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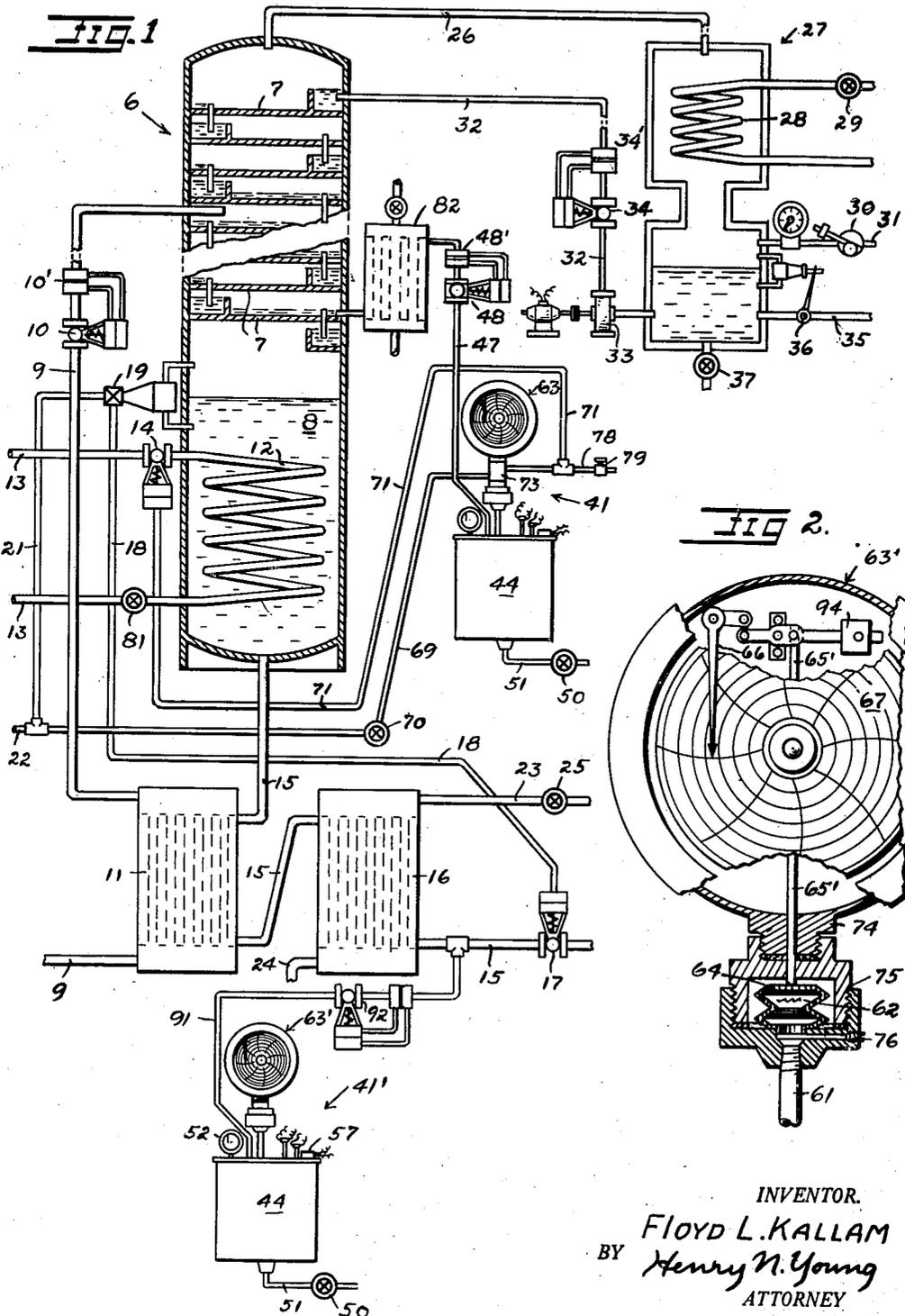
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VOLATILITY TESTER

