Oct. 26, 1954 C. D. ALWAY ET AL 2,692,820

METHOD AND MEANS FOR EFFECTING AUTOMATIC FRACTIONATION

Filed Oct. 28, 1950

10 Sheets-Sheet 1

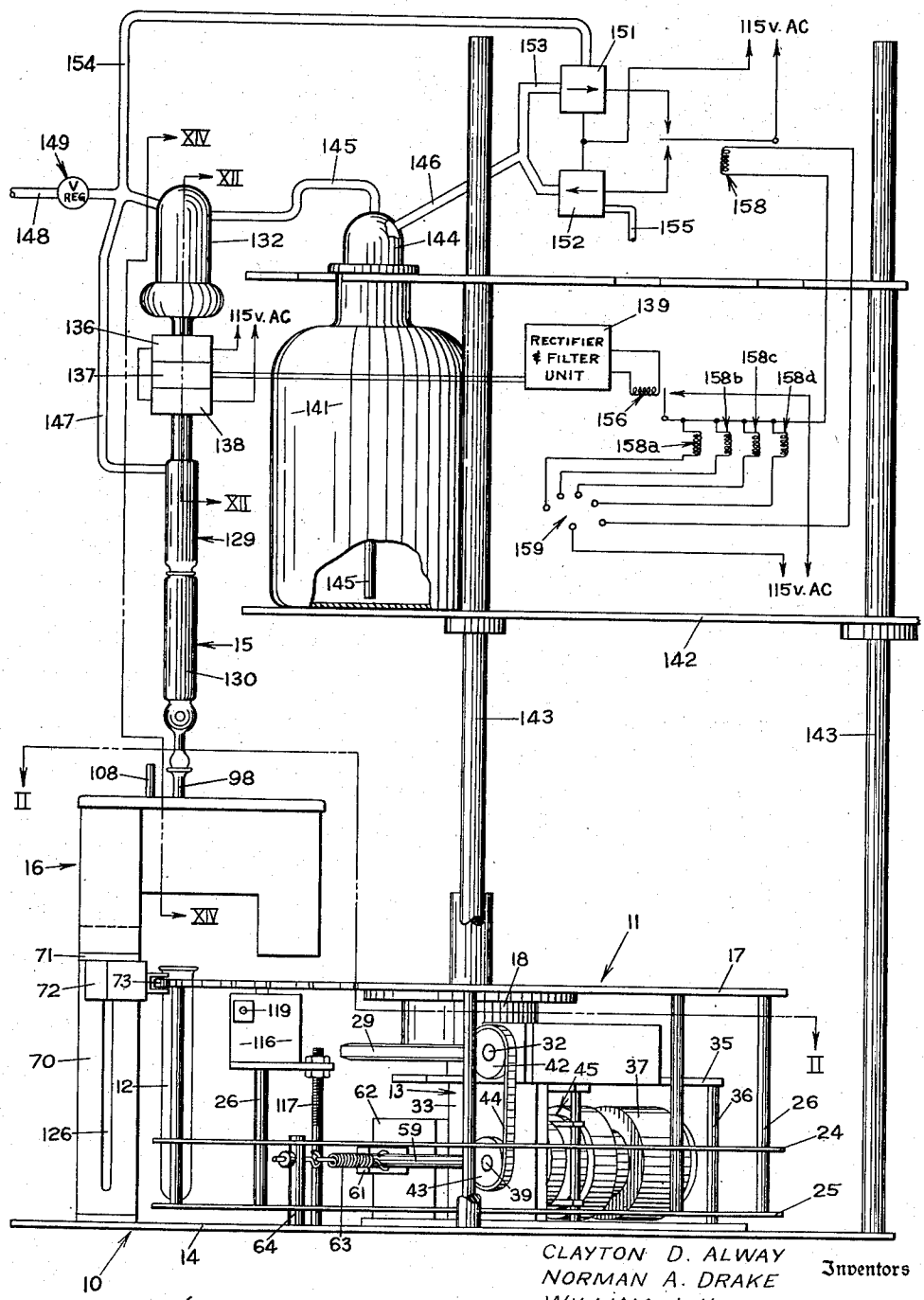


fig. 1

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METHOD AND MEANS FOR EFFECTING AUTOMATIC FRACTIONATION

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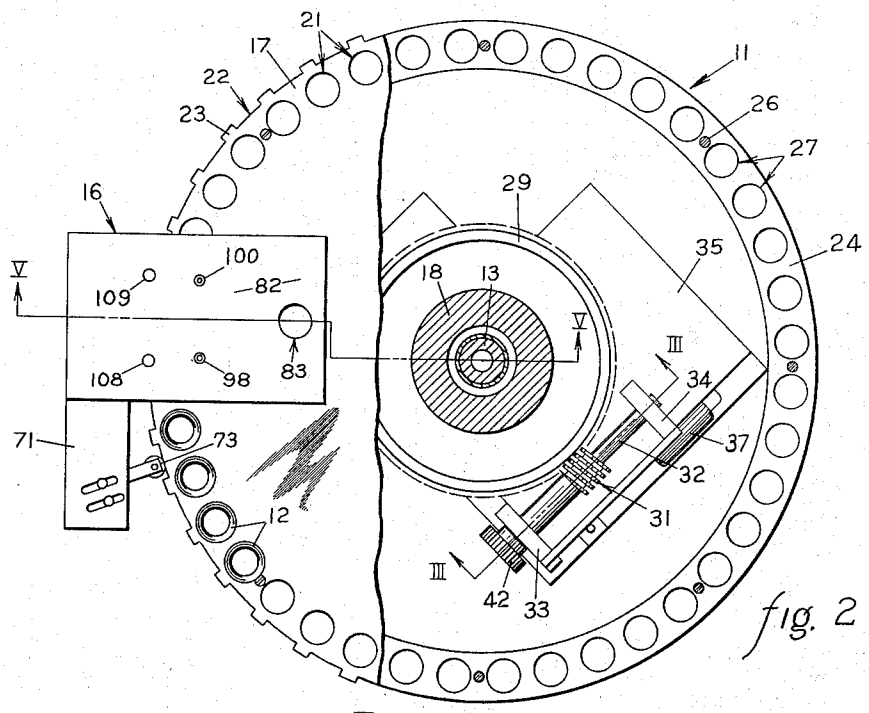


fig. 2

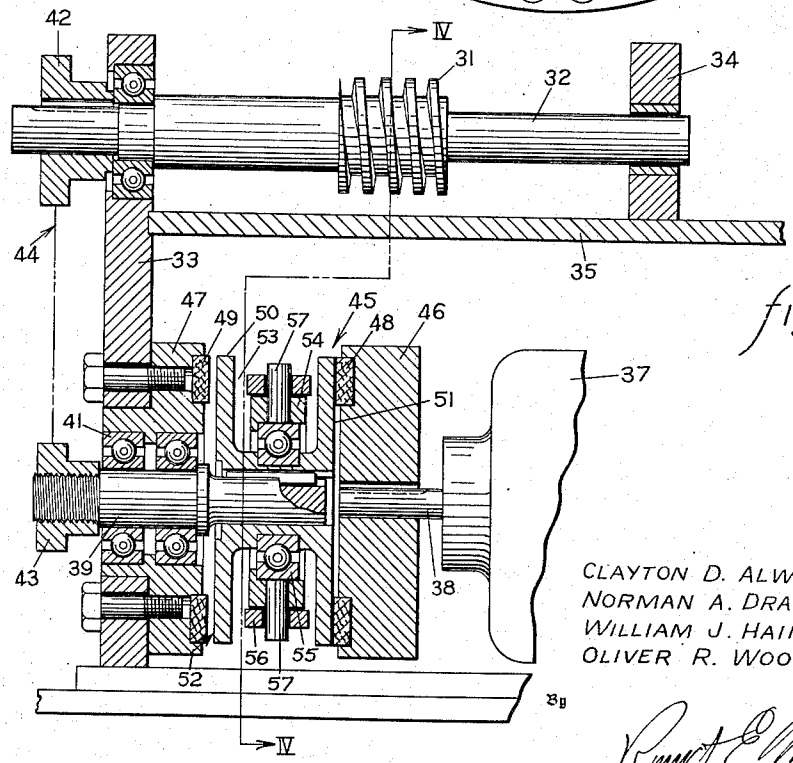


fig. 3

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METHOD AND MEANS FOR EFFECTING AUTOMATIC FRACTIONATION

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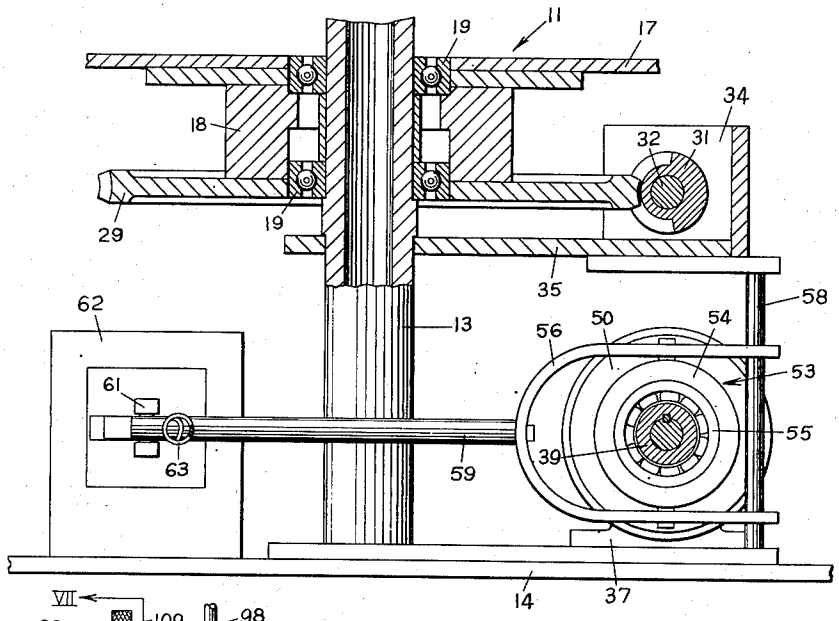


fig. 4

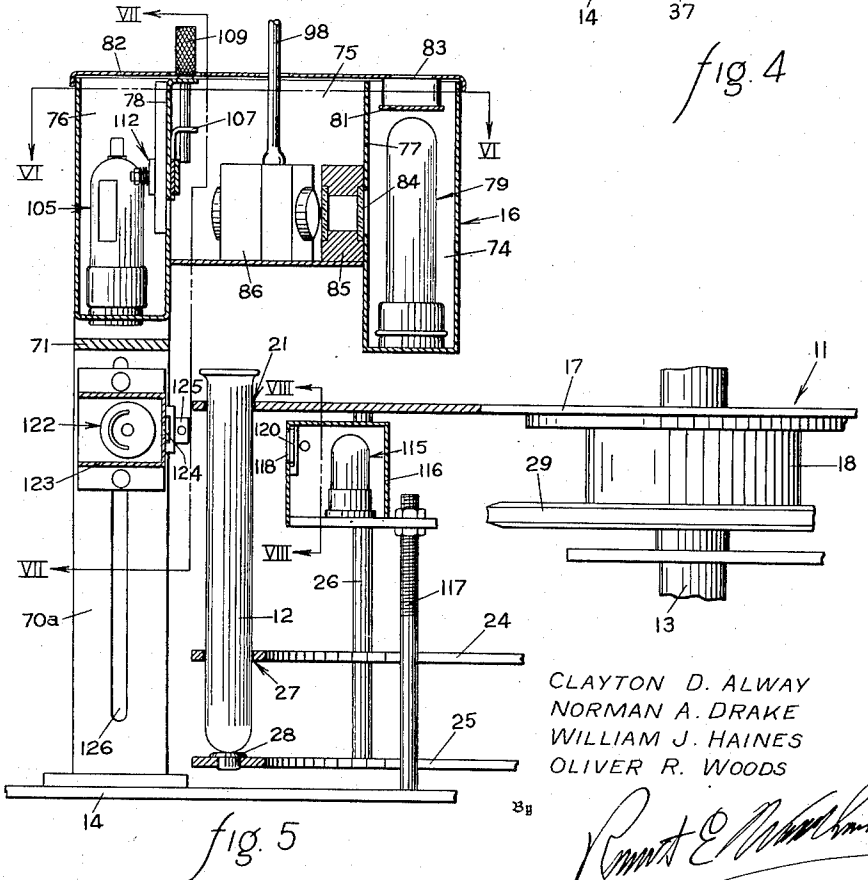


fig. 5

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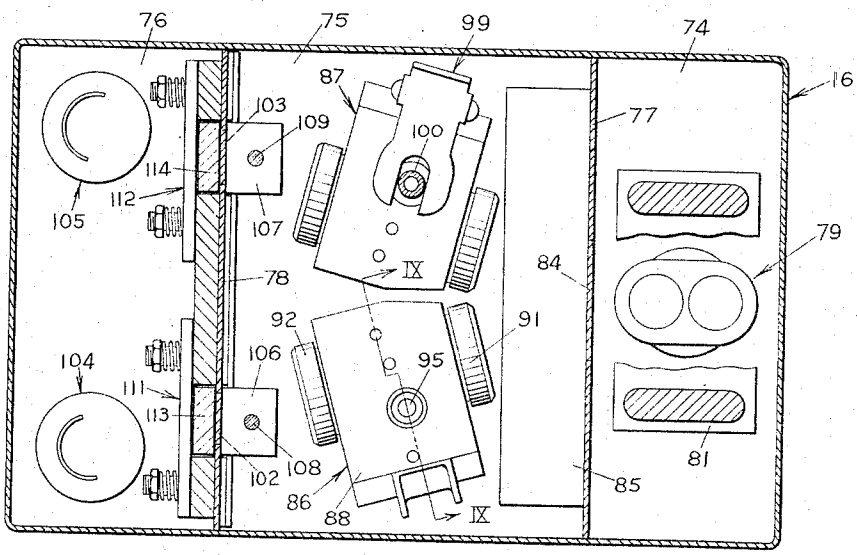


fig. 6

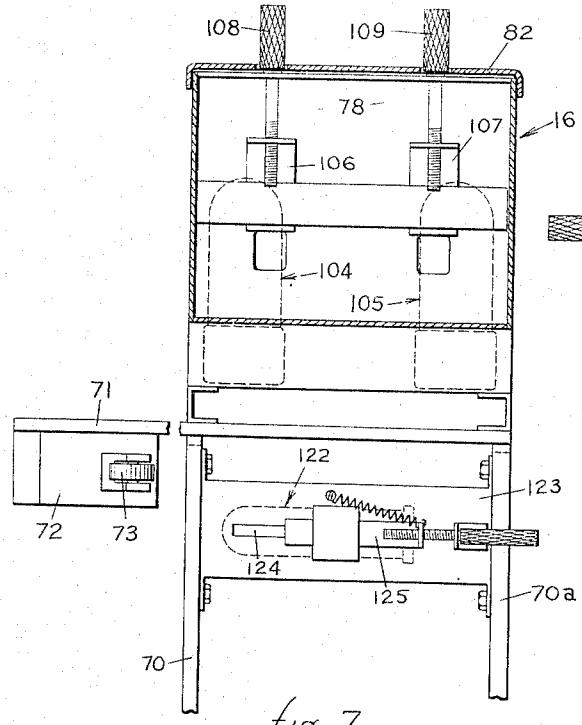


fig. 7

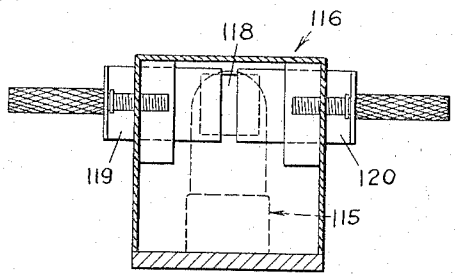


fig. 8

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METHOD AND MEANS FOR EFFECTING AUTOMATIC FRACTIONATION

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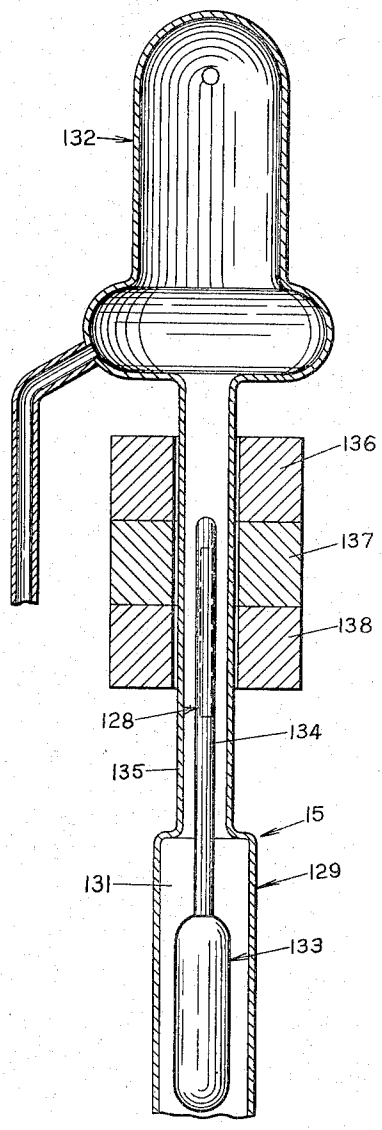


