop in pressure in the distillation column. For this purpose, additional contact rods 59 and 60 may be provided in the open and closed legs respectively of the manometer 36, with their points somewhat higher than the points of the contact rods 48 and 53. In the event that the mercury column in the manometer contacts with either of the rods 59 or 60 due to excessive rise or drop in pressure, a circuit is closed through the rod with which contact is made, connector 61, signal device 62 and connector 63 leading to the opposite pole of the source of current and the signal, which may be either an auditory signal, such as a buzzer, or a visual signal, such as a light, or both, is caused to operate to draw the attention of an operator to the emergency conditions existing in the system.

In order to secure a record of the temperature at which the various fractions pass out of the column 16 in vapor form, I provide in the upper portion thereof a thermo-couple 64 connected in the usual manner by connectors 65 and 65a with a recording pyrometer 66, suitably of the potentiometer type, the recording arm thereof being indicated by the numeral 67. As such pyrometers are of a type well known in the art, the detailed mechanism thereof is not shown. As is usual in such pyrometers, the recording arm 64, by which a record is made upon a traveling paper sheet 68 is operated by a shaft 69 and is caused to move to the left or clockwise with rise in temperature.

In the operation of such recording pyrometers, it has hitherto been customary to cause the paper sheet 68 upon which the record is made to be moved by clock-work so as to travel at a constant rate of speed, so that the record made indicates not only the temperature of the device under operation, but also the time at which the record is made. In accordance with the present invention, however, I provide means whereby the feed of the paper sheet 68 is controlled in accordance with the quantity of the vapor passing over, through the pressure rise in the fixed-volume container 38, in which the vapor fractions are received and maintained in vapor phase. As a result, a record made upon the sheet 68, showing the temperature of evolution of the successive vapor fractions in relation to their amount, provides at once an analytical distillation curve of the material under treatment. Means suitable for this purpose are shown on the drawing in Fig. 1 and details thereof in Figs. 6 to 9 respectively.

As hereinbefore set forth, a pressure sensitive device or manometer 40 is provided in direct communication with the vapor line 37 leading to the receiver 38. This may suitably be a U-tube manometer, having a closed leg 40a communicating with the evacuated receiver 38 and an open leg 40b, and containing a column of mercury or other suitable conductive liquid which is connected through a conductor 70 with one pole of a suitable source of electricity.

As succesive increments of vapor pass from the distillation column into the receiver 38, they cause a corresponding increase in pressure therein, the contents of the receiver being maintained at a substantially constant temperature and in vapor phase. These increments of pressure are made to cause a corresponding feed of the paper record sheet 68 of the pyrometer 66, for example, in the following manner:

The paper feed roll 71 of the pyrometer, which feed roll bears projections 72 passing through notches 73 in the paper sheet 68 to effect a positive feed thereof is mounted upon a drive shaft 74 which is driven through suitable reduction gears 75 from the shaft 76 of motor 77. On the shaft 74 there is likewise mounted, exteriorly of the pyrometer 66, a soft rubber roll 78, which contacts closely with a similar soft rubber roll 79 mounted upon a countershaft 80. Extending into the open leg 40b of the manometer 40, there is provided an elongated metallic contact rod 81, the end of which is, in its normal position, but a very slight distance above the mercury column, say 0.1 mm. The rod 81 extends vertically upward and passes between the rubber rolls 78 and 79 so that, when the motor 77 is driven to operate the shaft 74 and move the paper sheet 68 of the recording pyrometer, the rubber roll

78 is driven and coacts with the rubber roll 79 to raise the contact rod 81.

As the distillation proceeds, the vapor is permitted to pass from the distillation column through the lines 31 and 37 into the receiver 38 and increases the pressure therein, in proportion to the amount of vapor entering the receiver, the increase in pressure is communicated through the line 39 to the manometer 40. The mercury is thereby caused to rise in the leg 40^b of the manometer and contact with the rod 81, whereupon a circuit is closed through the mercury, the contact rod 81, the connecteor 82, the motor 77, and the connector 83 to the opposite pole of the source of current supply. The motor 77 is thereby energized to operate the shaft 74 and the feed roll 71 for the paper sheet 68 of the recorder. In doing so, through the movement of the rubber roll 78, the contact rod 81 is raised and as soon as it breaks contact with the mercury in the manometer, the circuit through the motor 77 is broken and operation thereof ceases. At the same time that the movement of the paper sheet 68 has been effected in this manner, the recording arm 67 of the pyrometer has been operated to indicate upon the paper sheet the temperature in the top of the column 16. The paper sheet 68 is thereby made to bear a record showing, by movement of the recording pen transversely of the sheet, the temperature at which various vapor fractions are permitted to pass from the top of the column, and by markings longitudinally of the sheet, the quantity of such fractions. The resulting record is thereby directly in the form of the usual distillation curve with temperatures at which the respective vapor fractions are given off and their quantities as its coordinates. Thus, as distillation proceeds, during the passage from the column of one fraction, say a propane fraction, the top of the column is at substantially constant temperature while the successive increments of pressure in the fixed-volume receiver 38 resulting from the passage of the propane vapor thereinto causes a series of operations of the paper feeding motor 77, resulting in a longitudinal marking or succession of markings on the paper record sheet 68 at the same temperature. As the passage of propane reaches its end, and the temperature at the top rises as butanes begins to pass over, there being but a very small passage of vapor during the transition, the rise in temperature is indicated by a transverse marking on the paper record sheet 68 with a relatively slight or no longitudinal marking thereupon. When the temperature has risen to the point where butane passes over, during the passage of the butanes, it is substantially stationary and the operation is repeated with the position of the recorder arm indicating the higher temperature at which the butane passes over. It is to be understood, of course, that the references to propane and butane hereinbefore are merely illustrative.