Filed July 20, 1935

2 Sheets-Sheet 1



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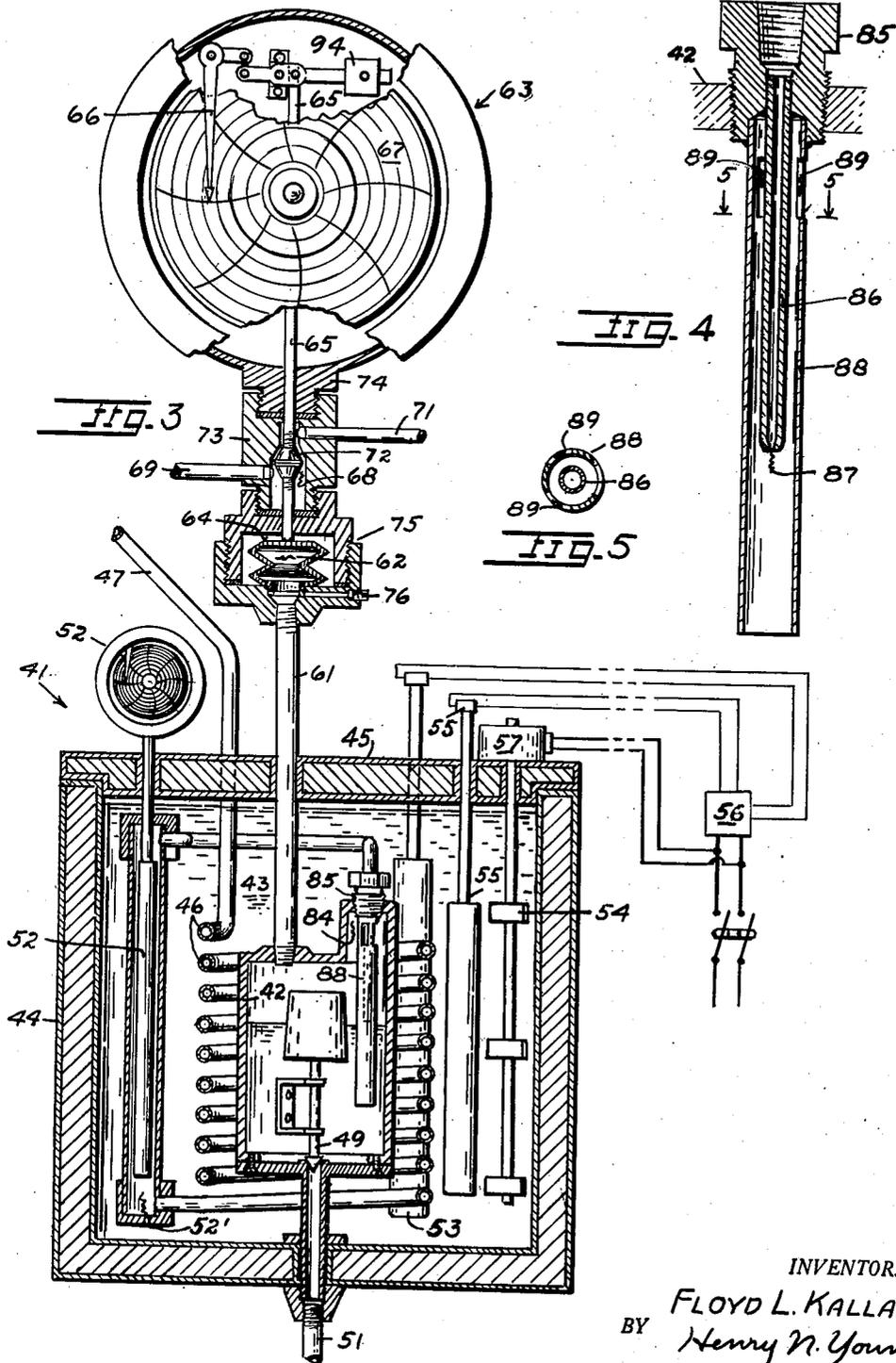
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# UNITED STATES PATENT OFFICE

2,119,786

## VOLATILITY TESTER

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Application July 20, 1935, Serial No. 32,365

7 Claims. (Cl. 73—50)

The invention relates to means for measuring the volatility of a continuous sample of a liquid, and the present application comprises a continuation in part of my copending application for United States Letters Patent on a Fractionator control device, Serial Number 703,574, filed Dec. 22, 1933, and subsequently issued into Patent No. 2,086,808 on July 13, 1937.

A general object of the invention is to provide, for association with a continuously operating fractionating apparatus, an improved means to directly and continuously indicate and/or record the volatility of a liquid in the apparatus for use in effecting a control of the apparatus so that a product of the apparatus may have a constant volatility.

A further and more specific object is to provide a generally improved volatility tester of the type described.

Yet another object is to provide a device of the character described which is readily applicable as an accessory to present fractionators.

The invention possesses other objects and features of advantage, some of which, with the foregoing, will be set forth or be apparent in the following description of a typical embodiment and application of the invention which is illustrated in the accompanying drawings, in which,

Figure 1 is a generally diagrammatic showing of an application of the volatility tester of the present invention is a fractionating system, certain elements of the apparatus being shown in sectional elevation.

Figure 2 is an enlarged fragmentary and partly sectional view of a meter portion of the tester.

Figure 3 is an enlarged sectional elevation through a testing unit embodying the present invention.

Figure 4 is an enlarged longitudinal section through a nozzle of the unit.