fig. 12

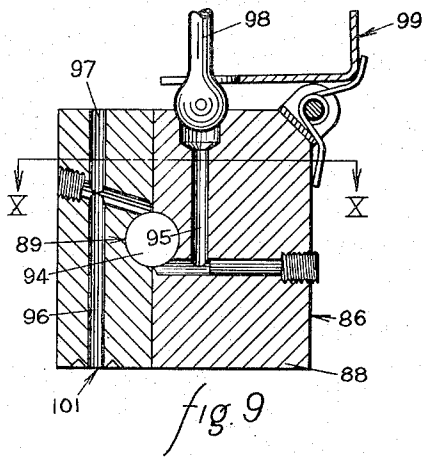


fig. 9

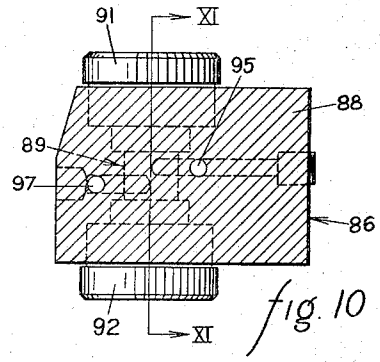


fig. 10

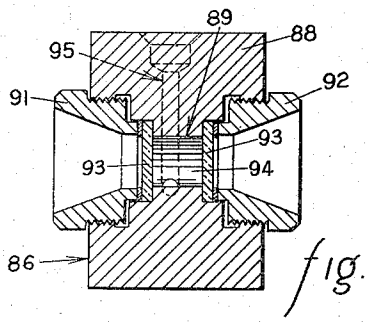


fig. 11

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METHOD AND MEANS FOR EFFECTING AUTOMATIC FRACTIONATION

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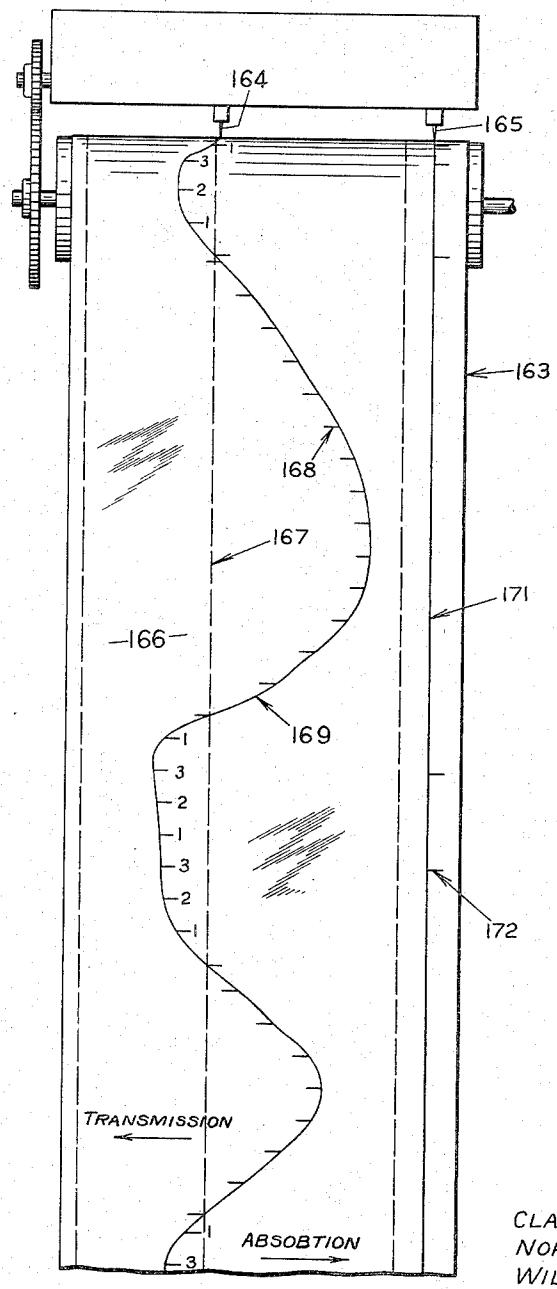


fig. 13

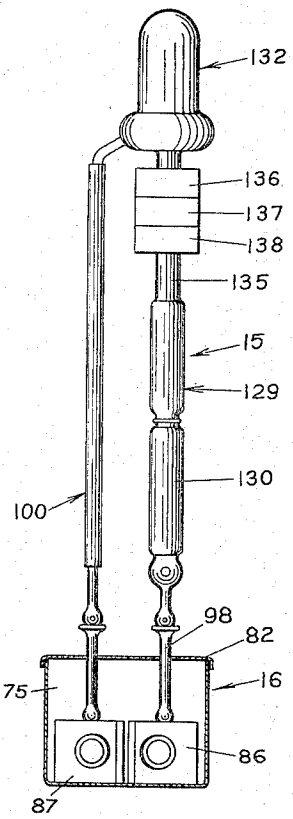


fig. 14

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10 Sheets-Sheet 7

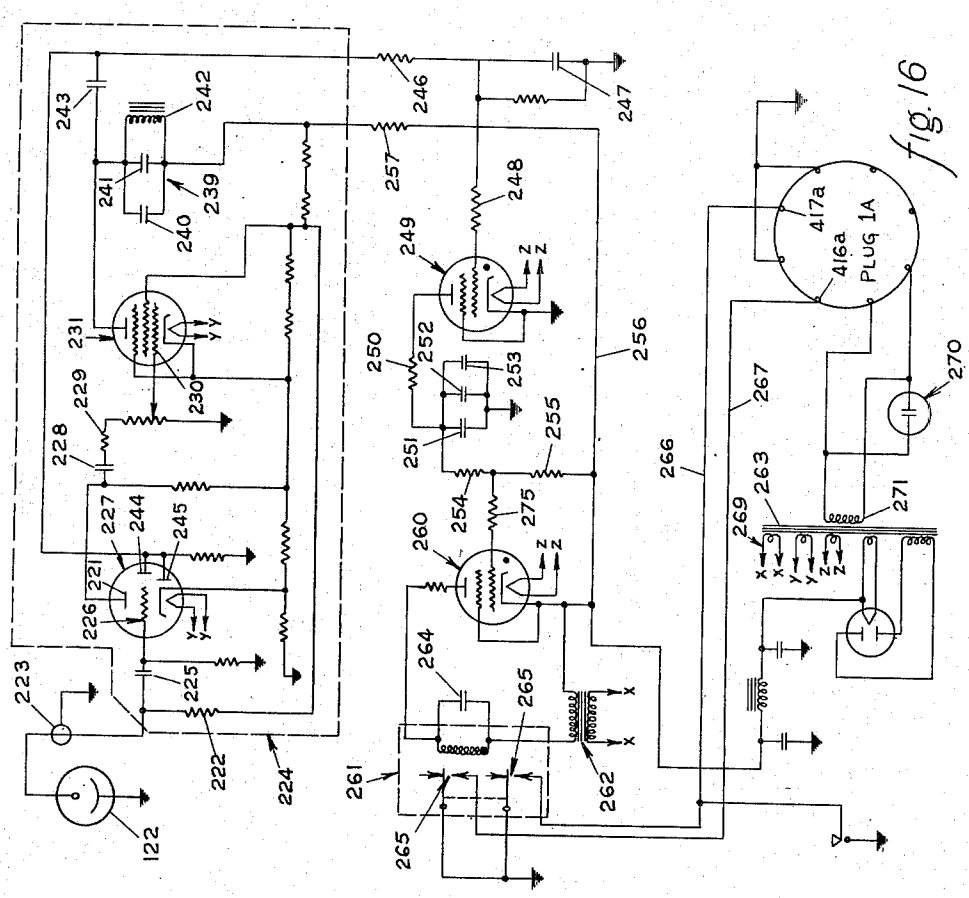


fig. 16

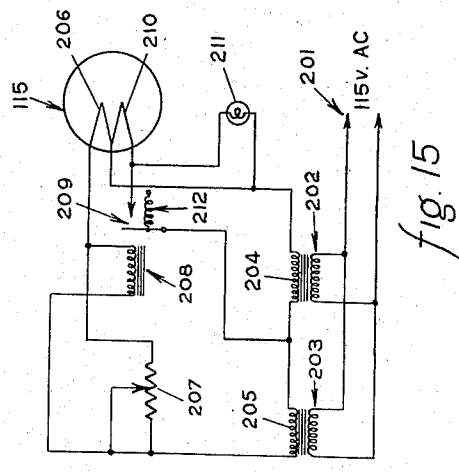


fig. 15

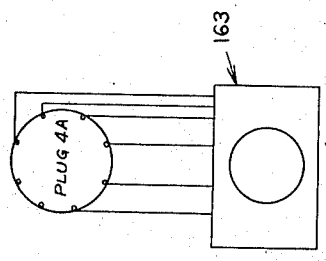


fig. 21

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METHOD AND MEANS FOR EFFECTING AUTOMATIC FRACTIONATION

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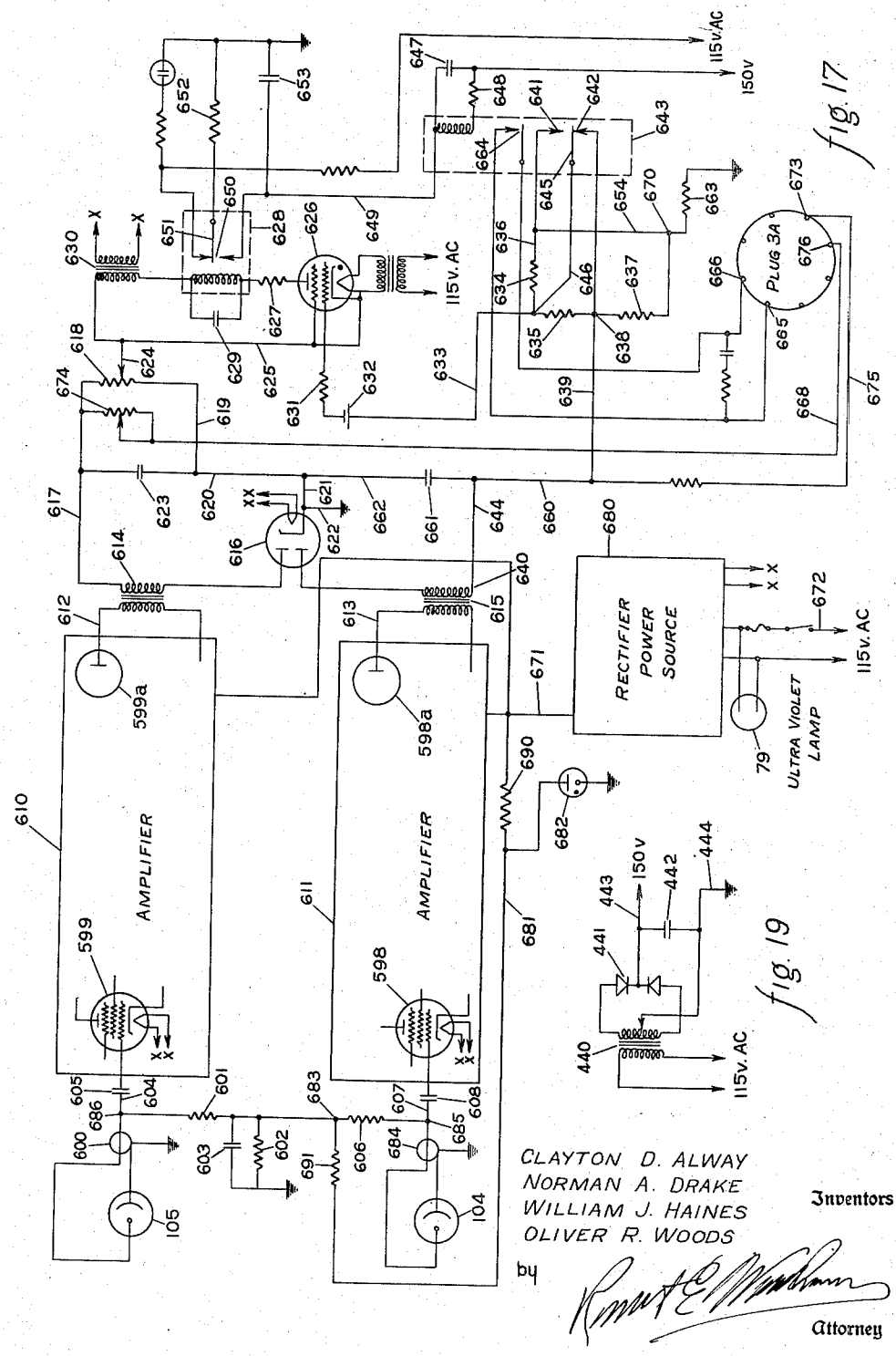


fig. 17

fig. 19

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METHOD AND MEANS FOR EFFECTING AUTOMATIC FRACTIONATION

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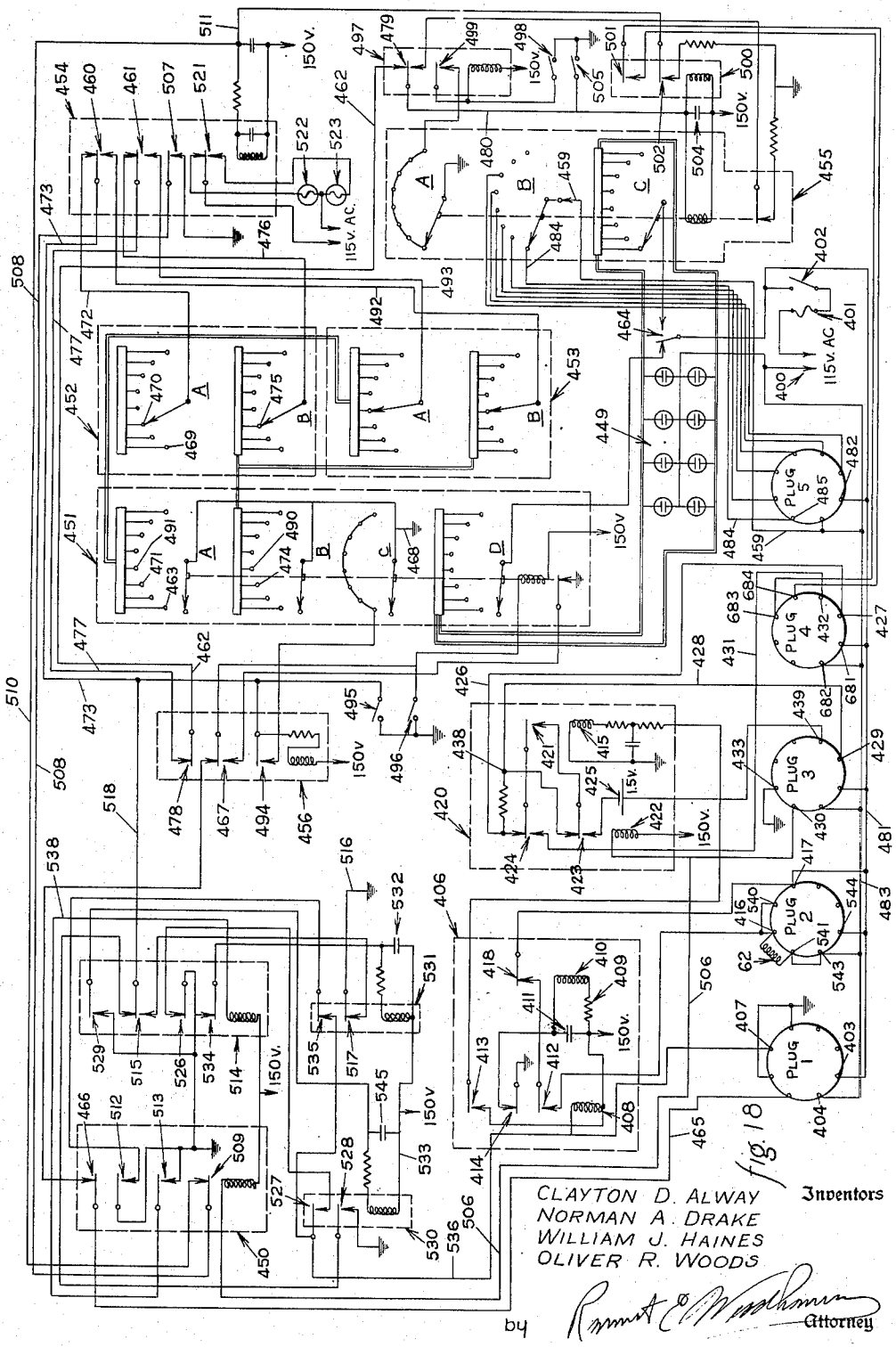