In the event of an unexpected or undesired rise in pressure in the receiver 78, suitable signaling means may be provided in connection with the pressure sensitive device or manometer 40 to call the attention of an operator thereto. For this purpose, a metal contact rod 84 may be provided in the open leg 40^b of the manometer 40 with its end at a point substantially above the normal level of the mercury therein. If, through undue or unexpected increase in pressure in the column 38, the mercury level rises to the end of rod 84 and contact is made therewith, a circuit is closed through the connector 85, the signal device 86 and the connector 87 leading to the opposite pole of the source of current with which the mercury is connected. The signal device 86, which may consist of a buzzer or auditory signal, or a light or other visual signal, or both, is operated and the attention of the operator called to the unusual conditions existing within the container 38. In some cases, it may be necessary to cut off the receiver 38 from the remainder of the system, as by the valve 88, re-evacuate it to the line 41, which is then closed off, and re-set the contact rod 81 to a point very slightly above the newly established level of the mercury in the manometer leg 40^b before proceeding with the distillation.

In conducting the distillation, it is desirable that the rate of distillation should vary, being greater during the distillation of a pure or relatively pure component, during which period the temperature of distillation remains substantially constant, and being reduced when the temperature rises, as in transition from one component to another, the reduction in rate of distillation being desirably proportioned to the rate of rise of temperature. Considered from the standpoint of the distillation curve, the rate of distillation should be regulated so as to be inversely proportional to the slope or tangent of the fractional distillation curve. In the drawings, means are shown whereby such control of the rate of distillation can be secured.

In the operation in accordance with the present invention, the rate of distillation is controlled by the valve 33 between the vapor line 31 leading from the top of the column and the line 37 leading to the lower pressure receiver 38. It is obvious that by control of the valve 33, the pressure drop on the distillation column and consequently the rate of distillation can be controlled. As shown more particularly in Fig. 10, the valve member 33 may consist of a casing 88 into which the vapor line 31 leads, and with an outlet 89 to which the line 37 is connected. Within the casing 88, a suitable valve seat 90 is provided at the inner opening of the outlet 89, on which seats the needle valve 91 mounted on a reciprocable valve stem 92 which projects through the back of the casing and is operated by the lever 93. To avoid leakage, a metallic bellows diaphragm 94 surrounds the valve stem 92 and is securely attached at one end to the back of the casing 88 and at the other to the valve stem.

Suitable means are provided for controlling the movement of the valve 91 as a function of the rise in temperature at the outlet of the distillation column, the quantity of vapor received from the distillation column in the receiver 38 or both.

As pointed out hereinbefore, temperature changes in the top of the distillation column are communicated through the usual thermocouple connections to the recorder arm 67 on shaft 69 of the pyrometer 66. The shaft 69 carries, at the back of the instrument, as shown more particularly in Fig. 11, an arm 95, at the end of which is mounted the rotatable pin 96 carrying a spring-pressed pawl 97. The pawl 97 engages a ratchet wheel 98 rotatably mounted on an arbor 99 carried in a suitable bearing on the support 100. At a slight distance from

the ratchet wheel 98 and in a parallel plane with respect thereto, there is provided a worm wheel 101 mounted on an arbor 102 coaxial with the arbor 99 of the ratchet wheel 98. The arbor 102 is mounted in a suitable bearing on the support 103. Contact arms 104 and 105 are mounted on the ratchet wheel 98 and the worm wheel 101 respectively, these contact arms being normally spaced from each other, but being adapted to engage on movement of the ratchet wheel 98. On the outer sides of the ratchet wheel 98 and worm wheel 101, the spring contacts 106 and 107 respectively are provided, for an electrical connection as hereinafter pointed out.

The worm wheel 101 meshes with and is driven by a worm 108 on a shaft 109 which is driven through suitable reduction gears 110 from motor 111. The shaft 109 extends through a sleeve cap 112 provided with a constricted portion 113, and extends through the cylindrical casing 104 of the actuating or tangent-control member, generally designated by the numeral 115. The cylinder 114, which is threaded at one end into the sleeve cap 113, is provided at its opposite end with a reduced extension 116 provided internally with a bore 117 into which the shaft 109 projects. The reduced portion 116 is extended as a cylindrical shaft member 118 which passes through a bearing 119 in a suitable support member 120. Within the cylindrical casing 114 of the actuating member 115, there is fixed to the shaft member 109 an externally screw-threaded collar 121, the threads of which mate with threads 122 formed internally of the cylindrical casing 114. The actuating or control member and its shaft extension 108 are axially movable with respect to shaft 109 and bearing 119.

The shaft extension 118 of the cylindrical casing 114 of the actuating mechanism 115 is provided at its outer end with a pulley 123 which is driven from a pulley 124 mounted on a shaft 124^a forming a part of the shaft of motor 77 and extending in the opposite direction from the shaft portion 124.

As pointed out hereinbefore, on rise in temperature in the top of the column 16, the recorder arm 67 of the pyrometer is moved clockwise. This movement is imparted through the shaft 67 to the arm 95 carrying the pawl 97, which is spring-pressed against the ratchet wheel 98. As a result, on rise in temperature in the column, the ratchet wheel 98 is caused to move, and the contact arm 104 is thereby brought into contact with the contact arm 105 on the worm wheel 101. When this takes place a circuit is closed from one pole of a source of current through connector 125, arm 106, ratchet wheel 98, contact arms 104 and 105, worm wheel 101, contact 107, connector 126, motor 111 and connector 127 to the opposite pole of the current source. The motor 111 is thereby energized and rotates the shaft 109. The rotation of the shaft 109 is communicated through the threaded collar 121 to the cylindrical casing 114 of the actuating mechanism 115, thereby causing axial movement of the latter which in turn is communicated to the forked end 128 of lever 93 (which embraces the constricted portion 113 of the sleeve cap 112), thereby tending to move the needle valve 91 toward its seat and reduce or close the opening between the vapor line 31 and the line 37 leading to the receiver 38. At the same time, the movement of the shaft 109, acting through the worm 108, rotates the worm wheel 101, thereby separating the contacts 105 and 104 and breaking the hereinbefore described circuit to the motor 111. In the event of further rise in temperature, the operation is repeated.

The actuating cylinder 116 is likewise affected by the increase in pressure in the vapor receiver 38 resulting from the receipt thereinto of vapors from the distillation column. As hereinbefore set forth, such increase in pressure results in a corresponding energizing of the motor 77, from which the paper feed mechanism for the recorder 66 is driven. From the motor shaft extension 124^a of the motor 77, drive pulley 124 causes corresponding movement of a pulley 123 on the shaft extension 118 of the actuating cylinder 115, causing it to rotate in such manner that if, at the time, the threaded collar 121 within the actuating cylinder is stationary, the axial movement of the actuating cylinder tends to open the valve and increase the rate of distillation. As hereinbefore described in connection with the recording mechanism, this same operation of motor 77 causes movement of contact rod 81, which breaks the current controlling motor 77. On further rise in pressure in receiver 38, the operation is repeated.

It will be clear from the above description of the operation of the actuating cylinder 115 or the operating lever 93 of the distillation control valve 33 that it is individually responsive to the quantities of individual vapor components discharged from the distillation column 16 and to changes in temperature in the column at the point of discharge, and mechanically compounds the effects due to both of these causes. As a result of proper selection of gear ratios, screw pitches and the like, the relative effect of each of these elements controlling the cylinder 115 and its movement may be modified, although, as hereinbefore stated, it is preferred that its response be so controlled as to correspond substantially and sensitively to changes in the tangent or slope of the distillation curve.

It will likewise be readily apparent that the operation of the actuating cylinder 115 and its effect upon the distillation control valve are closely interrelated with the operation of the control mechainsm for the supply of cooling liquid to the distillation column. For example, if the actuating cylinder 115 is operated to close or restrict the opening in valve 33 by a decrease in flow of the vapor component discharged from the column and an increase in temperature at the top of the column, the restriction of the valve 33 results in an increase in pressure in the column which is communicated to the pressure responsive device or manometer 36 and, through the mechanism hereinbefore described, causes an increase in the amount of cooling liquid supplied to the top of the column. Similarly, when, by a diminished rate of increase of temperature of the column or cessation of increase of temperature, and an increased flow of a vapor component discharged from the column, or both, the valve 33 is opened to a greater extent, there is a corresponding diminution of pressure on the column, which, acting through the pressure responsive device 36, causes a decreased supply of reflux cooling liquid to the column.

Although the present invention has been described in connection with the details of specific

method and apparatus embodying the same, it is not intended that these details shall be regarded as limitations upon the scope of the invention, except in so far as included in the accompanying claims.

I claim:

1. In apparatus for precise fractional distillation, a fractional distillation column, means for applying heat to the lower portion of the column, means for providing a cooling medium in cooling relationship to the upper portion of the column, and means for controlling the supply of cooling medium thereto, said controlling means including a manometer communicating with the outlet of said column, said manometer containing an electrically conductive liquid, a contact member in said manometer adapted to be contacted by the liquid therein on increase of pressure in the distillation column, and an electrical circuit closed on said contact to operate said means for controlling the supply of cooling fluid.

2. In apparatus for precise fractional distillation, a fractional distillation column, means for applying heat to the lower portion of the column, means for providing a cooling medium in cooling relationship to the upper portion of the column, and means for controlling the supply of cooling medium thereto, said controlling means including a valve for controlling the supply of cooling fluid to the distillation column, a normally open electrical circuit for controlling the operation of said valve, a manometer communicating with the outlet of said column, said manometer containing an electrically conductive liquid constituting a contact member for said circuit, and a second contact member adapted to be engaged by said liquid on change in pressure in the column to close said valve control circuit.

3. In distillation apparatus, a distillation column having means for supplying heat at its lower end, means for cooling the column at its upper end, said means comprising a vessel for cooling fluid in heat-conductive relationship to the upper portion of the column, a supply container for the cooling fluid, means for conducting the cooling fluid from said container to the cooling vessel, means for supplying a pressure fluid to the supply container to force the cooling medium into said vessel, a valve for controlling the supply of pressure fluid, pressure-responsive means connected with and sensitive to the pressure at the outlet of the column, and means operable thereby on change of pressure within the column to operate said valve to control the supply of pressure fluid to the supply container for the cooling medium.