fig. 18

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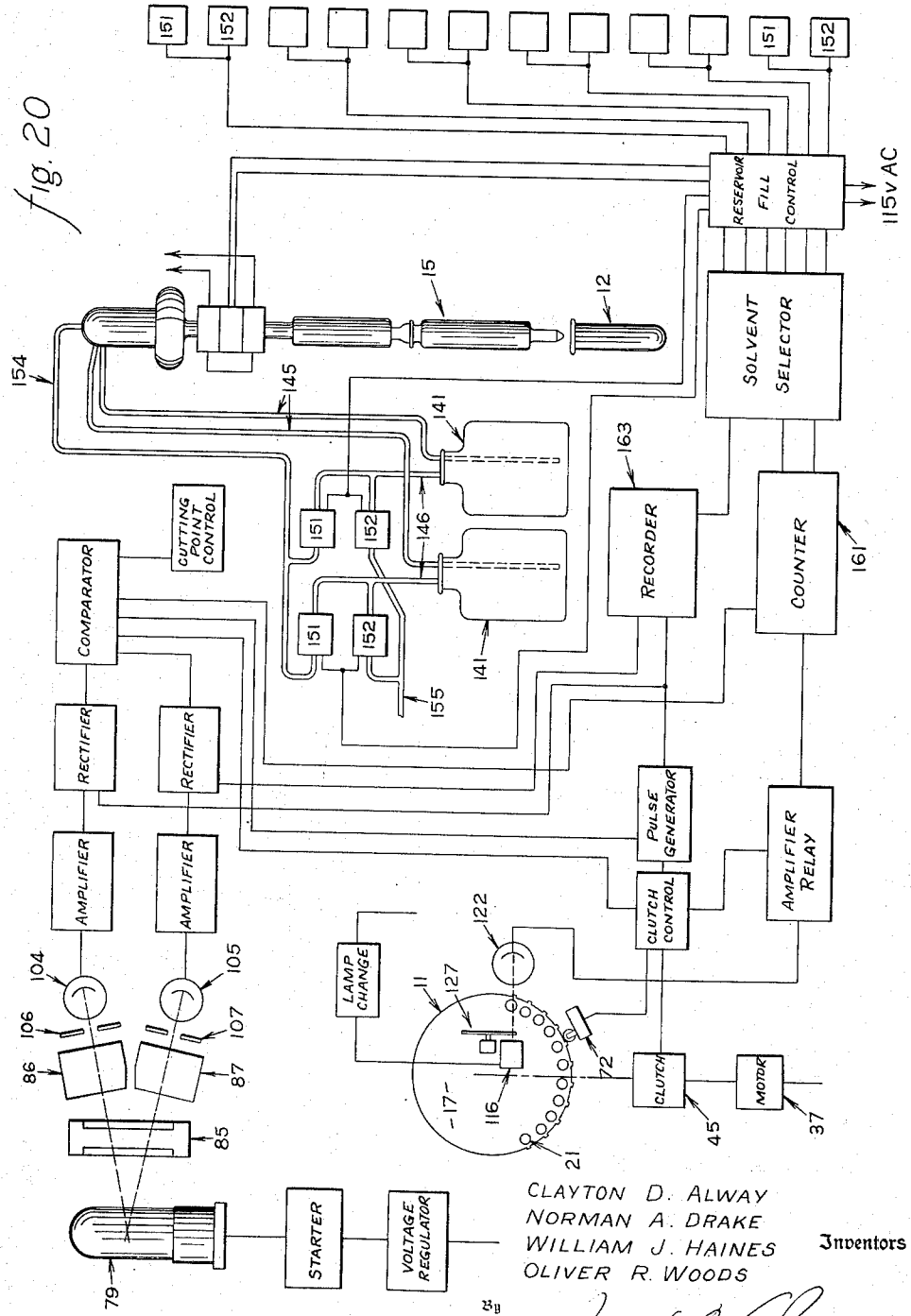
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Fig. 20



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Patented Oct. 26, 1954

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UNITED STATES PATENT OFFICE

2,692,820

METHOD AND MEANS FOR EFFECTING AUTOMATIC FRACTIONATION

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Application October 28, 1950, Serial No. 192,722

17 Claims. (Cl. 23—309)

1

This invention relates in general to analytical equipment and more specifically to an automatic instrument employing refraction and differential absorption of electromagnetic radiation for detecting, separating and collecting in fractions the effluent from a fractionating column.

The process of separating and collecting in fractions eluted compounds from a fractionating column has heretofore been conducted substantially manually. Thus, the continuous presence of a skilled operator for changing the receptacles in which the product is collected, for changing the solvent being introduced into the column, and for filling the proper number of receptacles with any given solvent and/or solute as products of the fractionation has been necessary where such processes have formerly been desired. However, due to the slow action of a fractionating column, an operation involving same often extends over a period of several days during much of which time the operator is frequently idle. Furthermore, unless shifts of operators are provided beyond the normal working hours, the process must be stopped overnight which often introduces further complications.

Accordingly, it becomes apparent that the provision of an entirely automatic instrument which can be pre-set by an operator to effect the above mentioned operation and then be disregarded, would not only relieve the operator for other work, but would also permit continuous operation of the process over extended periods of time. By the equipment hereinafter disclosed, collection of a series of fractions, which would require days by ordinary procedures, may now be accomplished in a few hours.

Accordingly, a primary object of this invention is the provision of an instrument for automatically separating and collecting the respective compounds eluted from a fractionating column.

A further object of this invention is the provision of an instrument, as aforesaid, having means for supporting a plurality of containers for collecting eluted compounds and further means for automatically positioning said containers during the collection of said eluted compounds.

A further object of this invention is the provision of an instrument, as aforesaid, having means for automatically controlling the amount of eluted compounds collected by said individual containers.

A further object of this invention is the provision of an instrument, as aforesaid, having

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means for introducing in a predetermined sequence a variety of solvents into a fractionating column.

A further object of this invention is the provision of an instrument, as aforesaid, having means for controlling the period during which any particular solvent is directed into said fractionating column and, accordingly, the number of containers which are filled with discharge from the column including a particular solvent.

A further object of this invention is the provision of an instrument, as aforesaid, having means insuring a continuous supply of a given solvent to the fractionating column.

A further object of this invention is the provision of an instrument, as aforesaid, which can be operated over a relatively long period of time without constant attention from an operator.

A further object of this invention is the provision of an instrument, as aforesaid, which greatly reduces and greatly simplifies the separation and collection in fractions of compounds eluted from a fractionating column, over methods and means presently available.

Other objects and purposes of this invention will become apparent to persons familiar with this type of operation upon referring to the accompanying drawings and upon reading the following specification.

For illustrations of a preferred embodiment of our invention, attention is directed to the accompanying drawings in which:

Figure 1 is a broken side elevation view of an automatic instrument for detecting, separating and collecting in fractions compounds eluted from a column, including schematically a portion of the electric circuitry involved.

Figure 2 is a sectional view substantially as taken along the line II—II of Figure 1.

Figure 3 is a sectional view substantially as taken along the line III—III of Figure 2.

Figure 4 is a sectional view substantially as taken along the line IV—IV of Figure 3.

Figure 5 is a sectional view substantially as taken along the line V—V of Figure 2.

Figure 6 is a sectional view substantially as taken along the line VI—VI of Figure 5.

Figure 7 is a sectional view substantially as taken along the line VII—VII of Figure 5.

Figure 8 is a sectional view substantially as taken along the line VIII—VIII of Figure 5.

Figure 9 is a sectional view substantially as taken along the line IX—IX of Figure 6, and for

convenience in showing is rotated approximately 90 degrees in a counterclockwise direction.

Figure 10 is a sectional view taken along the line X—X of Figure 9.

Figure 11 is a sectional view as taken along the line XI—XI of Figure 10.

Figure 12 is a sectional view taken substantially along the line XII—XII of Figure 1.

Figure 13 is a fragmentary view of a tape and means associated therewith for recording permanently and visually the operation of the instrument to which this invention relates.

Figure 14 is a sectional view substantially as taken along the line XIV—XIV of Figure 1.

Figure 15 illustrates diagrammatically a circuit energizing the fill control lamp.

Figure 16 illustrates diagrammatically the electric circuit responsive to the filling of an individual container for effecting moving of the next successive container into filling position.

Figure 17 shows diagrammatically a circuit adapted for use to distinguish the differential between the radiation passing through the sample being tested and through a standard.

Figure 18 represents the counter circuit together with the means directly connected thereto.

Figure 19 represents a suitable power supply circuit.

Figure 20 represents a schematic diagram of the entire mechanism showing primarily the relationship between the various portions of the electrical circuit together with their functional association with the mechanical parts.

Figure 21 illustrates the recorder connections.

GENERAL DESCRIPTION

In meeting those objects and purposes heretofore mentioned, as well as others incidental thereto and associated therewith, we have provided a device having a conveyor, such as a circular turntable, for supporting a plurality of open containers and for moving said containers sequentially into and out of position for filling thereof. Means are provided for supporting receptacles containing the several solvents to be used in the fractionating process. Further means are provided for supporting a fractionating column into which said solvents are directed. A differentiating head is disposed between the fractionating column and the container being filled at a given moment. The differentiating head is provided with means for determining when the concentration of eluted material being separated from the fractionating column by a particular solvent rises to, or drops below, a predetermined value. The differentiating head then initiates action to change the solvent and positions another container for collection of further solvents and eluted compounds dissolved therein. The differentiating head in the specific embodiment here chosen for illustrative purposes comprises a source of electromagnetic radiation of a wavelength absorbable in the compound desired and means responsive to the differential in absorption of such radiation passing (1) through one transparent cell containing the solvent and the said compound and (2) through another transparent cell containing only solvent. However, it will be appreciated as the detailed description progresses that the apparatus and method may also utilize other types of radiation energy as light of other wavelengths or an electron beam, and other characteristics than absorptiveness of the material being sought as its power of optical or magnetic rotation or index of refraction, and these items will be selected according to the characteristics of the material being separated.

An electrical system which effects and controls the operation of the several parts, comprises first, a counting device by which the solvent control valves are changed when a predetermined number of containers have been filled with the products of the fractionating column, and secondly, means responsive to control of the differentiating head to block operation of the counting device so long as said products maintain certain preselected absorption characteristics. A recorder is also provided independently of the valve changing means for indicating and recording the amount of said eluted material in the column effluent at any given time. The recording means is here shown as visual, but it could also be audible.

Means are also herein provided for urging the solvents from their respective receptacles into a collecting chamber attached to the inlet end of the fractionating column, and for maintaining a constant solvent level in said chamber.

For the purpose of convenience in the detailed description which follows, the terms "upper" and "lower," as used herein will be understood to refer to the instrument 10 when in its normal operating position, as appearing in Figure 1. The terms "inner" or "inwardly" and "outer" or "outwardly" shall have reference to the geometric center of the instrument 10 or parts thereof.

The method and device here disclosed may be used for either partition chromotography or the adsorption chromotography, or for other methods of solvent extraction wherein determination of the amount of eluted material present at a given time in the discharge from the column may be desired.

CONSTRUCTION

Turntable and mechanical portion of its actuating means

As shown in Figures 1, 2, 3, 4 and 5, the turntable 11, which supports and moves a plurality of containers 12 is rotatably supported upon a central pivot post 13, which is in turn mounted upon and secured to the base plate 14 whereby said turntable is free to rotate in a horizontal plane. The purpose of the turntable 11 and parts associated therewith is to move the containers 12 supported thereon successively into position for reception, one at a time, of liquid released from a fractionating column 15 and passing through a differentiating head 16.

Although a circular turntable 11 is specifically shown, for illustrative purposes only, as the means supporting the containers 12 to be filled with the solution flowing from the fractionating column, it will be readily realized that other conventional, conveyor means, such as a straight line conveyor, may be used alternatively within the scope of this invention.

The turntable 11 is comprised of a circular top plate or disk 17 (Figures 1 and 2) having an open center through which the pivot post 13 may extend. The disk 17 is secured, as by welding, to a support sleeve 18, which is rotatably supported upon the pivot post 13 by means of the bearings 19. The top disk 17, which is coaxial with the pivot post 13, is provided with a plurality of preferably equally spaced container openings 21 disposed along, and slightly inwardly of, the peripheral edge 22 of the disk 17, said container

openings 21 being equidistant from the axis thereof. The openings 21 are preferably circular and identical in diameter.

The peripheral edge 22 of the disk 17 is provided with a plurality of equally spaced cams or projections 23 lying within the plane of the disk and extending radially and equidistantly therefrom. The projections 23 are equal in number to the number of openings 21 in the disk 17 and are preferably, but not necessarily, aligned with said openings along respective radii of the disk.

A micro-switch 72, having a roller contact 73, is supported, as hereinafter described, adjacent to the turntable 11 so that said contact 73 is in continuous engagement with the peripheral edge 22 of the disk 17. The switch 72 is arranged so that it will be normally "closed" when the roller contact 73 is engaging the edge 22 between any pair of projections 23, and normally open when said contact is engaged by a projection 23.

The turntable 11 is also provided with intermediate and lower container support rings 24 and 25 respectively, which are spaced downwardly from, and suspended upon, the disk 17 by means of the connecting rods 26. The intermediate and lower rings preferably have outside circumferences substantially equal to the cirumference of the disk 17 and are coaxial with the pivot post 13. The intermediate ring 24 is provided with a plurality of container openings 27 therethrough, which openings are substantially equivalent in size, radial disposition, and vertical alignment with the container openings 21 in the plate 17 (Figure 5). Thus, containers 12, such as test tubes, inserted through the container openings 21 will also extend through the container openings 27.

The lower ring 25 is provided with sufficient horizontal surface and is so disposed to engage and support the lower ends of the containers 12 when they are positioned within the openings 21 and 27. Convenient means, such as the rubber bumpers 28 (Figure 5), may be inserted through appropriate openings of the lower ring 25 for the purpose of cushioning the support of the container 12.

A circular gear 29 (Figures 2, 4 and 5) is secured to the bottom of, and is coaxial with, the support sleeve 18 upon which the disk 17 is mounted. Thus, rotation of the gear 29 effects a rotation of the disk 17 and the rings 24 and 25 suspended therefrom. The gear 29 is engageable with and rotatable by the worm gear 31 secured upon a horizontal shaft 32 (Figures 2, 3 and 4). The shaft 32 is rotatably supported adjacent to one end thereof upon the vertical end plate 33, which is in turn supported upon the base plate 14 in any convenient, conventional manner. The other end of the shaft 32 is rotatably held in a bearing support 34 which is mounted upon a horizontal support plate 35 (Figures 1, 2, 3 and 4) mounted between and upon said end plate 33, another end plate 36 and the pivot post 13. An electric motor 37, or similar means, is secured upon the base plate 14 beneath the support plate 35 so that its shaft 38 is preferably parallel with and directly below the shaft 32, and extending toward the end plate 33. A stub shaft 39, which is coaxial with, extends toward, and is spaced slightly from, the motor shaft 38, is rotatably supported by means of the bearings 41 mounted within the end plate 33. Both the worm shaft 32 and the stub shaft 39 extend through and beyond the outer side of the end plate 33 for engagement by, and support of, a pair of pulleys 42 and 43, respectively, which are mutually engaged by a chain 44 or similar drive means (Figures 1 and 3).

A clutch 45 (Figures 1, 3 and 5) is mounted upon the motor shaft 38 and the stub shaft 39 between the motor 37 and the end plate 33 to effect intermittent, controlled rotation of the worm gear 31.