4. In distillation apparatus, a distillation column having means for supplying heat at its lower end, means for cooling the column at its upper end, said means comprising a vessel for cooling fluid in heat-conductive relationship to the upper portion of the column, a supply container for the cooling fluid, means for conducting the cooling fluid from said container to the cooling vessel, means for supplying a pressure fluid to the supply container to force the cooling medium into said vessel, a valve for controlling the supply of pressure fluid, pressure-responsive means connected with and sensitive to the pressure at the outlet of the column, and means operable thereby on rise of pressure within the column to open said valve to increase the supply of pressure fluid to the supply container for the cooling fluid.

5. In distillation apparatus, a distillation column having means for supplying heat at its lower end, means for cooling the column at its upper end, said means comprising a vessel for cooling fluid in heat-conductive relationship to the upper portion of the column, a supply container for the cooling fluid, means for conducting the cooling fluid from said container to the cooling vessel, means for supplying a pressure fluid to the supply container to force the cooling medium into said vessel, a valve for controlling the supply of pressure fluid, a normally open electrical circuit for operating said valve, a manometer communicating with the outlet of the column, said manometer containing an electrically conductive liquid constituting a contact for said circuit, and an electrical contact within the manometer, adapted to be engaged by the conductive liquid in the manometer on rise of pressure at the outlet of the column to close said electrical circuit to operate said valve to control the supply of pressure fluid to the supply container for the cooling medium.

6. In distilling apparatus, a distillation column having means for supplying heat at its lower end, means for cooling the column at its upper end, said means comprising a vessel for cooling fluid in heat-conductive relationship to the upper portion of the column, a supply container for the cooling fluid, means for conducting the cooling fluid from said container to the cooling vessel, means for supplying a pressure fluid to the supply container to force the cooling medium into said vessel, a valve for controlling the supply of pressure fluid, means venting excess of pressure fluid from said supply container, pressure-responsive means connected with the outlet of the column, and means operable thereby on rise of pressure within the column to operate said valve to increase the supply of pressure fluid to said supply container for the cooling medium.

7. In apparatus for precise fractional distillation, a distilling column having heating means at the lower portion thereof, cooling means at the upper portion thereof, a vapor outlet line connecting with the upper portion of the column, a valve in said vapor outlet line, means responsive to pressure change at the outlet of said column to control said cooling means, and means responsive to pressure drop in said column to close said valve.

8. In apparatus for precise fractional distillation, a distilling column having heating means at the lower portion thereof, a vapor outlet line connecting with the upper portion of the column, a valve in said vapor outlet line, means for supplying a cooling fluid in heat-conductive relationship with the upper portion of said column, said means including a control valve, a pressure-responsive device communicating with the outlet of the column, means actuated by said pressure-responsive device on increase in pressure at the outlet of said column to open said control valve, and means actuated by said pressure-responsive device on decrease in pressure in said column to close the valve in said vapor outlet conduit.

9. In apparatus for precise fractional distillation, a distilling column having heating means at the lower portion thereof, a vapor outlet line connecting with the upper portion of the column, a valve in said vapor outlet line, means for supplying a cooling fluid in heat-conductive relationship with the upper portion of said column,

said means including a supply container for cooling medium, a conduit for supplying a pressure fluid to said container to force the cooling fluid therefrom to the column, a valve in said pressure medium conduit, a pressure-responsive device communicating with the outlet of said column, means actuated by said pressure responsive device on increase in pressure at the outlet of said column to open the valve in said pressure fluid supply conduit and means actuated by said pressure-responsive device on decrease in pressure in said column to close the valve in said vapor outlet conduit.

10. In apparatus for precise fractional distillation, a distilling column having heating means at the lower portion thereof, a vapor outlet line connecting with the upper portion of the column, a valve in said vapor outlet line, said valve including a normally open electrical control circuit, means for supplying a cooling fluid in heat-conductive relationship with the upper portion of said column, a control valve therefor, a normally open electrical control circuit therefor, a manometer communicating with the outlet of the column, said manometer containing an electrically conductive liquid constituting a contact for each of said circuits, an electrical contact within the manometer adapted to be engaged by the conductive liquid therein on rise of pressure in the outlet of the column to close said last mentioned electrical circuit to increase the supply of cooling fluid to the column, and an electrical contact within the manometer adapted to be engaged by the conductive liquid therein on decrease of pressure at the outlet of the column to close said first mentioned electrical circuit and close the valve in the vapor outlet line.

11. In apparatus for precise fractional distillation, a distillation column, means for applying heat at the lower end of the column, means for cooling the upper portion of the column, a vapor outlet leading from the upper portion of the column, a valve in said outlet line for controlling the flow of vapor therethrough, and means responsive to the amount of vapor passing through the vapor outlet line to control said valve.

12. In apparatus for fractional distillation, a distillation column, means for applying heat at the lower end of the column, means for cooling the upper portion of the column, a vapor outlet leading from the upper portion of the column, a valve in said outlet line for controlling the flow of vapor therethrough, and means responsive to the amount of vapor passing through the vapor outlet to actuate said valve, to open it on increase in the amount of vapor passing therethrough and to close said valve on decrease in amount of vapor passing therethrough.

13. In apparatus for precise fractional distillation, a distillation column, means for applying heat at the lower end of the column, means for cooling the upper portion of the column, a vapor outlet leading from the upper portion of the column, a valve in said outlet line for controlling the flow of vapor therethrough, a receiver into which said vapor line discharges, and means responsive to pressure changes within the receiver to control said valve.

14. In apparatus for precise fractional distillation, a distillation column, means for applying heat at the lower end of the column, means for cooling the upper portion of the column, a vapor outlet leading from the upper portion of the column, a valve in said outlet line for controlling the flow of vapor therethrough, a receiver into which said vapor line discharges, means responsive to pressure changes within the receiver to control said valve, said pressure-responsive means comprising a pressure-responsive device, a motor, an electrical circuit for said motor adapted to be closed by said pressure-responsive device, and means operated by said motor to control said valve.

15. In apparatus for precise fractional distillation, a distillation column, means for applying heat at the lower end of the column, means for cooling the upper portion of the column, a vapor outlet leading from the upper portion of the column, a valve in said outlet line for controlling the flow of vapor therethrough, a receiver into which said vapor line discharges, means responsive to pressure changes within the receiver to control said valve, said pressure-responsive means comprising a pressure-responsive device, a motor, an electrical circuit for said motor adapted to be closed by said pressure-responsive device, and means operated by said motor to operate said valve and to interrupt the motor circuit.

16. In apparatus for precise fractional distillation, a distillation column, means for applying heat at the lower end of the column, means for cooling the upper portion of the column, a vapor outlet leading from the upper portion of the column, a valve in said outlet line for controlling the flow of vapor therethrough, means for operating said valve including a normally open electrical control circuit, a receiver into which the vapor line discharges, pressure-responsive means connected to the receiver and adapted to close said circuit on rise in pressure in said receiver, thereby operating said valve.

17. In apparatus for fractional distillation, a distilling column having heating means at the lower portion thereof, means for cooling the upper portion of the column, a vapor outlet from the column, a control valve in said vapor outlet adapted to vary the rate of flow of vapors therethrough, means for operating said control valve. said means being responsive to the amount of vapors passing therethrough, and means responsive to pressures at the outlet of the column for controlling the said cooling means.

18. In apparatus for fractional distillation, a distilling column having heating means at the lower portion thereof, means of cooling the upper portion of the column, a vapor outlet from the column, a control valve in said vapor outlet adapted to vary the rate of flow of vapors therethrough, means for operating said control valve effective to cause an opening movement thereof on increase in amount of vapors passing therethrough and a closing movement of said valve on a decrease in amount of vapors passing therethrough, and means responsive to pressures at the outlet of the column for controlling the said cooling means.

19. In apparatus for fractional distillation, a distilling column and means for applying heat to the lower portion thereof, means for supplying a cooling medium in heat conductive relationship to the upper portion of the column, a vapor outlet line leading from said column, a control valve in said line adapted to control the rate of flow of vapors therethrough, and thereby modify pressure conditions at the outlet of the column, a receiver into which said vapor line discharges thereby varying the pressure therein, means responsive to pressure changes in said

receiver for controlling the operation of said control valve, and means responsive to pressure at the outlet of said column to control the supply of cooling medium to the upper portion thereof.

20. In apparatus for fractional distillation, a distilling column and means for applying heat to the lower portion thereof, means for supplying a cooling medium in heat conductive relationship to the upper portion of the column, a vapor outlet line leading from said column, a control valve in said line adapted to control the rate of flow of vapors therethrough, and thereby modify pressure conditions at the outlet of the column, a receiver into which said vapor line discharges thereby varying the pressure therein, means responsive to pressure changes in said receiver for controlling the operation of said control valve, said means including a normally open electrical control circuit for said control valve and a pressure-responsive device connected to the receiver and adapted to close said circuit on rise in pressure in said receiver, thereby operating said valve, and means responsive to pressure at the outlet of said column to control the supply of cooling medium to the upper portion thereof.

21. In apparatus for fractional distillation, a distilling column and means for applying heat to the lower portion thereof, means for supplying a cooling medium in heat conductive relationship to the upper portion of the column, a vapor outlet line leading from said column, a control valve in said line adapted to control the rate of flow of vapors therethrough, and thereby modify pressure conditions at the outlet of the column, a receiver into which said vapor line discharges, thereby varying the pressure therein, means responsive to pressure changes in said receiver for controll ng the operation of said control valve, said means including a motor, a normally open electrical circuit therefor, a pressure-responsive device connected to the receiver and adapted to close said circuit on rise in pressure in said receiver, thereby operating said valve, and means responsive to pressure at the outlet of said column to control the supply of cooling medium to the upper portion thereof.

22. In apparatus for fractional distillation, a distilling column and means for supplying heat to the lower portion thereof, means for cooling the upper portion of said column, a vapor outlet line leading from the upper portion of said column, a control valve in said vapor outlet line, said control valve being operable to vary the rate of flow of vapors through said line, and means responsive to the temperature at the outlet of said column to operate said control valve, and means responsive to pressures at the outlet of said column to control said cooling means.

23. In apparatus for fractional distillation, a distilling column, means for supplying heat to the lower portion thereof, means for supplying a cooling medium in heat conductive relationship to the upper portion thereof, a vapor outlet line leading from said column, a control valve in said vapor outlet line, said control valve being adapted to vary the rate of flow therethrough, means responsive to temperature conditions in the upper portion of said column for operating said control valve, and means responsive to pressure conditions at the outlet of said column for controlling the supply of cooling medium in heat conductive relationship therewith.