In this particular embodiment, the clutch 45 (Figures 3 and 4) is comprised of one circular clutch plate 46 mounted upon and rotatable with the motor shaft 38, and another circular clutch plate 47 is mounted upon the inner side of the end plate 33 around the stub shaft 39. The clutch plates 46 and 47 are provided with circular clutch bands 48 and 49, respectively, on their opposite faces. A circular clutch member 50, having a pair of parallel faces 51 and 52, is secured upon the stub shaft 39 between the clutch plates 46 and 47 for axial movement between the clutch plates 46 and 47. The faces 51 and 52 are engageable with the clutch bands 48 and 49, respectively. Thus, upon appropriate axial movement of the clutch member 50 toward the clutch plate 46, the clutch face 51 will engage the clutch band 48 in a conventional manner and rotation of the motor shaft 38 will thereby effect rotation of the stub shaft 39. Movement of the clutch element 50 toward the clutch plate 47, will cause engagement between the clutch face 52 and the clutch band 49, thereby causing the clutch member 50, if rotating, to stop quickly, also in a conventional manner.

The clutch member 50 (Figure 3) is provided with a relatively deep, annular groove 53 between its two parallel faces 51 and 52. A sleeve 54 encircles the clutch member 50 within the annular groove 53, thereof, and is rotatably mounted upon the clutch member 50 by means of a bearing 55. A yoke 56 (Figures 3 and 4) extends about the sleeve 54 and its arms are pivotally secured to the sleeve 54 by means of the pins 57. The arms of said yoke 56 are rotatably supported at their free ends upon a substantially vertical rod 58 extending therethrough and secured at its opposite ends to the base plate 14 and the support plate 35. Accordingly, movement of the yoke 56 in a horizontal plane will effect axial movement of the clutch member 50 back and forth between the clutch plates 46 and 47.

A horizontal bar 59 (Figures 1 and 4) is secured to the yoke 56 and extends therefrom for pivotal engagement at its opposite end by the actuating arm 61 of the solenoid 62 mounted on the base plate 14.

A resilient means, such as the spring 63, is connected at one end to the bar 59, adjacent to the actuating arm 61, and is anchored at its other end upon a post 64 mounted upon the base plate 14. The spring 63, being under tension tends to urge the bar 59 away from the solenoid 62 whereby the yoke 56, acting through the pins 57, urges the face 52 of the clutch member 50 against the clutch band 49 on the clutch plate 47, thereby preventing rotation of the clutch member 50. Actuation of the solenoid 62 urges the bar 59 toward the solenoid, thereby causing the face 51 of the clutch member 50 to engage the clutch band 48 on the clutch plate 46, whereupon the motor 37 may effect a rotation of the stub shaft 39.

During normal operation of the instrument

10, the motor 37 is in continuous operation and the clutch 45 provides for intermittent movement of the turntable 11 upon appropriate energization of the solenoid 62 by the micro-switch 72 or by other means hereinafter described.

DIFFERENTIATING HEAD

The differentiating head 16 (Figures 1, 2 and 5) is supported upon a horizontal cross bar 71 which is secured to the tops of a pair of parallel support bars 70 and 70a which are in turn mounted upon the base plate 14 adjacent to the turntable 11, so that said head 16 extends over the top disk 17 of the turntable 11. The horizontal cross bar 71 extends laterally beyond the support bar 70 and adjacent to the peripheral edge 22 of the top disk 17 for the support of the micro-switch 72 in the manner described hereinabove.

The differentiating head 16 is divided into inner, intermediate and outer compartments 74, 75 and 76, respectively, by reference to the pivot post 13 of the turntable 11, with appropriate inner and outer partitions 77 and 78 between the compartments 74 and 75 and the compartments 75 and 76, respectively.

A source of radiant energy, such as an ultra-violet tube 79 (Figures 5 and 6), is centrally disposed within the inner compartment 74 and is electrically connected in any conventional manner to a source of power. As mentioned above, the type of radiation may vary within the scope of this invention and may be any type or wavelength which will be absorbed, or otherwise physically affected, by the compounds being collected. While it is preferred that such absorption be in a substantially lineal relationship to the concentration of said compounds in the eluted solution, other relationships may be used where less accuracy is required or upon proper compensation of the electrical circuit responsive to such absorption. However, in this particular embodiment for extracting steroids containing a conjugated diene system, e. g., cortical steroids, ultra-violet light having a wavelength of 2,537 Angstrom units has been found advantageous. A baffle plate 81 is supported from the top 82 of the differentiating head 16 for preventing said ultra-violet radiation of the tube 79 from escaping through a vent opening 83 in the top 82. A light opening 84 is provided in the partition 77 adjacent to the tube 79 through which the radiation emanating therefrom may pass into the intermediate compartment 75.

A filter 85 (Figures 5 and 6) is disposed within the intermediate compartment 75 adjacent to the light opening 84 in the partition 77 for the purpose of controlling the wavelength of the radiation passing into the intermediate compartment 75. However, where the rotary power of the eluted material is being utilized, this filter may be replaced by any suitable means, as a Nicol prism, for polarizing the light from source 79 into a selected plane.

A pair of absorption cells 86 and 87, which are preferably mirror images of each other (Figures 5, 6, 9, 10, 11 and 14), are disposed within the intermediate compartment 75 adjacent to each other and so that radiation emanating from the tube 79 and passing through the light opening 84 will be intercepted by both of said cells. Inasmuch as the cells 86 and 87 are in this embodiment substantially identical, only the cell 86, through which the solvent and compounds dissolved therein pass, will be described with the understanding that such description applies in substance to the cell 87 through which the solvent only passes.

As shown in Figures 6, 9, 10 and 11, the absorption cell 86 is comprised of a block shaped body member 88, having a substantially cylindrical light passage 89 passing horizontally therethrough. The light passage 89 is provided with cylindrical portions of enlarged diameter at the opposite ends thereof which are internally threaded for reception of a pair of externally threaded light gathering elements 91 and 92. Filters 93 are disposed in the passage 89 (Figure 11) between the elements 91 and 92 and the body member 88 so that they provide an absorption chamber 94 within the body member 88.

A fluid inlet canal 95, which opens through the upper surface (Figure 9) of the body member 88, extends through said body member and communicates with the absorption chamber 94 near the bottom thereof. A fluid outlet canal 96, which opens through the bottom surface of the body member 88, communicates with the absorption chamber 94 near the top thereof. A vent canal 97 communicates between the top surface of the body member 88 and the outlet canal 96. The inlet canal 95, where it opens through the top of the body member 88, is provided with an enlarged portion properly beveled for reception of a transfer tube 98 (Figures 9 and 14) from the fractionating column 15. The transfer tube 98 may be held in snug contact with the walls of the opening of the inlet canal 95 by any convenient means, such as the clamping device 99 (Figures 6 and 9) secured to the body member 88. Accordingly, solvent and any materials dissolved therein flowing from the fractionating column 15 through the transfer tube 98 enter the bottom on the absorption chamber 94 through the inlet canal 95 and are carried off therefrom through the outlet canal 96. Therefore, in addition to the fact that the absorption cell 86 must be positioned to permit light from the tube 79 to pass through the light gathering elements 91 and 92 and the absorption chamber 94 thereof, said cells must also be disposed so that the lower end 101 of the outlet canal 96 is disposed directly over the center of the path of the containers 12 mounted on the turntable 11. The axes of the light passages 89 through the cells 86 and 87 are disposed at an angle which converges at a point substantially at the center of the tube 79, whereby radiation from the tube 79 will pass through the absorption chamber 94 of both cells.

The outer partition 78 is provided with a pair of spaced, light openings 102 and 103 (Figures 5, 6 and 7), which are intercepted, respectively, by said axes of the light passages through the absorption cells 86 and 87. Thus, radiation from the tube 79 passes through the light opening 84, the filter 85, the absorption chambers 94 of the absorption cells 86 and 87, and the light openings 102 and 103, respectively, in the outer partition 78. A pair of cells sensitive to the radiant energy beam from the source 79, as photo-electric tubes 104 and 105, are disposed within the outer compartment 76 of the differentiating head 16 so that said radiation passing through the light openings 102 and 103 will strike the filaments of the photo-electric tubes 104 and 105, respectively.

Where the rotatory power of the material is being utilized, suitable devices, such as a pair of Nicol prisms, may be inserted in the retaining devices 111 and 112 (Figure 6) in place of

the filters 113 and 114 for cooperation with the similar prism replacing the filter 85. Thus, the light falling on the photo-cell 104 will vary in intensity according to the magnitude of rotation imparted thereto. In this embodiment, that is, utilizing the particular comparator circuit here shown, said intensity should decrease as said rotation increases, but this is a matter of choice.

A pair of shutters 106 and 107 (Figures 5, 6 and 7) are mounted upon the outer partition 78 within the intermediate compartment 75 for controlling the aperture of the light openings 102 and 103, respectively. The shutters 106 and 107 are controlled by thumb screws 108 and 109 which extend above the top 82 of the differentiating head 16 for manual engagement. Resilient retaining devices 111 and 112 (Figures 5 and 6) are mounted on the outer partition 78 within the outer compartment 76 for supporting filters 113 and 114 across the light openings 102 and 103, respectively. Accordingly, radiation from the tube 79 may be controlled first by the filter 85, secondly by the filters 93 in the absorption cells 86 and 87, and finally by the filters 113 and 114, if necessary.

A by-pass tube 100 (Figures 6 and 14) through which pure solvent may be fed may be connected to the upper end of the inlet canal of the absorption cell 87 in the same manner in which the transfer tube 98 is mounted on the absorption cell 86.

FLUID LEVEL CONTROL

The fluid lever control in the containers 12 at which the turntable 11 is moved, and which, therefore, must be accurately maintained throughout a particular test, is accomplished in this embodiment by utilizing the diffraction characteristics of light, directed at the container being filled, when the fluid level rises into the path of a beam of such light, or other electromagnetic radiation diffractible by the solvents used and to which a photo-cell will be sensitive. In view of the foregoing, however, other types of radiant energy, as an electron beam, or other characteristics of electro-magnetic radiation, as rotation of a polarized beam thereof, may be used here also.

In this embodiment, a radiant energy source, such as the lamp 115, is disposed within a lamp housing 116 vertically, adjustably supported upon a post 117 mounted upon the base plate 14 beneath the top disk 17 of the turntable 11 and directly below the inner end of the differentiating head 16 (Figures 1, 5 and 8). A light opening 118 is provided in that side wall of the lamp housing 116 facing the support bars 70 upon which the differentiating head 16 is mounted. A pair of shutters 119 and 120 which may be similar to said shutters 106 and 107, are mounted on the lamp housing 116 adjacent to the light opening 118 for controlling the aperture thereof. The lamp 115 is disposed within the lamp housing 116 so that radiation therefrom can pass through the light opening 118, in a substantially horizontal direction.

Means electrically sensitive to the radiant energy from the source 115, as a photo-electric tube 122 (Figures 5 and 7) is horizontally disposed within a tube housing 123 mounted between the support bars 70 below the head 16 and having a horizontal slot 124 in the sidewall thereof facing the lamp housing 116. The slot 124 is normally spaced in substantially the same horizontal plane as the light opening 118 in the lamp housing 116. A shutter 125 is mounted externally of the housing 123 for controlling the aperture of the slot 124. Thus, by appropriate control of the shutters 119 and 120 of the lamp housing 116 and the shutter 125 on the housing 123, radiation emanating from the lamp 115 may be directed so that it will not enter the open portion of the slot 124 when passing through a single media. However, as is well known and understood, when the container 12 disposed between the lamp housing 116 and the tube housing 123 and, being filled with a liquid, becomes filled to the point where the radiation from the lamp 115 passes through the liquid, the radiation from the lamp 115 is diffracted by the liquid and enters the open portion of the slot 124, thereby striking the cathode of the photo-electric tube 122. The adjustment slots 126 in the support bars 70 provide for vertical adjustment of the tube housing 123. Thus, by appropriate vertical adjustment of the lamp housing 116 and the tube housing 123, the level to which a container 12 is filled before it is automatically replaced by another container may be accurately controlled.

A light chopper 127 (Figure 20) may be disposed in the path of the radiation emanating from the lamp 115 toward the photo tube 122 to control the frequency of impulses striking said tube 122. Thus, the effects of stray radiation from outside sources striking the tube 122 can be blocked by tuning the tube circuit to that frequency produced by the chopper 127.

FRACTIONATING COLUMN

The fractionating column 15 Figures 1, 12 and 14) is comprised of a fractionating chamber 130, downwardly from which extends the transfer tube 98, and upwardly from which extends the solvent flow control 129. A collecting or manifold chamber 132 is mounted above and connected to the solvent flow control 129. As shown in Figure 12, the solvent flow control 129 has a chamber 131 containing a float 133, having a hollow stem 134 secured thereto and extending upwardly therefrom and into the pipe 135 which connects the solvent control chamber 131 with the manifold chamber 132. A rod 128 of magnetic metal is disposed within the stem 134.

The connection pipe 135 is surrounded by three independent sets of field windings 136, 137 and 138 (Figures 1 and 12), which are held snugly adjacent to each other. The upper and lower field windings 136 and 138 are in series across a power supply, and the intermediate field winding 137 is electrically connected to a rectifier filter 139. The rod 128 in the stem 134 provides the core for the field windings 136, 137 and 138. When the float 133 is raised by the solvent in the solvent control chamber 131, the rod 128 extends between said field winding and a current flow is impressed upon the center windings 137 by the upper and lower windings 136 and 138, which in turn energizes the rectifier and filter unit 139 to initiate electrical operations hereinafter described in detail. Briefly, however, energization of the rectifier and filter unit 139 interrupts the forced flow of solvent from a solvent supply, such as in the receptacle 141 (Figure 1), into the manifold chamber 132 at the top of the fractionating column 15.

One or more receptacles 141 may be supported upon a platform 142 (Figure 1) which is held horizontally above the turntable 11 by means of the platform posts 143, which are secured at their lower ends to the base plate 14. This particular arrangement of supporting the re-

ceptacle 141 is not essential but has been found convenient.

Each receptacle 141 (Figure 1) is provided with a hollow stopper 144 through which an outlet pipe 145 extends downwardly adjacent to, but spaced slightly from, the bottom of the receptacle 141. The outlet pipe 145 communicates directly with the manifold chamber 132 (Figure 1) near the upper end thereof. An inlet pipe 146 communicates with the chamber in the hollow stopper 144, which chamber communicates in turn with the inside of the receptacle 141. The purpose of this arrangement is to force a gas through the inlet pipe 146 into the receptacle 141, whereby the solvent within said receptacle is urged up through the outlet pipe 145 and into the manifold chamber 132.

A by-pass pipe 147 (Figure 1) communicates between the upper end of the manifold chamber 132 and the upper end of the solvent control chamber 131 (Figure 12).

A gas under pressure, from means not shown, is fed into the by-pass pipe 147 through the supply pipe 148. A pressure regulating valve 149 is provided in the supply pipe 148.

Each receptacle 141 is provided with a pair of electrically actuated, regulating valves 151 and 152 (Figures 1 and 20) with a connecting pipe 153 therebetween. The inlet pipe 146, which communicates with the hollow chamber in the stopper 144, also communicates with the connecting pipe 153. A pressure pipe 154 communicates between the regulating valve 151 and the junction of the supply pipe 148 with the by-pass pipe 147. Thus, the gas flowing through the pipe 148 at a pressure regulated by the valve 149, flows into the solvent control chamber 131, the manifold chamber 132 and the regulating valve 151. An auxiliary supply pipe 155 connected to the auxiliary supply of gas under pressure, not shown, communicates with the regulator valve 152.

The range of the pressure regulating valve 149 is preferably variable from zero to ten p.s.i. The pressure of the gas supplied through the auxiliary supply pipe 155 is preferably approximately two p. s. i. in excess of the pressure of the gas in the supply pipe 154. The regulator valves 151 and 152 are arranged so that one will be closed when the other is open. Thus, when the valve 151 is closed, as shown in Figure 1, the pressure in the inlet pipe 146 will be about two p. s. i. in excess of the pressure in the outlet pipe 145, thereby causing the said solvent to flow under pressure through the outlet pipe 145 and into the manifold chamber 132. When the positions of the regulator valves 151 and 152 are electrically reversed, the auxiliary gas pressure is shut-off and the pressure in the pipes 145 and 146 will become equal, permitting gravity to draw the liquid in the outlet pipe 145 back into the receptacle 141. The electric circuitry involved in reversing the regulator valves 151 and 152 upon energization of the energizing filter 139, will be dealt with in detail hereinafter.