24. In apparatus for fractional distillation, a distilling column, means for supplying heat to the lower portion thereof, means for supplying a cooling medium in heat conductive relationship to the upper portion thereof, a vapor outlet line leading from said column, a control valve in said vapor outlet line, said control valve being adapted to vary the rate of flow therethrough, means responsive to temperature conditions in the upper portion of said column for operating said control valve, said means including a movable member responsive to changes in temperature in the upper portion of the column, and means operated by said movable member on rise in temperature in the column to reduce the opening in said control valve, and means responsive to pressure conditions at the outlet of said column for controlling the supply of cooling medium in heat conductive relationship therewith.

25. In apparatus for fractional distillation, a distilling column, means for supplying heat to the lower portion thereof, means for supplying a cooling medium in heat conductive relationship to the upper portion thereof, a vapor outlet line leading from said column, a control valve in said vapor outlet line, said control valve being adapted to vary the rate of flow therethrough, means responsive to temperature conditions in the upper portion of said column for operating said control valve, said means including an actuating member, a movable member responsive to temperature changes in the upper portion of said column, and means operable by said movable member on rise in temperature in the column to cause said actuating member to operate said valve, and means responsive to pressure conditions at the outlet of said column for controlling the supply of cooling medium in heat conductive relationship therewith.

26. In apparatus for fractional distillation, a distilling column, means for supplying heat to the lower portion of said column, means for cooling the upper portion of the column, a vapor outlet line leading from the upper portion of the column, a control valve in said vapor line adapted to control the rate of flow of vapors therethrough, and means responsive to temperature conditions in the upper portion of said column for operating said control valve.

27. In apparatus for fractional distillation, a distilling column, means for supplying heat to the lower portion of said column, means for cooling the upper portion of the column, a vapor outlet line leading from the upper portion of the column, a control valve in said vapor line adapted to control the rate of flow of vapors therethrough, and means for operating said control valve, said means including a movable member responsive to changes in temperature in the upper portion of the column, and means operated by said movable member on rise in temperature in the column to reduce the opening in said control valve.

28. In apparatus for fractional distillation, a distilling column, means for supplying heat to the lower portion of said column, means for cooling the upper portion of the column, a vapor outlet line leading from the upper portion of the column, a control valve in said vapor line adapted to control the rate of flow of vapors therethrough, and means for operating said control valve, said means including an actuating member, a movable member responsive to temperature changes in the upper portion of said column, and means operable by said movable mem-

ber on rise in temperature in the column to cause said actuating member to operate said valve.

29. In apparatus for fractional distillation, a distilling column, means for supplying heat to the lower portion of said column, means for cooling the upper portion of the column, a vapor outlet line leading from the upper portion of the column, a control valve in said vapor line adapted to control the rate of flow of vapors therethrough, and means for operating said control valve, said means including an actuating member, and means for causing movement of said actuating member, said means including a normally open electrical circuit and contact means for said electrical circuit operated on movement of said movable member on rise in temperature in the column to close said circuit, thereby causing said actuating means to operate said control valve.

30. In apparatus for fractional distillation, a distilling column, means for supplying heat to the lower portion of said column, means for cooling the upper portion of the column, a vapor outlet line leading from the upper portion of the column, a control valve in said vapor line adapted to control the rate of flow of vapors therethrough, and means for operating said control valve, said means including an actuating member, said means for causing movement of said actuating member, said means including a normally open electrical circuit and contact means for said electrical circuit operated on movement of said movable member on rise in temperature in the column to close said circuit, thereby causing said actuating means to operate said control valve, and means operated simultaneously with the operation of said actuating means to separate said contacts and break said circuit.

31. In apparatus for fractional distillation, a distilling column having means for applying heat to the lower portion thereof and means for cooling the upper portion thereof, a vapor line leading from the upper portion of said column, a control valve in said vapor line for controlling the rate of flow of vapors through said outlet line, and means coordinately responsive to the amount of vapors flowing through said line and to the temperature in the upper portion of the column for operating said control valve.

32. In apparatus for fractional distillation, a distilling column having means for applying heat to the lower portion thereof and means for cooling the upper portion thereof, a vapor line leading from the upper portion of said column, a control valve in said vapor line for controlling the rate of flow of vapors through said outlet line, and means for operating said control valve, said means being coordinately responsive to the temperature in the upper portion of the column and to the amount of vapors flowing through said line to enlarge the opening in the valve on increase in the amount of vapors flowing through said line and to constrict said opening on decrease thereof.

33. In apparatus for fractional distillation, a distilling column having means for applying heat to the lower portion thereof and means for cooling the upper portion thereof, a vapor line leading from the upper portion of said column, a control valve in said vapor line for controlling the rate of flow of vapors through said outlet line, said vapor line discharging into a receiver, and means coordinately responsive to changes of pressure in said receiver and to changes of temperature in the outlet of the column for operating said control valve.

34. In apparatus for fractional distillation, a distilling column having means for applying heat to the lower portion thereof and means for cooling the upper portion thereof, a vapor line leading from the upper portion of said column, a control valve in said vapor line for controlling the rate of flow of vapors through said outlet line, and actuating mechanism for operating said control valve, said actuating mechanism comprising a movable element operable in response to the amount of vapor passing through said outlet line, and a movable member responsive to changes in temperature at the outlet of the column.

35. In apparatus for fractional distillation, a distilling column having means for applying heat to the lower portion thereof and means for cooling the upper portion thereof, a vapor line leading from the upper portion of said column, a control valve in said vapor line for controlling the rate of flow of vapors through said outlet line, and actuating mechanism for operating said control valve, said actuating mechanism comprising a movable element operable in response to changes of pressure in said receiver and a movable member responsive to changes in temperature at the outlet of the column.