Briefly, each pair of regulator valves 151 and 152 (Figure 20) is provided with a solenoid 158 (Figure 1) which is energized upon closing of the circuit thereof by the rectifier solenoid 156. Energization of the valve solenoid 158 (Figure 1) effects an electrical closing of the valve 151 and opening of valve 152, which causes solvent to flow from the receptacle 141 into the manifold 132. When this circuit including solenoid 158 is opened by de-energization of the solenoid 156, the valve 152 is electrically closed and valve 151 is electrically opened. The solenoids 158*a* through 158*d* (Figure 1) are provided for use with additional pairs of regulating valves (Figure 20) which operate in identically the same manner with appropriate solvent receptacles. The plug 159 is connected to a suitable electrical device, discussed hereinafter, for closing the circuit containing that one of the solenoids 158 through 158*d* controlling the flow of the desired solvent according to a predetermined pattern.

RECORDER

Referring to Figure 13, it will be seen that the instrument 10 is provided with recorder 163 comprising a pair of pens 164 and 165 and a recording tape 166 upon which a visual record is made of the percentage of absorption or transmission of ultra-violet radiation from the tube 79 by the solvent and compounds dissolved therein passing through the absorption cell 86. The radiation from the tube 79 is of such wave-length that it will be absorbed by the compounds dissolved in the solvent. Thus, the amount of radiation passing through the cell 86 is visually compared to the amount of radiation from the tube 79 passing through the absorption cell 87, through which cell the plain solvent is passing.

A minimum condition of absorption represented by the dotted line 167, is established below which the amount of compound in the solvent being collected by the containers and indicated by said absorption becomes too small for useful purposes. Obviously, this minimum can vary as desired or required. The indices 168 along the curve 169 (Figure 13) made by the pen 164 represent points at which the filling of a new container 12 commences. It will be noted that such indices always point toward the line 167 and, therefore, extend leftwardly of the curve 169 when the curve is on the rightward side of the line 167, and extend rightwardly when the curve 169 is on the leftward side of the line 167. When the indices are pointing leftwardly, the containers are being filled with useful compounds. When the indices are pointing rightwardly, a predetermined number of containers are being filled with non-useful liquid. If the pen 164 does not move to the right side of the line 167 by the time this number of bottles is filled, the solvent is automatically changed. The instrument 10 can be set by electrical means, hereinafter described in detail, so that a specific, preselected number of containers 12 will be filled with any particular solvent after the amount of compounds dissolved in the solvent and being collected in the containers drops below the minimum line 167. In general, this preselected delay in changing solvents insures satisfactory dissolving of the materials in the fraction chamber by the particular solvent if any is capable of occurring, and it also provides ample time for secondary dissolving to take place, before a solvent is changed. After the solvent is changed, the device provides sufficient time for proper dissolving to occur between the new solvent and the compounds in the fraction chamber 130, whereby the pen 164 will move to the rightward side of the line 167. As shown in Figure 13, the instrument 10 in this embodiment is preselected to fill three containers after the curve 169 has dropped below the minimum line 167. Thus, as long as the curve 169 remains to the left of the line 167, the solvent being fed into the fractionating column 15 will be changed after three containers 12 are filled

with that particular solvent. As long as the curve 169 remains to the right of the line 167, no change will be made in the solvent.

The line 171 (Figure 13) made on the tape 166 by the pen 165 records by the indices 172 points at which the solvent being fed into the fractionating column 15 is changed. As shown in Figure 20, two or more solvents in separate receptacles 141 are normally used in carrying out an operation of the instrument 10.

MECHANICAL OPERATION

The turntable 11 is provided with a plurality of containers 12, and as many solvents as are required to carry out a particular operation are placed in receptacles 141, mounted upon the platform 142 and connected, as by the outlet pipes 145 (Figures 1 and 20), to the manifold chamber 132 at the top of the fractionating column 15. The inlet pipe 146 from each stopper 144 on the receptacle 141 is connected to a pair of regulating valves 151 and 152. The electrical circuitry hereinafter described, of the instrument 10, is turned on and the first solvent will commence collecting fractions dissolvable therein. A gas under pressure is fed into the receptacle 141 (Figure 1) through the supply pipe 155, the valve 152 and the inlet pipe 146, thereby urging the solvent in the receptacle to flow through the manifold 132. This solvent in the manifold chamber 132 passes down through the connection pipe 135, into the solvent control chamber 131 and then down through the fraction chamber 130, in which certain compounds are held. The auxiliary pressure will continue to force the solvent into the manifold chamber 132 until the float 133 has been raised to a level where the stem 134 thereof extends between the field windings 136, 137 and 138. The rod 128 (Figure 12), acting as a core, induces a current flow through the solenoid 158 which results in energization of the solenoid whereby the valve 152 is closed and the valve 151 is opened. Since the pressure of the gas in the pipes 145 and 146 is neutralized by this change of valves, the solvent will stop flowing into the manifold 132. However, as soon as the float 133 drops down, the solenoid 158 will be deenergized and the valves 151 and 152 will be reversed, thereby permitting the gas passing through the auxiliary supply pipe 155, which has a higher pressure than the gas passing through the valve 149, to urge the solvent out of the receptacle 141.

The solvent in the manifold chamber 132, the connection pipe 135 and the solvent control chamber 131 (Figures 1, 5, 12 and 14), will slowly pass through the fractionating chamber 130 while taking into solution those compounds dissolvable therein. The solvent and solute will then pass through the transfer tube 98 into the absorption cell 86 and thence into a container held in the turntable 11 directly under the outlet 101 in the cell 86. As the solvent and solute pass through the absorption cell 86, the solute therein will absorb portions of the radiation from the tube 79. At the same time, solvent passes from the manifold chamber 132 through the bypass tube 100 directly into the absorption cell 87 through which the radiation from the tube 79 also passes. Accordingly, the radiation striking the photo-electric tube 105 will be a function solely of the absorption of the solvent, whereas the radiation from the tube 79 passing through the absorption cell 86 and striking the photo-electric tube 104 will be a function of both the absorption of the solvent and of the solute. Hence, the differential will be a function of only the solute. As long as the absorption by such solute remains above the minimum graphically shown by the dotted line 167 in Figure 13, the solvent being fed from a receptacle 141 to the manifold chamber 132 will remain the same. As each container 12 is filled to a predetermined level with such solute and solvent, radiation from the lamp 115 will be intercepted by the liquid in the container 12 and diffracted thereby through the slot 124 onto the filament of the photo-tube 122. By means of switch circuitry, described hereinafter, the tube 122 will then initiate energization of the solenoid 62 (Figures 1 and 4) whereby the clutch 45 will engage the motor 37, thereby causing the turntable 11 to rotate until the contact 73 of the switch 72 engages a projection 23 on the edge 22 of the top disk 17, whereupon the turntable is stopped abruptly by the braking function of the clutch 45.

The energization of the solenoid 62 by the tube 122 is of only sufficient duration, to effect such rotation of the turntable that the contact 73, will become disengaged from a projection 23, where it will be whenever the turntable 11 is not rotating. Immediately thereafter, the switch 72 takes over and continues the energization of the solenoid 62 until the contact 73 again engages a projection 23. Upon deenergization of the solenoid 62, the spring 63 causes the clutch member 50 to engage the clutch plate 47, whereby the rotation of the turntable 11 is immediately arrested.

Such rotation of the turntable will place the next adjacent container into position for filling. This sequence of operations continues as long as the absorption of radiation from the tube 79 by the fluid passing through the absorption cell 86 remains above a predetermined or preselected minimum.

As soon as the absorption drops below said predetermined minimum, the counter 161 (Figure 20) goes into operation to automatically count a preselected number of containers to be filled by the solvent and solute, even though the absorption is below said minimum, after which said counter then initiates a change in the solvent being fed into the manifold chamber 132. The counter 161 is automatically reset and commences counting the preselected number of containers as they are filled with the new solvent and such solute as may be removed by the new solvent from the fraction chamber 130. If the amount of solute, or compounds dissolved by the next solvent do not absorb radiation from the lamp 79 above said minimum before the preselected number of containers is filled with this new solvent, as indicated in the central portion of Figure 13, the counter 161 will initiate a change to a third solvent. After such changing of solvents, the counter automatically resets itself and commences to count containers containing the third solvent. However, if this particular solvent dissolves sufficient compounds from the fractionating chamber 130 to absorb radiation from the tube 79 above said minimum, the counter will then permit filling of containers 12 with this solvent and solute indefinitely until the absorption by the solute dissolved in the solvent again drops below the preselected minimum, as determined by the relative absorption of the two cells 86 and 87.

ELECTRICAL SYSTEM

Although a large portion of the circuits herein utilized are conventional, sufficient of them have been particularly adapted to the mechanical parts herein disclosed and for the specific purposes herein contemplated, that it is deemed desirable to describe said circuits in detail in the interest of presenting a complete disclosure. It should be understood, however, that these items of circuitry are herein described for illustrative purposes only and they may in many instances be substituted or completely replaced by other conventional circuits, providing only that the particular functions and purposes herein described are preserved.

FILL CONTROL LAMP CIRCUIT

The fill control lamp 115 may be of any conventional type. However, for purposes of safety it is preferred that said lamp have a pair of filaments, one of which is normally energized and the second of which becomes energized only upon the failure of the first. Figure 15 illustrates a suitable circuit.

In this circuit (Figure 15) the source 201 energizes the primary transformer windings 202 and 203 which in turn energize the secondary transformer windings 204 and 205, respectively. The secondary windings are connected in series with the main filament 206 of the fill control lamp 115 and with the variable resistor 207. Thus, in normal operation the filament 206 will be energized by current induced in said secondaries and passing through the filament and through the variable resistor 207. During such normal operation the winding of the relay 208, which is connected in parallel with the variable resistance 207, is likewise energized and holds the relay contacts 209 open against the action of a spring 212 normally tending to close said contacts.

The auxiliary filament 210 of the lamp 115 is connected in series with the secondary winding 204 and the contacts 209. The pilot lamp 211 is connected at any convenient point in this circuit. Here it is in parallel with the auxiliary filament 210 and, therefore, also in series with the contacts 209. Thus, when the main filament 206 burns out, the circuit including the relay winding 208 is broken and the contacts 209 close. Accordingly, the secondary winding 204 energizes the auxiliary filament 210 and simultaneously energizes the pilot lamp 211, which warns the operator that the main filament 206 has burned out, that the machine is operating upon the auxiliary filament 210 and that, therefore, the fill control lamp 115 should be replaced. By placing the pilot lamp 211 in parallel with the auxiliary filament 210, the said pilot lamp will be illuminated upon opening of the relay contacts 209 regardless of whether or not the filament 210 is illuminated. Thus, a warning is provided even though the auxiliary filament 210 has also become damaged and fails to operate.

FILL CONTROL CIRCUIT

The fill control circuit (Figure 16) operates upon the energization of the photo-tube 122 by the fill control lamp 115 and effects initial movement of the turntable 11 each time a given container is filled to a predetermined level. A portion of the circuit may be arranged to respond only to pulses from the photo-tube 122 having the frequency provided by the light chopper 127 (Figure 20) in order to insure that the turntable 11 will move only in response to light emanating from the lamp 115 and not to stray light from the outside sources. Obviously, this portion of the circuit may be removed where no light chopper is used, but it is here shown in the interest of completeness. Further, this circuit is provided with time delay means in order that the movement of the turntable will be initiated only when the container is actually filled to the proper level and will not be initiated by splashing or by drops of solution passing through the light beam.

Referring now to Figure 16, the photo-tube 122 is connected through a shielded cable 223 to the amplifier circuit indicated generally at 224, which circuit may be conventional. In this particular instance, however, the pulses from the photo-tube 122 flow through the load resistor 222 and the resultant voltage differential is applied through the capacitor 225 of the grid 226 of the amplifier tube 227, which amplifier tube is conveniently of the 6AT6-type. The amplifier tube 227 is arranged to operate on the lower portion of its characteristic curve so that its output is sharply limited. That is, the A. C.-voltage at the anode 224 thereof cannot rise above approximately one volt regardless of the voltage on the grid 226. The output at the anode 224 of amplifier 227 is coupled through the capacitor 228 and the resistor 229 to the grid 230 of the amplifier tube 231, which amplifier is conveniently of the 6AU6-type. Other suitable conductors connect the resistor 222 in a conventional manner through other resistors to the cathodes of the respective amplifier tubes 227 and 231, and to the shield grids of the amplifier 231.

The output of the amplifier tube 231 is conducted to a parallel resonant circuit 239 composed of the capacitors 240 and 241 and the reluctance 242, which elements are arranged to tune said resonant circuit 239 to the frequency of the light chopper 127 which, in this instance, is 540 cycles per second. Thus, unless the voltage applied to the grid 226 of the amplifier 227 has a frequency of 540 cycles per second, or some simple fraction thereof, very little voltage is developed across the parallel resonant circuit 239.

The output of the amplifier tube 231 is also coupled through the capacitor 243 to a suitable rectifier which in this instance is provided by the diode plates 244 and 245 in the tube 227. The rectified voltage is then filtered by the resistor 246 and the capacitor 247 and fed through resistor 248 to the grid of the thyratron 249.

The anode of the thyratron 249 is connected through the resistance 250 to one side of each of the three capacitors 251, 252, and 253, the other side of each of said three capacitors being connected to ground. The side of said capacitor to which the resistance 250 is connected is also connected in series with resistances 254 and 255, which are in turn connected to the conductor 256. Said conductor 256 is connected through the resistance 257 to that side of the parallel resonant circuit 239 opposite from that side to which the anode of the amplifier 231 is connected.

The thyratron 260 has its grid connected intermediate the resistances 254 and 255, and its cathode is connected to the conductor 256. A relay 261 has one end of its winding connected to the anode of the thyratron 260 and the other end of its winding connected through the secondary winding of a transformer 262 to the cathode of the thyratron 260. The primary winding of said transformer 262 is energized from any convenient

source, such as the secondary winding 269 of the transformer 263. Said transformer 263 also energizes the heaters of the two thyratrons and of the two amplifier tubes in a conventional manner. The capacitor 264 bridges the winding of the relay 261. The contacts 265 are controlled by the winding of the relay 261 and, when closed, complete a circuit through the conductors 266 and 267 which are connected to the poles 416a and 417a of the connecting plug 1A. A power plug 270 is connected to the primary winding 271 of the transformer 263 and provides power for the operation of this portion of the circuit. The micro-switch 72 is connected across the poles 540 and 417 of plug 2 in parallel with the turntable starting circuit 406 (Figure 18). The solenoid 62 is connected to the poles 416 and 541 of plug 2.

If an empty container 12 is in the fill position, so that the photocell 122 is not energized, no voltage is applied to the grid of the thyratron 249. Thus, the thyratron 249 will conduct at a sufficiently low anode potential as to effect a substantially continuous conduction and the capacitors 251, 252 and 253 are thus kept continually discharged therethrough. Under these circumstances current flows through the resistance 255 from the source provided by the transformer 263. In fill position, one of the cams 23 will hold the micro-switch 72 open so that the solenoid 62 is not energized until the turntable starting circuit 406 again provides a connection between terminals 416 and 417 of plug 2. The voltage drop across said resistance 255 is applied through the resistance 275 to the grid of the thyratron 260. This negative bias prevents the thyratron 260 from conducting in response to the potential supplied by the transformer 262 and thus the winding of relay 261 is de-energized and the contacts 265 remain open. This, by reason of subsequent circuitry, results in the turntable 11 remaining motionless.

However, when the container in fill position is filled, the photo-tube becomes energized, and a negative potential appears on the grid of the thyratron 249 and prevents it from conducting. This first allows the capacitors 251, 252 and 253 to charge in reponse to the potential provided by the source 263. However, as these capacitors become charged, the voltage drop across the resistor 255 becomes so small that the negative bias on the grid of the thyratron 260 is diminished sufficiently to permit said thyratron 260 to conduct. The capacitors 251, 252 and 253 provide sufficient delay between energization of the photo-tube 122 and conduction of the thyratron 260 that the turntable starting circuit 406 will respond only to a filled container and not to splashing or to drops of solution falling from the fractionating column 15. Conduction by thyratron 260 energizes the relay 261 thereby closing the contacts 265 thereof. The capacitor 264 is provided to prevent the relay 261 from chattering.

Closure of the contacts 265 operates through subsequent circuitry to effect movement of the turntable 11 in a manner described in detail elsewhere herein.

COMPARATOR CIRCUIT

The comparator circuit (Figure 17), which is essentially responsive to the differential in output between the two photo-tubes 104 and 105, opens or closes a relay whereby a connection is made which actuates a portion of the hereinafter described counting and control circuit. Said differential in photo-tube output varies according to the concentration of a selected solute in a given solvent flowing at a given time from the fractionating column 15, as described hereinabove. The comparator circuit also provides a small output potential which is connected to the recorder 163 (Figure 20) for recording the operation of the comparator circuit.

The comparator circuit includes a pair of amplifier circuits 610 and 611, one connected to each of the photo-tubes 104 and 105, which photo-tubes may be of any conventional type capable of producing an electrical response corresponding to change in light intensity falling thereon. Each of said photo-tubes is connected through its respective amplifier circuit to said comparator circuit, which latter opens and closes a control relay.

Now considering the comparator circuit (Figure 17) in more detail, the power source 630 is connected through the conductors 671, the resistor 690, the conductor 681 and the resistor 691 to the junction 683. The voltage regulator tube 682 and said resistors 690 and 691 assure a constant voltage being maintained at said junction point. From said junction point 683, one portion of the current travels through the resistor 602 to ground. The capacitor 603 bridges said resistor and functions to isolate two photo-tube circuits. Another portion of said current travels through resistor 601 and the shielded cable 600 to and through the photo-electric tube 105 to ground. The final portion travels through the resistor 605 and the shielded cable 684 to and through the photo-electric tube 104 to ground. Variations in light falling upon the photo-electric tubes will vary their respective conductivity characteristics and hence vary the potentials at the junction points 685 and 686. Conductors 607 and 604 connect said junction points 685 and 686, respectively, through the capacitors 608 and 606 to the control grids of the amplifier tubes 598 and 599, also respectively. Said amplifier tubes are each the first of the series of such tubes in the amplifier circuits 611 and 610, respectively, which circuits are conventional and are hence indicated only schematically.

The anode output from the tubes 599a and 598a in each of said amplifier circuits 610 and 611, respectively, is conducted by the conductors 612 and 613, respectively, to the primary windings of a pair of transformers 614 and 615. One end of each of the secondary windings of said transformer 614 is connected through the conductor 617 and a high resistance 618, of the order of 100 thousand ohms, to ground through the conductors 619, 620, 621 and 622. The cathode of the rectifier 616 is also connected to ground, conveniently through the conductor 622. A capacitor 623 may also advantageously be provided to smooth fluctuations in the output of the rectifier 616. The tap 624 is adjustable with respect to the resistor 618 and connects same through the conductor 625 to the cathode of the thyratron 626. The anode of the thyratron 626 is connected through the protective resistance 627 to one end of the winding of the relay 628, to which the capacitor 629 is connected in parallel, and the other end of said winding is connected to one end of the secondary winding of the transformer 630. The primary winding of the transformer 630 is connected to a 6.3 volt power source described in more detail elsewhere herein. The other end of the secondary winding of the transformer 630 is connected to the cathode of said thyratron 626.

The grid of the thyratron 626 is connected

through a resistor 631 to one side of the small, biasing battery 632 which battery provides on the order of 1.5 volts. The other side of said battery 632 is connected through the conductor 633 to a point between a pair of relatively large resistors 634 and 635, which in a preferred embodiment of this invention are of the order of 56 thousand ohms. The other end of the resistor 634 is connected through the conductor 636 and 654, and through a smaller, such as a 10 thousand ohm, resistor 637 to the junction point 638. The other end of the resistor 635 is also connected to the junction point 638, which junction point is then connected through the conductors 639, 650 and 644 to the end 640 of the secondary winding of the transformer 615. The contacts 641 and 642 of the relay 643 are connected, respectively, to the conductor 636 and to the junction point 638. The armature 645 of the relay 643 is connected through conductor 646 to the conductor 633.

Referring again to the transformer 615, the end 640 of its secondary winding is connected through the conductor 644 and 660 and the capacitor 661 to ground through the conductors 662, 621 and 622. The conductor 654 is also connected to ground through a high resistance 663, such as 100 thousand ohms.

The winding of the relay 643 has a capacitor 647 connected in parallel therewith and a resistor 648 in series with said winding and capacitor 647. The conductor between said resistor 648 and said capacitor 647 is connected to a 150 volts D. C. source, and the conductor between said winding and the capacitor 647 is connected by the conductor 649 to the contact 650 of the relay 628. The armature 651 of the relay 628 is connected through the resistance 652 to ground, thereby to complete the circuit for the winding of the relay 643. The capacitor 653 and the resistor 652 are provided to prevent arcing of the contacts 651 and 650.

The contact 664 of the relay 643 is connected on its respective two sides to the contacts 665 and 666, which co-act with the contacts 430 and 433 appearing on plug 3 in Figure 18. The contact 664 opens and closes the circuit between the connections 430 and 430A which function to operate the circuitry shown in Figure 18 and described in more detail hereinafter.

With the photo-tube 104 exposed to radiation from the lamp 79 passing through the solvent only, the output of the transformer 615 is comparatively steady. This output passes through the thyratron 626, when said thyratron is conducting, and energizes the winding of the relay 628 by which to hold the contact 650 closed. This energizes the winding of the relay 643 and holds the contact 664 closed, as well as holding the armature 645 against the contacts 641. Since only solvent passes through the cell 87, no radiation passing therethrough from lamp 79 will be absorbed thereby before falling upon photo-tube 105. Thus, the output of transformer 614 will be comparatively constant.

Assuming now that the solution discharged from the fractionating column 15 has insufficient solute dissolved therein to absorb radiation from the lamps 79, said radiation will fall upon the photo-tube 104 and the output voltage of the transformer 615 will equal the output voltage of the transformer 614. The adjustability of the point of contact between the conductor 624 and the resistor 618 provides the means for calibrating the system correctly to this balanced condition. With the relay 628 actuated and the contact 650 closed, the relay 643 is actuated and the armature 645 is connected to the contacts 641. Since current from the secondary winding of the transformer 615 passes through the conductors 644, 660 and 639, the resistance 637 and resistance 663 to ground, when the relay 643 is positioned as shown in Figure 17, a potential is taken from the junction point 638 through the contacts 642, the conductor 646 and the conductor 633 to the control grid of the thyratron 626. In the energized position of the relay 643 a potential is taken from the junction point 670 in the conductor 654 and carried through the contact 641 and the conductors 646 and 633 to the grid of said thyraton 626.

Thus, when there is no absorption of radiation from lamp 79 by the solution discharged from the fractionating column there will be a potential applied to the conductor 644 of the same value as that supplied to the conductor 617. This is conducted, as above described, to the grid of the thyratron 626 and, together with the biasing potential of the battery 632, acts to block the thyratron. So long as the thyratron is blocked, the relay 628 remains open as shown and the relay 643, as appearing in Figure 17, is not energized. Thus, the connections between the terminals 665 and 666 is open at the contacts 664 of relay 643.

However, as absorption of radiation from lamp 79 increases, the output of the photo-tube 104 decreases, and the potential on the control grid of the thyratron 626 will also decrease. When this grid potential decreases sufficiently to permit the thyratron to fire, the relay 628 becomes energized and through the closing of the contact 650 the relay 643 is also energized. This closes the contact 664 and provides a connection between the terminals 665 and 666. Simultaneously this connects the armature 645 to the contact 641 and thereby connects the grid of said thyratron 626 to the conductor 644 through the resistor 637, instead of directly as before. Thus, since the potential in this instance applied to the grid of said thyratron 626 is taken after the occurrence of the drop through the resistance 637, this grid potential of said thyratron immediately becomes still more negative. Accordingly, in order to cause the thyratron again to conduct, the potential in the conductor 644 must become somewhat more positive than would have been necessary had a direct connection between said grid and said junction point 638 been maintained. The purpose of this is to insure that the concentration of solute in the product of the fractionating column has actually changed a material amount before the system is caused to respond thereto, and to prevent response of the system to the minor changes in said concentration which are always present and which, if not damped out, cause a chattering of the entire equipment whenever they occur near the critical point. It will, of course, be understood that the balancing as such, of the outputs of the two photo-tubes 104 and 105 occurs merely by balancing of the grid and cathode of the thyratron 626 and effecting the opening and closing of the contact 664 according to whether or not said thyratron conducts.

The power supply for the foregoing equipment is of any conventional sort by which a rectified and reasonably smooth direct current is supplied by the rectifier 680 to the conductor 671 from the alternating source 672 and by which a small volt-

age, such as 6.3 volts, may be supplied to such points (*x*—*x*) as the heaters of the various tubes and the primary winding of the transformer **630**.

The conductor **668** is connected from the terminal **676** of plug 3A to the conductor **617** through the variable resistance **674**, and the conductor **675** is connected from the terminal **673** to the conductor **644**. Thus, the comparative voltages, consituting the final output from the photo-cells **105** and **104**, are supplied to the recorder **163** and a chart **166** (Figure 13) may automatically be made at any given time showing the percentage of absorption of light in the beam of light falling on the photo-tube **104** with respect to that falling on the photo-tube **105**. Inasmuch as the absorption of light from lamp **79** by the solute from the column **15** is a substantially lineal function of the concentration of said solute in the solution, this chart will provide a satisfactory record of the concentration of a given solute at any given time.

TIMING AND ASSOCIATED OPERATING MECHANISM

The timing circuit (Figure 18) consists of a series of step relays which operates each time the turntable **11** is rotated to move a filled container **12** from beneath the fractionating column and replace it with an empty container. Basically, the timing mechanism operates in such a manner that, when the turntable has moved a predetermined number of times, it will actuate the valve solenoids so that the solvent which has been supplied to the fractionating column is shut off and a new solvent is introduced. However, there is also provided in the timing circuit means by which the counting function is stopped for so long as the product of the fractionating column, as analyzed by the comparator circuit, has certain predetermined minimum characteristics. Therefore, so long as a selected compound is brought down from the fractionating column in a predetermined amount, the mechanism will continue to fill containers with such material. However, when a specified number of such containers has been filled with product from the fractionating column having less than said predetermined minimum characteristics, the counting mechanism will operate and change the solvent.

In view of the foregoing, it is clear that a wide variety of circuits may be provided for performing these functions and the invention contemplates the use of any thereof. The illustrated, and hereinafter described, circuit is, therefore, presented as one specific example for the purposes of illustration only.

Referring now to Figure 18, it will be understood that plug **1**, as shown therein connects wth plug *1a*, shown in Figure 16, plug **2** with plug *2a*, and so on.

Energy is supplied to this portion of the circuit from the power source **400**, through a suitable fuse **401** and a switch **402**, to the terminals **403** and **404**.

The 150 volt D. C. power supply for other parts of the circuit may be of any conventional type such as shown in Figure 19, wherein the 115 volt A. C. source is supplied to a transformer **440**, which in turn supplies a conventional rectifier **441** and capacitor **442** circuit for supplying a substantially constant potential of selected amount between the conductor **443** and the conductor **444**. For convenience in the several circuits shown and described herein, including particularly Figure 18, connection to this supply is shown merely as "150v" and referred to generally as a 150 volt source.

Attention is now directed to the turntable starting circuit which is substantially enclosed by the broken line **406** (Figure 18), and by which an impulse received from the fill control circuit (Figure 16) is caused to energize the solenoid **62** by which rotation of the turntable **11** is initiated. The terminal **407** of plug **1** engages the terminal *417a* of plug *1a* (Figure 16) and the conductor **266** leading therefrom. In Figure 18, terminal **407** is connected to one side of the relay winding **408**. The other side of said winding is connected through the resistor **409** and the relay winding **410** to one side of the capacitor **411**. The other side of said capacitor **411** is connected to the conductor between the winding **408** and the resistance **409** and then to a 150 volt supply.

The winding **408** is associated with normally open relay contacts **412** and **413** and with a normally closed contact **414**. The said other side of the winding **408** is connectable through the relay contact **413** to the relay winding **415**, in the pulse recorder circuit **420**, and thence to ground for purposes appearing hereinafter. The terminals **416** and **417** of plug **2** are connectable through relay contact **412** and through a normally closed relay contact **418**, associated with the winding **410**.

Thus, an impulse entering the system at the terminal **407** will energize the relay **408** and, eventually, also the relay **410**. However, because of the presence of the capacitor **411** the relay **410** is energized a fraction of a second later than the relay **408**. With the changing of the contacts **412**, **413**, and **414** from the positions shown in Figure 18, there is now provided a circuit from the 150 volt source through the contact **413** to the relay winding **415** in the pulse generator for the recorder and this is eventually manifested on the automatic recording device **163** by which the machine may be caused to keep a record of the number of containers utilized. Similarily contact **412** closes and provides a connection between the contacts **416** and **417** of plug **2** and thereby actuates the solenoid **62** which operates the turntable clutch **45** and thereby causes the turntable to turn. As soon as the micro-switch **72** moves off the cam **23**, it makes contact and holds the solenoid **62** energized. A moment after contact **412** closes, the relay winding **410** operates and opens the contact **418**. This breaks the solenoid supply through the fill control circuit and the solenoid remains energized only through the micro-switch **72**. When it reaches its next operating position, the next cam **23** opens the micro-switch **72** (Figure 2) by which the solenoid **62** is de-energized and the spring **63** operates the brake and causes the turntable **11** to stop in its next operating position. The capacity of the capacitor **411** is so chosen that the fill control circuit will not cease supplying the solenoid until after the micro-switch **72** has closed.

The pulse recorder circuit and its optional equipment are indicated by the broken line **420**. It functions solely to operate the automatic recording device **163** (Figure 13) by which the containers filled with product from the fractionating column may be visually counted and, by suitable connection thereof with the comparator circuit, the record will also indicate the amount of selected material from the fractionating column which is contained in a given container. This circuit **420** consists of a relay winding **415**,

operating with a normally open contact 421 for providing a pulse in one direction, and a relay winding 422 operating the relay contacts 423 and 424, together with a battery 425, to provide a pulse in the other direction.

When the turntable operates and a pulse comes from the circuit 406 through the relay contact 413, the winding 415 is energized and closes the relay contact 421. This enables an output voltage from the comparator circuit appearing at the terminal 429 of plug 3 to travel through the conductor 428 to the junction 438 and thence through the upper, normally closed side of the contacts 423, thence through the now closed contact 421, through the normally closed side of the contacts 424 and thence through the conductor 426 to the terminal 427 of plug 4, whereby to appropriately energize the recorder 163.

However, when the comparator circuit indicates that the concentration of solute in the product of the fractionating column has passed a predetermined concentration, a pulse will also be supplied to the terminal 430 in plug 3, which energizes the winding 422. This shifts the contacts 423 and 424 and enables the battery 425 to provide a pulse traveling through the lower side of the contacts 423, through the contact 421, through the lower side of the contacts 424, through the conductor 431 and thence to the terminal 432 of plug 4, thus energizing the recorder 163 in the opposite direction.

Attention is now given to the operation of the stepping relay, that is, the counting circuit.

Referring again to Figure 18, the relay 450 controls the interpositioning of the camparator circuit; the step relay 451 effects the counting; the multiple-switch 452 actuates the solvent valves after one predetermined number of counts by the step relay 451; and the multiple-switch 453 controls the solvent valves after a different count by the step relay 451. The relay 454 determines which of said multiple-switches are effective at a given time. The relay 455 responds to the count of a step relay as selected by one of said multiple-switches and controls both the position of the solvent valves and the illumination of the indicator lamps 449, which latter may be used if desired. Individual parts are all of standard construction and may be of many commercial types. The relay 456 is a reset relay by which the step relay 451 is caused to return to its beginning point upon the occurrence of any of a variety of circumstances.

The relay 450 is provided with four simultaneously operating contacts 466, 512, 513 and 509, which are normally positioned as shown in Figure 18, but which upon actuation of the relay will move into their opposite positions. The relay 456 has three contacts of which the contacts 478 and 494 merely open and close and contact 467 moves from one pole to the other. In rest position these contacts are as appearing in Figure 18, but upon actuation of the relay winding they move into their respectively opposite positions. The step relay 451 has four banks with simultaneously operating arms and this relay is so constructed that each arm moves clockwise one step, as appearing in Figure 18 upon each actuation of its relay winding. The switches 452 and 453 each have a pair of simultaneously acting arms and multiple banks of terminals. The terminals of bank B of switch 452 are connected to the respective terminals of bank B in switch 453, and respectively to the terminals of bank B of the step relay 451. The terminals of the bank A of the two multiple-switches 452 and 453 are also interconnected, respectively, to each other and respectively to corresponding terminals of bank A of the step relay 451. The arms of banks A and B of switch 452 are connected to the upper side of the contacts 460 and 461 of the relay 454 and the arms of banks A and B of switch 453 are connected to the lower side of the contacts 461 and 460, respectively, of relay 454. Thus, the position of said pair of contacts 460 and 461, as detailed hereinafter, determines which of said switches 452 and 453 is operating with the step relay 451 in a given instance.