36. In apparatus for fractional distillation, a distilling column having means for applying heat to the lower portion thereof and means for supplying a cooling medium in heat conductive relationship to the upper portion of the column, a vapor line leading from the upper portion of said column, a control valve in said vapor line for controlling the rate of flow of vapors through said outlet line, and means coordinately responsive to the amount of vapors flowing through said line and to the temperature in the upper portion of the column for operating said control valve, and means responsive to pressure changes at the outlet of the column for controlling the supply of cooling medium thereto.

37. In apparatus for fractional distillation, a distilling column having means for applying heat to the lower portion thereof and means for supplying a cooling medium in heat conductive relationship to the upper portion of the column, a vapor line leading from the upper portion of said column, a control valve in said vapor line for controlling the rate of flow of vapors through said outlet line, said vapors discharging into a receiver, and means coordinately responsive to the amount of change of pressure in said receiver and to the temperature in the upper portion of the column for operating said control valve, and means responsive to pressure changes at the outlet of the column for controlling the supply of cooling medium thereto.

38. In apparatus for fractional distillation, a distilling column having means for applying heat to the lower portion thereof and means for supplying a cooling medium in heat conductive relationship to the upper portion of the column, a vapor line leading from the upper portion of said column, a control valve in said vapor line for controlling the rate of flow of vapors through said outlet line, and actuating mechanism for operating said control valve, said actuating mechanism comprising a movable element operable in response to the amount of vapor passing through said outlet line, and a movable member responsive to changes in temperature at the outlet of the column, and means

responsive to pressure changes at the outlet of the column for controlling the supply of cooling medium thereto.

39. In apparatus for fractional distillation, a distilling column having means for applying heat to the lower portion thereof and means for supplying a cooling medium in heat conductive relationship to the upper portion of the column, a vapor line leading from the upper portion of said column, a control valve in said vapor line for controlling the rate of flow of vapors through said outlet line, and actuating mechanism for operating said control valve, said actuating mechanism comprising a movable element operable in response to changes of pressure in said receiver and a movable member responsive to changes in temperature at the outlet of the column, and means responsive to pressure changes at the outlet of the column for controlling the supply of cooling medium thereto.

40. In distillation apparatus, a distilling column and means for controlling the operation of said column, said means comprising a control member, and operating mechanism therefor including an actuating device, said actuating device having a movable element, and means for imparting motion thereto, said means including an electrical motor mechanism and a normally open electrical circuit therefor, a pressure-responsive device providing a contact for said circuit, a movable contact member providing a second contact for said circuit, said movable contact member being normally separated from said first mentioned contact member and adapted to be engaged thereby on movement of the pressure-responsive device, thereby closing the circuit to said motor.

41. In distillation apparatus, a distilling column and means for controlling the operation of said column, said means comprising a control member, and operating mechanism therefor including an actuating device, said actuating device having a movable element, and means for imparting motion thereto, said means including an electrical motor mechanism and a normally open electrical circuit therefor, a pressure-responsive device providing a contact for said circuit, a movable contact member providing a second contact for said circuit, said movable contact member being normally separated from said first mentioned contact member and adapted to be engaged thereby on movement of the pressure-responsive device, thereby closing the circuit to said motor, and means operated by said motor engaging said movable contact member to separate it from the first mentioned contact member.

42. In distillation apparatus, a distilling column and means for controlling the operation of said column, said means comprising a control member, and operating mechanism therefor including an actuating device, said actuating device having a movable element, and means for imparting motion thereto, said means including an electrical motor mechanism and a normally open electrical circuit therefor, a pressure-responsive device providing a contact for said circuit, a movable contact member providing a second contact for said circuit, said movable contact member being normally separated from said first mentioned contact member and adapted to be engaged thereby on movement of the pressure-responsive device, thereby closing the circuit to said motor, and means operated by said motor engaging said movable contact member to separate it from the first mentioned contact member, said means including a shaft driven from said motor, a roll member carried on said shaft, an opposing roll member, a countershaft on which said opposing roll member is carried, said roll members engaging the movable contact member to effect movement thereof.

43. In distillation apparatus, a distilling column and means for controlling the operation of said column, said means comprising a control member, and operating mechanism therefor including a movable actuating device, said actuating mechanism comprising a movable member, and means for imparting movement thereto, said means comprising a normally open electrical control circuit, a movable member actuated by rise in temperature in the upper portion of the column, contact means carried by said movable member to close said electrical circuit, and means effective on operation of said first mentioned movable member to break said electrical control circuit and thereby render said movement-imparting means inoperative.

44. In distillation apparatus, a distillation column, a vapor outlet line therefor, a valve in said line for controlling the operation of said column, means for operating said valve to vary the flow of vapors through the outlet line, said means comprising a valve operating member, actuating means therefor comprising a movable member, means for imparting movement thereto, said means comprising a member responsive to the amount of vapor passing through said outlet line, and a second movable member coordinately connected with said first movable member, and means for operating said second movable member, said means including a member responsive to temperature changes in the upper portion of the column.

45. In distillation apparatus, a distillation column, a vapor outlet line therefor, a valve in said line for controlling the operation of said column, means for operating said valve to vary the flow of vapors to the outlet line, said means comprising a valve operating member, actuating means therefor comprising a movable member, means for operating said movable member, said means including a normally open electrical control circuit, and means responsive to the amount of vapor passing through said vapor line for closing said control circuit, a second movable member coordinately connected with the first movable member, means for operating said second movable member, said means comprising a normally open electrical circuit, and means responsive to temperature changes at the outlet of the column for closing said second electrical control circuit.