The step relay 455 is provided with three simultaneously operating banks of switches A, B and C. An impulse comprising the output of the step relay 451 and one of the multiple-switches, hereinafter described in detail, is carried by conductor 462 through the relay 497 to the winding of step relay 455 for energizing same. Bank A of the step relay 455 is connected to function as a homing connection for said relay. The contacts of bank B are respectively connected through plug 5 and plug 5a (Figure 1) to the several solvent valve solenoids 158 and the several terminals of bank C are connected, if desired, to a series of indicating lamps 449. The lamps 449 will indicate the position of said respective relays depending upon whether their energization comes from bank C of relay 455 or bank D of relay 451 which is determined by the switch 464.

Examining this part of the circuit in more detail, it will be understood that a pulse emanating from the fill control circuit (Figure 16) through the plugs 1a and 1 will follow conductor 465 through contact 466 of relay 450 and through contact 467 of relay 456 to the winding of the step relay 451. Thus, each time the winding of relay 261 (Figure 16) closes the contacts 265, the winding of relay 451 is energized and each of the arms thereof move one step.

When positioned as shown in Figure 18, no energy flows through the step relay 451. When the arm of bank A of relay 451 moves to terminal 463, the ground connection 468 becomes connected through terminal 463 to terminal 469 in bank A of switch 452. Since, in the position shown, switch 452 has been set for operation with terminal 470, no further connection is made by this position of the step relay. However, as the arm of the step relay 451 continues to advance, it eventually contacts terminal 471 whereby a connection is completed to terminal 470 in bank A of the switch 452 and current flows through conductor 472 to contact 460 of relay 454. Contact 460 being normally closed, as shown in Figure 18, the current flows through the relay 454, the conductor 473 and to the winding of the relay 456. This actuates same and initiates the homing function for the step relay in a manner described hereinafter.

At this same time the arm of bank B of relay 451 is in contact with terminal 474 thereof and a connection is thus made to terminal 475 in bank B of the multiple-switch 452. This conducts current through the conductor 476 through the normally closed side of contacts 461 of the relay 454 and thence through conductor 477, through the normally closed contact 478 of the relay 456 and then through the conductor 462 to the normally closed side of the contacts 479 in relay 497. From the contact 479, the pulse follows conductor 480 and energizes the winding of relay 455. This advances the arms of said relay 455 one step in the usual manner. Bank B of relay 455 is, in this specific embodiment, the bank which is connected

to the solvent valve solenoids 158 and hence the power source 270 shown in Figure 16 is connected to those terminals of plug 1a which contact terminals 404 and 403 of plug 1. Therminal 403 is connected through conductor 481 to terminal 482 of plug 5 and, terminal 404 is connected through conductors 483 and 459 to the arm of bank B of step relay 455 and thence, in the particular setting shown in Figure 18, through conductor 484 to terminal 485 of plug 5. Other terminals of plug 5 are correspondingly connected. Thus, the particular solvent valve which is held open is determined by the position of the arm of bank B of stepping relay 455.

Accordingly, in this particular setting of the apparatus, each pulse from the fill control circuit (Figure 16) will move the step relay 451 one step and, as soon as arms thereof reach the terminals corresponding to the terminals upon which the arms of the multiple-switch 452 have been preset, a current will be supplied to the winding of the step relay 455 and the connection change to a new solvent valve solenoid. Thus, one solvent valve is closed and one is opened after a number of pulses from the fill control circuit, which number is controlled by the manual setting of the arms of the multiple switch 452.

However, it is sometimes desirable to count one number of containers 12 following the first opening of a particular solvent valve and to count a different number following the completion of a band of product from the fractionating column. In order to provide for these two sets of counts, the multiple switch 453 is also provided, together with the relay 454. The switch 453 has the terminals of its banks A and B connected, respectively, to the terminals of banks A and B of the stepping relay 451 in the same manner as the switch 452. Its arms, however, are connected to the opposite sides of the contacts 460 and 461 from those to which the arms of the switch 452 are respectively connected. Thus, in the position shown in Figure 18, the switch 452 is operatively connected to the system. However, upon the actuation of the winding in relay 454 the contacts will be reversed, which will result in disconnecting the multiple-switch 452 and connecting switch 453 in its place. Since these two switches may have their arms set independently of each other, it will be apparent that the number of steps taken by the step relay 451 prior to the passage of current through the contacts of relay 454 will be determined by the position of the contacts 460 and 461 of the relay 454.

Thus, when the winding of the relay 454 is energized and its contacts are reversed from the positions shown in Figure 18 and the arms of banks B and A of the relay 451 have reached the terminals 490 and 491, respectively, current is conducted from the step relay 451 to bank B of the switch 453, thence through the connector 492 to the lower side of the contacts 460 and thence through the conductor 473 to the reset relay for initiating same in the manner to be described hereinafter.

Simultaneously current is conducted through bank A of switch 453 and through the conductor 493 to the lower side of the contacts 461 and thence through the conductor 477 through the same path as previously described through the contact 478 of relay 456 and back to the conductor 462 to energize the winding of the relay 455, and again advance said step relay 455 and effect a further change of the solvent valves.

Thus, the solvent valves will be actuated after whatever number of steps of the step relay is selected by whichever of the multiple switches 452 and 453 is connected into the circuit.

Upon the operation of a solvent valve it becomes desirable to return the step relay 451 to zero position. This is effected by the reset relay 456 which, as above described, is actuated each time a solvent valve is changed. Upon the energizing of the relay 456, the contact 494 closes to lock the relay in reset position through the bank C of the step relay 451. The contact 478 is opened in order to terminate further flow through the winding of relay 455 and thus make sure that relay 455 is advanced only one step. The contacts 467 change to the position opposite that shown in Figure 18 and thus the vibrator portion of the step relay 451 is connected into an energized circuit and the step relay 451 returns to zero in its usual manner. Upon the attainment of zero position, the connection through contact 494 is broken by bank C of the step relay 451 and the reset relay 456 returns to its normal operating position as shown in Figure 18. The switch 495 may be used manually to initiate the homing function just described and the switch 496 may be used to advance the step relay by hand through its normal operating cycle.

The step relay 455 is similarly provided with a reset relay 497 which functions on the actuation of the switch 498. This closes the contact 499 to hold the relay in its energized position and shifts the contacts 479 to initiate operation of the vibrator of said relay 455. This then returns relay 455 to its zero position in the usual manner.

Turning now to the means operating the relay 454, it will be recalled that this relay is intended to operate with each change of solvent. Thus, each pulse in conductor 480 operating the winding of stepping relay 455 also operates the winding of relay 500, which closes the normally open contacts 501 and 502 therein. Closure of contact 501 completes the circuit between the terminals 683 and 684 of plug 4 which is connected to the recording mechanism 163 for indicating thereon that a change in solvent has taken place. Closure of contact 502 causes energizing of the winding of the relay 454. The resistor and capacitors connected with said winding of relay 454 are provided to delay the full energization and therefore the actuation of relay 454 a sufficient length of time after the pulse appears in the conductor 480 to ensure that the two step relays have had sufficient time to complete their resetting to their respective zero positions before the relay 454 effects switching from switch 452 to switch 453, or vice versa. This is to prevent the recycling which might otherwise occur if one switch is set near its extreme rightward position and the other switch is set near its extreme leftward position and the switching is completed while the step relay occupies a position intermediate the setting of said two switches.

When the winding of the relay 454 is energized, the normally open contact 507 is closed and a circuit is thereby established through the conductor 508, through the normally closed contact 509 in relay 450 and back through the conductor 510 which is connected through the winding of the relay 454 a 150 volt source. Thus, momentary energizing of said winding of relay 454 will close this circuit and thus hold said relay in the position for keeping switch 453 in the circuit, until said circuit through the contact 507 is

broken by the actuation by the relay 450 and the consequent opening of the contact 509 therein.

The capacitor 504 is provided to hold the relay 500 in closed position sufficiently long that the capacitors associated with relay 454 may receive sufficient charge to effect the time delay provided by their presence.

The contacts 521 in the relay 454 are respectively connected to the lamp 522 and the lamp 523, which lamps indicate the position of said relay 454 and thereby indicate which of the two multiple switches is operating at a given moment. The switch 505 may be provided to effect manual stepping of the relay 455.

The operation of the comparator circuit (Figure 17) with the foregoing described counting and control circuit will now be apparent.

The relay 450, it will be observed, is here provided with a pair of normally closed contacts 466 and 509, and a pair of normally open contacts 512 and 513. The winding of relay 450 is connected at its one end to a 150 volt D. C. source and its other end is connected through the conductor 506 to terminal 430 of plug 3. Thus a connection made in the comparator circuit (Figure 17) will energize the winding of the relay 450 and reverse its several contacts from the positions shown in Figure 18. By thus opening the contact 466, subsequent pulses from the fill control circuit will be stopped at the open contacts 466 and the step relay will not operate. By opening the contacts 509 the circuit including the conductor 510 and the winding of the relay 454 will be opened and the contacts of this relay will all be returned to the position shown in the drawing. Accordingly, switch 453 will be cut out of the circuit and switch 452 will be connected.

The circuits including the relays 531 tnd 530 may, if desired, be provided to effect turning of the turntable 11, for the purpose of placing an empty container in filling position, either when the concentration of a selected solute in the product of the fractionating column becomes less than said predetermined minimum, or when said concentration, after being less than said minimum, becomes greater than said minimum. This latter feature ensures that fluid containing more than the minimum of solute will not be mixed in any one container with fluid containing less than said minimum of solute. This is accomplished by conducting an impulse from the comparator circuit (Figure 17) to the circuit 406 (Figure 18) and causing same to actuate the solenoid 62 of the turntable 11 even though the container then in filling position is not yet filled.

For this purpose the winding of the relay 531 is connected in parallel with the capacitor 532, one side of which is connected by the conductor 533 to a 150 volt source, and the other side of which is connected through the normally closed contacts 534 of the relay 514 to ground. Thus, when the relay 450 is actuated by the pulse from the comparator circuit in the manner above described, the contact 512 closes and an impulse is conducted thereby through the normally closed contact 535 of relay 531 and through the conductor 536 to the winding 408 of the turntable starting circuit 406. This effects movement of the table in the same manner as above described for movement effected by the filling of a container. However, once the comparator circuit has been actuated, the circuit must be broken in order to permit the subsequent operation of the table to continue under the sole control of the fill control circuit. This is accomplished by the relays 514 and 531. When the relay 450 is energized the contact 513 closes and, through the conductor 538, the winding of the relay 514 is energized. This opens the contacts 534 and de-energizes the winding of the relay 531. However, such de-energizing is delayed slightly by the capacitor 532 and such delay is of sufficient duration that the above mentioned impulse for initiating rotation of the turntable has time to travel through the contacts 535 of relay 531 as above described. When the relay 531 is de-energized, the contacts 535 open and this circuit has no further function. A similar pulse is conducted momentarily through contacts 517 and 515 and through the conductor 518 to actuate the relay 456 and reset the step relay to zero.

When the selected solute in the product from the fractionating column becomes less than the predetermined amount, the relay 530 also functions to shift the container and again reset the step relay. As soon as the relay 514 is energized the contacts 529 are closed, thereby energizing the relay 530 which closes its contacts 527 and 528. Thus, a connection is made through the contact 526 of the relay 514 and the contact 527 of the relay 530 to the winding 408 of the relay in the turntable starting circuit 406. This circuit, however, is not complete due to the contacts 526 being opened during the period that the winding of the relay 514 is energized, which continues for so long as a band of solute is coming down from the fractionating column. However, as soon as said band terminates, the relay 514 becomes deenergized due to the opening of the contacts 513 of the relay 450 and thus the contacts 526 return to closed position and an impulse is delivered to the fill control circuit. Simultaneously, the contact 529 is broken which deenergizes the winding of the relay 530, but the capacitor 545 in parallel with said winding of relay 530 delays said de-energizing sufficiently to permit said pulse to pass the contacts 527 and thus initiate a movement of the turntable 11. Thus, when a band has ended, the containers will be shifted by action of the comparator circuit, but immediately thereafter this portion of the circuit is disconnected and further rotation of the turntable 11 will be under the control solely of the fill control circuit. The contacts 528 of the relay 530 will function, in a manner which will be obvious in view of the foregoing, through the contacts 515 of the relay 514 to reset the step relay 451 to zero in the event that it has, for any reason, moved out of such position during the gathering of the band.

THE RECORDER ACTUATING CONNECTIONS

The recorder 163 (Figures 13, 20 and 21), although optional, is convenient for the purpose of providing a visual record of the entire fractionating process. Said recorder 163 may be conventional in structure, circuitry and operation. It is powered from any convenient source, such as the terminals 681 and 682 of plug 4 (Figure 18), or, alternatively, from a completely independent source. Said recorder 163 preferably provides a continuous record proportionate to the relative potentials applied to it through the terminals 673 and 676 of plug 3a in the comparator circuit (Figure 17). It is also connected so that the potential applied to it at the terminals 683 and 684 of plug 4 (Figure 18) will cause a momentary movement of the pen 165 (Figure 13) recording an indication that a solvent has been changed. Said recorder 163 is further connected so that an im-

pulse appearing at the terminals 427 and 432 of plug 4 will cause a momentary movement of the pen 164 indicating that a container has been filled and an empty one put in its place.

SUMMARY

Figure 20 illustrates schematically the entire mechanism described in the foregoing paragraphs and illustrated in detail in the other figures of the drawings. In view of the complexity of the several parts, Figure 20 is believed helpful and desirable to summarize the entire disclosure and operation of the instrument 10. However, since said parts are believed to have been fully described herein, further specific reference to Figure 20 is believed unnecessary.

The operation of the apparatus, both mechanical and electrical, will now be summarized. Assuming that insufficient amounts of solute are being dissolved by a solvent passing through the fractionating column at the beginning of operation of the instrument 10, but that the product is being collected in a container, the comparator circuit will not be activated. Under these circumstances, the turntable is motionless; the fluid control mechanism is activated; the step relay is ready for operation, but is unactivated; the relay 454 is energized and holding the switch 453 in the circuit; and the step relay 455 is unenergized, but ready for operation. When the container is filled, the fill-control circuit will close the contacts 265 (Figure 16) and, through the circuit 406 (Figure 18), actuate the clutch solenoid 62 to turn the table 11 to place the succeeding, empty container 12 under the fractionating column. Simultaneously a pulse is delivered to the winding of the step relay 451 and its several arms each move to the first terminal of each respective bank therein.

Assuming that the multiple switches 452 and 453 are set at a point beyond the first terminal, such as the respective points shown in Figure 18, nothing further will happen, and a second container will be filled in the same manner as the first. According to the setting of the switch 453 in Figure 18, four of such containers will be filled even though said solvent is dissolving in sufficient amounts of solute to close the contact 664 in the relay 643 of the comparator circuit. Upon the filling of the fourth container the step relay will effect the following operations: make a contact through switch 453 and relay 454 to activate step relay 455 and move bank B thereof from the first terminal to the next adjacent terminal and thus effect a change in solvent introduced into the fractionating column 15; energize the reset relay 456, also through switch 453, by which the step relay 451 is returned to zero; supply an impulse to the recorder 163 to record the fact that the next solvent is now being used; the position of the relay 454 will be unchanged. If the step relay again goes through four positions without enough solute being dissolved by the new solvent to actuate the comparator circuit, the above described operation will be repeated. However, if sufficient solute is dissolved this time to actuate the comparator circuit, the following will occur: the relay 450 will operate and break the circuit to the winding of the step relay 451, thus terminating the counting function; energizing of the turntable starting circuit 406 through relays 514 and 531 to bring the next container into filling position; the circuit to the relay 454 will also open thus de-energizing this relay and taking the multiple switch 453 out of the circuit and

placing the multiple switch 452 into the circuit. This condition will continue for so long as the comparator circuit is activated, although each time that a container is filled the fill-control circuit, including the turntable solenoid 62 and the portion of the recorder responsive thereto, will each be independently operated.