46. In distillation apparatus, a distillation column, a vapor outlet line therefor, a valve in said line for controlling the operation of said column, means for operating said valve to vary the flow of vapors to the outlet line, said means comprising a valve operating member, actuating means therefor comprising a movable member, means for operating said movable member, said means including a normally open electrical control circuit, and means responsive to the amount of vapor passing through said vapor line for closing said control circuit, and means operating to open said electrical control circuit on operation of said movable member, a second movable member coordinately connected with the first

movable member, means for operating said second movable member, said means comprising a normally open electrical circuit, and means responsive to temperature changes at the outlet of the column for closing said second electrical control circuit.

47. In distillation apparatus, a distillation column, a vapor outlet line therefor, a valve in said line for controlling the operation of said column, means for operating said valve to vary the flow of vapors to the outlet line, said means comprising a valve operating member, actuating means therefor comprising a movable member, means for operating said movable member, said means including a normally open electrical control circuit, and means responsive to the amount of vapor passing through said vapor line for closing said control circuit, a second movable member coordinately connected with the first movable member, means for operating said second movable member, said means comprising a normally open electrical circuit, and means responsive to temperature changes at the outlet of the column for closing said second electrical control circuit, and means operating to open said electrical control circuit on operation of said movable member.

48. In combination with a distillation column, means for receiving vapors delivered therefrom and retaining said vapors in vapor form whereby the amount of vapors received is indicated by changes of pressure therein, and recording means comprising a temperature-responsive member, a thermo-coupled connection from said column to said temperature responsive member, a pressure-responsive member, and means interconnecting said pressure responsive member with said receiver.

49. In combination with a distillation column, means for receiving vapors delivered therefrom and retain said vapors in vapor form whereby the amount of vapors received is indicated by changes of pressure therein, and recording means comprising a temperature responsive member, said temperature responsive member having a movable recorder arm, a thermo-couple circuit from said column to said temperature responsive means, whereby the recording made by said arm indicates the temperature in said column, and means for feeding a paper sheet in position to be engaged by said arm, said means including a pressure-sensitive member, and means connecting said pressure-sensitive member to said receiver whereby the longitudinal movement of said paper is controlled by the amount of vapor received therein.

50. In combination with a distillation column, means for receiving vapors delivered therefrom and retain said vapors in vapor form whereby the amount of vapors received is indicated by changes of pressure therein, and recording means comprising a temperature responsive member, said temperature responsive member having a movable recorder arm, a thermo-couple circuit from said column to said temperature responsive means, whereby the recording made by said arm indicates the temperature in said column, and means for feeding a sheet of paper in position to be engaged by said recorder arm, said means including a drive shaft, means for rotating said shaft, said means including a motor and a normally open control circuit therefor, a pressure-sensitive device interconnected with said receiver, said pressure-sensitive device having a contact means for said control circuit, and a second contact member adapted to be engaged by the first mentioned contact member to close said circuit on rise of pressure in said receiver.

51. In combination with a distillation column, means for receiving vapors delivered therefrom and retain said vapors in vapor form whereby the amount of vapors received is indicated by changes of pressure therein, and recording means comprising a temperature responsive member, said temperature responsive member having a movable recorder arm, a thermo-couple circuit from said column to said temperature responsive means, whereby the recording made by said arm indicates the temperature in said column, and means for feeding a sheet in position to be engaged by said recording arm, said means including a drive shaft, means for rotating said shaft, said means including a motor and a normally open control circuit therefor, a pressure-sensitive device interconnected with said receiver, said pressure-sensitive device having a contact means for said control circuit, and a movable rod forming a second contact member for said circuit and adapted to be engaged by said first mentioned contact on increase of pressure in said receiver to close said control circuit and thereby energize said motor and actuate said feeding means.

52. In combination with a distillation column, means for receiving vapors delivered therefrom and retain said vapors in vapor form whereby the amount of vapors received is indicated by changes of pressure therein, and recording means comprising a temperature responsive member, said temperature responsive member having a movable recorder arm, a thermo-couple circuit from said column to said temperature responsive means, whereby the recording made by said arm indicates the temperature in said column, and means for feeding a sheet of paper in position to be engaged by said recorder arm, said means including a drive shaft, means for rotating said shaft, said means including a motor and a normally open control circuit therefor, a pressure-sensitive device interconnected with said receiver, said pressure-sensitive device having a contact means for said control circuit, a movable rod constituting a second contact member for said control circuit, whereby on increase of pressure in said receiver, said first mentioned contact means engages said movable rod to close said control circuit and energize said motor, thereby operating said shaft to feed the paper, and means carried by said shaft for moving said rod, thereby breaking contact with said first mentioned contact member and opening said circuit to de-energize said motor.

53. In combination with a distillation column, means for receiving vapors delivered therefrom and retaining said vapors in vapor form whereby the amount of vapors received is indicated by changes of pressure therein, a pressure-responsive member connected with the receiver, a temperature-responsive means, a thermocouple connection from the column to said temperature-responsive means, a control valve between the column and the vapor-receiving means, recording means operated by said pressure-responsive member and said temperature-responsive means to record pressure and temperature and to operate said control valve.

WALTER J. PODBIELNIAK.