When a band of solute is completed and the concentration of solute in the solution falls below the predetermined minimum, the contact 664 of relay 643 in the comparator circuit will open and the following changes will occur: the relay 450 will be de-energized and will return to a position shown in Figure 18; the turntable will bring a new container into filling position; the winding of the step relay 451 will be energized and will again commence counting the containers filled with the product of the column 15; the relay 454 (Figure 18) will remain de-energized and the multiple switch 452 will remain in the circuit. As soon as the number of containers, determined by the multiple switch 452, has been filled with solution having less than the predetermined amount of solute therein, a connection will then be made through the switch 452 and the relay 454 to the relay 455 for again changing the solvent, and simultaneously another connection will be made by the relay 456 for returning the step relay 451 to zero position, and, lastly, a pulse will be conducted to the recorder 163 for indicating visually another change in solvents. The cycle will then repeat, and it will continue to do so, for as long as the operator desires. If it is desired to supply a larger number of solvents to the apparatus then there are connections on bank B of step relay 455, it will be necessary to return this relay to zero and replace the first group of solvents in the respective receptacles 141 with the next appropriate group.

CONCLUSION

It will be understood that the certain parts herein described, both the mechanical portions thereof and the electrical circuits utilized, are matters of choice and that they may be widely substituted by other equally well known elements. Such substitutions are contemplated within the scope of this invention and, unless they are specifically excluded from the scope of the hereinafter appended claims, they are to be considered as included within the meaning thereof.

We claim:

1. The method of carrying out a chromatographic fractionating operation employing a series of solvents in conjunction with a column, comprising the steps: withdrawing solution from a fractionating column; passing through said solution a beam of radiant energy of a character capable of being physically affected by a selected material expected to be in said solution; measuring the magnitude by which said beam is affected thereby and utilizing the affecting of said beam for changing solvents when said magnitude changes beyond a predetermined value.

2. In a device for separating a mixture of soluble materials, the combination comprising: a fractionating column having an inlet port and an outlet port; a plurality of containers providing independent sources of a plurality of solvents and means including valves for selectively conducting one of said solvents to the inlet port of said fractionating column; first opto-electric means including a conduit connected to the outlet port of said fractionating column for generating an electric potential variable proportionally

to, and immediately upon the occurrence of changes in, the concentration of a solute having predetermined characteristics in the discharge from said fractionating column; a conveyor for successively advancing a plurality of containers into and out of position with respect to said conduit to receive said discharge; second opto-electric means responsive to the fluid level in the one of said containers being filled at a given time for effecting the advancement of said conveyor upon each filling of successive containers to a predetermined level, said second opto-electric means including counting means connected to said solvent valves for closing one and opening another thereof after a predetermined number of advancements of said conveyor; means responsive to said electric potential blocking operation of said counting means when said electric potential is above a predetermined value; whereby containers will continue to be filled so long as the concentration of a selected solute in the discharge from said column remains above a predetermined value and a counted number of containers will be filled after said concentration falls below said predetermined value and after said counted number of containers have been filled the solvent introduced into said inlet port will be changed.

3. In a device for separating a mixture of soluble materials, the combination comprising: a fractionating column having an inlet port and an outlet port; means providing independent sources for a plurality of solvents and means including valves for selectively conducting one of said solvents to said fractionating column; a source of radiant energy of such characteristics that it is physically affected by being passed through a solution wherein the solute is one of said soluble materials; a cell connected to the discharge port of said column for receiving solution discharged therefrom and said cell including a pair of aligned windows, one being on each side of a space through which solution received from said column is conducted, which windows are transparent to a beam of said radiant energy, said source being positioned to pass said beam through said aligned windows; a conduit connected to said cell for conducting solution away from said cell; first opto-electric means, including a first photo-electric responsive device positioned for receiving said beam after same passes through said cell and a comparator circuit, generating an electric potential variable in response to the concentration of a solute having predetermined characteristics in the discharge from said fractionating column; a conveyor successively advancing a plurality of containers into and out of position with respect to said conduit to receive said discharge; second opto-electric means responsive to the fluid level in the one of said containers being filled at a given time for effecting the advancement of said conveyor upon each filling of successive containers to a predetermined level, said second opto-electric means including counting means connected to said solvent valves for closing one and opening another thereof after a predetermined number of said advancements of said container, said counting means having a step relay and an electric circuitry associated with the winding of said step relay; means responsive to a predetermined value of said electric potential for breaking said electric circuitry, whereby upon said counting means is blocked and the solvent introduced into the inlet port of said fractionating column remains the same so long as said value is maintained.

4. In a device for separating a mixture of soluble materials, the combination comprising: a fractionating chamber having an inlet opening and a drain; means providing independent sources of a plurality of solvents and means including valves for selectively conducting one of said solvents to the inlet of said fractionating chamber; a source of electromagnetic radiant energy of such characteristics that it is absorbed by being passed through a solution wherein the solute contains one of said soluble materials; a cell connected to said drain for receiving solution discharged from said column and said cell being so positioned, and for including means, permitting a beam of said electromagnetic radiation to pass through said cell, and means including a conduit connected to said cell for conducting solution away from said cell; opto-electric means including a photoelectric responsive device positioned for receiving electromagnetic radiation passing through said cell and a comparator circuit, said opto-electric means providing an electric potential in excess of a critical value upon the attainment of a predetermined quantity of solute in the discharge from said fractionating column; a conveyor supporting a plurality of containers and arranged to advanced said containers successively for receiving said discharge directly from said conduit; means automatically advancing said conveyor each time a container is filled to a predetermined level; independent means automatically counting the advancements of said conveyor and adapted to close one solvent valve and open another after a predetermined number of said advancements; means responsive to said electric potential, when in excess of said critical value, for blocking said counting means.

5. In a device for separating a mixture of soluble materials, the combination comprising: a vertically arranged tubular member containing said mixture and including an inlet port and an outlet port; means providing independent sources of a plurality of solvents and means including valves for selectively conducting one of said solvents to said inlet port of said tubular member; a conveyor for successively advancing a plurality of containers into and out of filling position below the outlet port of said member to receive the discharge therefrom; opto-electric means positioned adjacent said filling position and responsive to the fluid level in a container being filled for effecting advancement of said conveyor, and electric circuitry actuated by said opto-electric means and including counting means connected to said solvent valves for closing one and opening another thereof after a predetermined number of said advancements; automatically operating opto-electric means positioned adjacent the discharge port of said tubular member and including electric circuitry comprising a comparator circuit, said opto-electric means being responsive to the concentration of a selected solute in the discharge from said tubular member for blocking and unblocking the operation of said counting means and connected to said counting means.

6. In a device effecting automatic fractionating of the soluble constituents of a mixture, the combination comprising: a fractionating column including an inlet port and an outlet port; means including valves for successively introducing into the inlet port of said column a plurality of solvents; a source of electro-magnetic radiation of predetermined wave-length; first liquid conductive means transparent to said electro-magnetic radiation exposed to said radiation and a first

conduit conducting discharge from said column to said first liquid conductive means; second liquid conductive means transparent to said electromagnetic radiation exposed to said radiation and a second conduit connecting the source of the solvent being introduced at any given time into said inlet port to said second liquid conductive means; a pair of photo-electric means responsive to said radiation and intercepting, respectively, those portions of said radiation having passed through said first and second liquid conductive means; a pair of amplifier circuits connected, respectively, to the outputs of said photo-electric means; a relay; a comparator circuit connected to the output of each of said respective amplifier circuits for opening and closing said relay in response to the differential between the output of one amplifier circuit and a predetermined percentage of the output of the other amplifier circuit; and electric circuit means responsive to the position of said relay for controlling the operation of said valves.

7. In a device for automatically effecting the separating of a mixture of compounds by a fractional extraction process, the combination comprising: a fractionating column including an inlet port and an outlet port; means including valves for successively introducing into the inlet port of said fractionating column a plurality of solvents; means producing a beam of light of a narrow band of predetermined wave-length; means positioned in the path of one portion of said beam for conducting the product of said column therethrough, said means including a pair of windows transparent to said beam and aligned in said path and a conduit connecting the discharge port of said column to said last named means; means conducting solvent through another portion of the path of said beam, said means also including a pair of windows transparent to said beam and aligned in its path and a conduit connecting the source of solvent being introduced into said column at any given time with said last named means; a pair of photo-tubes positioned, respectively, within each of said portions of said beam after same has passed said windows; an amplifier circuit operatively connected with the output of each of said photo-tubes; a relay; a comparator circuit connected to the output of both amplifier circuits and including means opening and closing said relay in response to whether the voltage output of one amplifier circuit is greater or less than a predetermined percentage of the voltage output of the other amplifier circuit; electrically responsive solvent control means controlling said valves; electrical means affecting the operation of said solvent control means in response to the position of said relay.

8. Apparatus as defined in claim 7 wherein said comparator circuit includes a gas filled electric discharge device, means connecting one of said amplifier circuits to the cathode of said electric discharge device, means connecting the other of said amplifier circuits to the control electrode of said electric discharge device, a relay and means responsive to conduction or non-conduction of said electric discharge device for opening and closing said relay.

9. In a device for automatically effecting the separating of a mixture of compounds by a fractional extraction process, the combination comprising: a fractionating column including an inlet port and an outlet port; means including valves for successively introducing into the inlet port of said fractionating column a plurality of

solvents; means producing a beam of light of a predetermined wave-length; first cell means positioned in the path of one portion of said beam for conducting the product of said fractionating column therethrough, said means including a pair of windows transparent to said beam and aligned in said path and a conduit connecting the discharge port of said column with said first cell means; second cell means conducting solvent through the path of another portion of said beam, said means also including a pair of windows transparent to said beam and aligned in its path and a conduit connecting the source of solvent being introduced into said column at any given time with said second cell means; a pair of photo-tubes positioned, respectively, within the paths of each of said portions of said beam after same have passed said windows; an amplifier circuit operatively connected with the output of each of said photo-tubes; a relay; a comparator circuit connected to the output of both said amplifier circuits and including electric means opening and closing said relay in response to the differential between the voltage output of one amplifier circuit and a predetermined percentage of the voltage output of the other amplifier circuit; a counting circuit including means physically, automatically and immediately reacting to a predetermined volume of discharge from said fractionating column for closing the valve to one solvent and opening the valve to another solvent; and an electric circuit responsive to the position of said relay for blocking said counting circuit as long as said differential in voltage outputs is in excess of a predetermined value.

10. In a device for automatically effecting the separating of a mixture of compounds by a fractional extraction process, the combination comprising: a fractionating column including an inlet port and an outlet port; means including valves for successively introducing into said inlet port of said fractionating column a plurality of solvents; means producing a beam of light of a narrow band of predetermined wave-lengths; first cell means positioned in the path of one portion of said beam for conducting the product of said fractionating column therethrough, said means including a pair of windows transparent to said beam and aligned with said path and a first conduit connecting the discharge port of said column with said first cell means; second cell means conducting solvent through the path of another portion of said beam; said means also including a pair of windows transparent to said beam and aligned in its path and a second conduit connecting the source of solvent being introduced into the column at any given time with said second cell means; a pair of photo-tubes positioned, respectively, within the paths of each of said portions of said beam after same have passed said windows; an amplifier circuit operatively connected with the output of each of said photo-tubes; a comparator circuit connected to the output of both said amplifier circuits and including a gas filled electric discharge device, means connecting one of said amplifier circuits to the cathode of said electric discharge device, means connecting the other of said amplifier circuits to the control electrode of said electric discharge device, a relay and means responsive to conduction or non-conduction of said electric discharge device for opening and closing said relay, said last-named means including a pair of alternative circuits and a switch, said circuits and said switch alternatively connecting, accord-

ing to the position of said switch, the control electrode of said electric discharge device with said other amplifier circuit, one of said alternative circuits having substantially greater resistance therein than the other, and means for changing said switch from one of said alternative circuits to the other with each change in the conduction condition of said electric discharge device.

11. A device as claimed in claim 5 wherein there is also provided a conveyor for successively advancing containers into position for immediately receiving solution being discharged from said fractionating column and through said first cell means; and circuitry connected to said comparator circuit and responsive to each change therein to or from relay actuating condition for advancing said conveyor, at the beginning and end of a band of solute of predetermined characteristics being discharged from the fractionating column.

12. In a device for automatically effecting the separating of a mixture of compounds by a fractional extraction process, the combination comprising: a fractionating column including an inlet port and an outlet port; means including valves for successively introducing into said inlet port of said fractionating column a plurality of solvents; a first conduit connected to said outlet port; a second conduit connected to the source of solvent being introduced into said fractionating column at any given time a conveyor for carrying a plurality of containers into position for receiving discharge directly from said first conduit; circuitry including a step relay and first opto-electric means energized by the discharge through said first conduit of a sufficient quantity of solution for filling one of said containers for actuating said relay one step each time one of said containers is filled, and further circuitry connected to said last named circuitry responsive to the attainment of a predetermined position of said step relay for closing one solvent valve and opening another solvent valve; a relay interposed in the circuit of the winding of said step relay; a comparator circuit and second opto-electric apparatus operatively associated with said first conduit and with said second conduit for automatically actuating said comparator circuit in response to the differential between the light absorbing characteristics of a solvent being introduced into said fractionating column at a given time and the light absorbing characteristics of the product of said column at said given time, and an electric circuit having its input connected to the output of said comparator circuit and having its output connected to the winding of said relay whereby said last-named circuit will be responsive to said actuation of said comparator circuit for opening and closing said relay; whereby, upon the existence of a differential in the light absorbing characteristics between the product of said fractionating column and the solvent used at a given time in excess of a predetermined value, said step relay will be held inactive.

13. Apparatus as described in claim 12 including a beam of illumination and a photo-sensitive means responsive thereto, said beam and said photo-sensitive means being so positioned with respect to a container receiving discharge from the fractionating column that said beam will be refracted by the liquid discharge in the container onto said means; and an electric circuitry actuated by energization of said means to move said conveyor and thereby move one container out of filling position and another container into filling position.

14. A device as described in claim 2 having also the following elements: a clutch and brake for alternately effecting movement of said conveyor and for braking same and electrically responsive means for effecting alternative engagement of said clutch and of said brake; a plurality of cams along said conveyor, each corresponding to one filling position thereof; a switch and cam follower operating said switch in response to movement of cams, said switch being in circuit open position when said conveyor is at rest with one of said containers in filling position and said switch being in circuit closed position during movement of said conveyor from one filling position to the next; opto-electric means responsive to the liquid level in said containers and providing an electrical impulse of relatively short duration to said electrically responsive means independently of said switch; an electric circuit connected to said switch and effective to energize said electrically responsive means when said switch is closed; whereby movement of said conveyor will be started by said opto-electric means when said liquid level is reached and will be continued to the next filling position by the closing of said switch and then braked.

15. Apparatus for carrying out a chromatographic fractionating operation to extract a selected material from a mixture including said material and employing a series of solvents in conjunction with a column, the combination comprising: a chromatographic fractionating column including an inlet port and a discharge port; means including conduits and a plurality of electrically actuable valves for introducing a plurality of selected solvents successively into the inlet port of said column; a source of radiant energy of such characteristics that it is physically affected by being passed through a solution comprising one of said solvents and said material; a cell connected to the discharge port of said column for receiving solution discharged therefrom, said cell including means permitting a beam of said radiant energy to pass through said cell, and said source being positioned with respect to said cell to pass a beam of said radiant energy through said cell; conduit means connected to said cell for conducting said solution away from said cell; photo-electric means receiving said beam after said beam passes through said cell and responsive to the magnitude to which said beam is affected by passing through solution within said cell; and an electrical circuit connected to said photo-electric means for actuating said valves immediately upon the diminution to a predetermined point of the concentration of said material in the solution within said cell for closing one of said valves and opening another thereof.

16. Apparatus as described in claim 15 wherein said electric circuit includes a comparator circuit and including means automatically resetting said counting means to zero whenever the output from the comparator circuit exceeds said critical electric potential.

17. Apparatus for carrying out a chromatographic fractionating operation to extract a selected material from a mixture including said material and employing a series of solvents in conjunction with a column, the combination comprising: a chromatographic fractionating column including an inlet port and a discharge port; means including conduits and a plurality

of electrically actuable valves for introducing a plurality of selected solvents successively into the inlet port of said column; a source of electromagnetic radiation of such wave-length that it is absorbed by being passed through a solution comprising one of said solvents and said material, said absorbing being proportional to the concentration of said material in said solution; a cell connected to the discharge port of said column for receiving solution discharged therefrom, said cell including means permitting a beam of said electro-magnetic radiation to pass through said cell, and said source being positioned with respect to said cell to pass a beam of said radiant energy through said cell; conduit means connected to said cell for conducting said solution away from said cell; photo-electric means receiving said beam after said beam passes through said cell and responsive to the intensity of said beam after it has passed through solution within said cell; and electrical means connected to said photo-electric means for actuating said valves immediately upon the diminution to a predetermined point of the concentration of said material in the solution within said cell for closing one of said valves and opening another thereof.

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