Figure 5 is a transverse section at 5—5 in Figure 4.

As particularly illustrated, the device of the present invention is associated with fractionating apparatus in which the controlled product comprises a mixture of the less volatile fractions of hydro-carbon liquids such as fuel oils and gasolines and alcohols, etc. The apparatus shown comprises a conventional bubble-tray tower 6 having provided in the upper and major portion thereof a plurality of superimposed bubble trays 7 of suitable structure. The lower tower portion is seen to provide a kettle portion 8 in which to heat the liquid being treated. A duct 9 supplies the raw liquor to the bubble trays at a constant

rate; as shown, the valve 10 is provided for controlling the flow of liquor through the duct 9, and a heat-exchanger 11 is operatively interposed in said duct whereby to preheat the liquor.

The liquid in the kettle 8 may be heated to the desired temperature by means of a heating coil 12 interposed in a pipe 13 through which a heated fluid is continuously passed; in practice, the heating fluid is usually steam and the liquid in the kettle is heated to boiling therewith. A valve 14 controls the fluid flow through the pipe 13 and coil 12 whereby, with the other operative factors of the apparatus held constant, the rate of heat discharge from the coil may be adjusted to a desired degree.

The unevaporated liquid is arranged to constantly escape at the bottom of the kettle through a duct 15. As particularly disclosed, the heat-exchanger 11 is interposed in the duct 15 whereby the inflowing liquor to the fractionator is preheated and the liquid discharging through the duct 15 is partially cooled therein. A second heat-exchanger 16 is interposed in the duct 15 at the delivery side of the heat-exchanger 11 for completing the cooling of the discharged liquid.

For maintaining a constant liquid level in the kettle, a valve 17 is provided in the duct 15 at the discharge side of the heat-exchanger 16. The valve 17 is preferably arranged for automatic operation; as shown, this valve is of the diaphragm-motor type and is connected by means of an air duct 18 with a float-controlled valve 19 which is operated by the liquid level in the kettle. A duct 21 supplies the operating air to the valve 19 from a compressed-air main 22 connected with a suitable source (not shown) of compressed air. It is to be understood, however, that the valve 17 might be arranged for mechanical, rather than pneumatic, operation by means of a suitable float-actuated mechanism.

A cooling liquid from any suitable source is arranged to flow to and from the heat-exchanger 16 through ducts 23 and 24 respectively, and a flow control valve 25 is provided in a said duct. Both generally, and for a specific purpose to be hereinafter brought out, it is desirable that the liquid product be delivered from the heat-exchanger 16 at a substantially fixed and predetermined temperature, and the control of said temperature is conveniently arranged to be effected through an automatic or manual adjustment of the valve 25.

The gaseous products resulting from the heating and other treatments of the liquor in the tower 6 are arranged to flow from the top of the

tower through a duct 26 and to a dephlegmator or condenser 27 wherein a portion of the gases are condensed to form a distillation product which comprises the top product and is known in the art as a reflux liquid. The desired cooling in the condenser 27 is arranged to be effected by means of a cooling coil 28, a valve 29 controlling the fluid flow rate through the coil. The uncondensed gases from the condenser 27 are arranged to escape, as to waste or for use as fuel, through a duct 31 having a suitable back-pressure valve 30 therein.

Part of the condensate, or reflux, in the condenser 27 is arranged to be delivered back to the tower through a duct 32, a motor driven pump 33 being interposed in said duct for the purpose. A valve 34 is also interposed in said duct at the discharge side of the pump 33. Excess reflux is discharged from the condenser through a duct 35, the latter duct having therein a float-controlled valve 36 for automatic operation to maintain a constant reflux level in the condenser. A drain valve 37 for removing any condensed water is preferably provided at the bottom of the condenser.

It will be understood that the present apparatus is particularly arranged to fractionate the raw liquor delivered thereto into three parts, namely, a bottom fraction of lowest volatility which is discharged through the duct 15 as a liquid product, a condensed top fraction which is discharged through the duct 35 as a liquid top product, and the lighter and high-volatility fraction which does not condense at ordinary temperatures and is discharged through the duct 31 in gaseous form.

With the various control valves set, an operative balance of the pressures and temperatures in the apparatus is created and the aforesaid products are each produced under fixed conditions of pressure and temperature. If the raw liquor is of constant composition, the said products will all be of constant composition, and continuous operation of the apparatus would continuously produce uniform results. As a matter of fact, however, raw liquors for fractionation, such as crude oil and natural gasoline from wells, are very seldom of an unvarying composition, hence fractions thereof which are separated in a refining apparatus without regulation to meet variations in their compositions are neither of constant composition or volatility. No regulation of fractionating apparatus is generally possible for the production of products which are of both constant composition and volatility, and adjustment must therefore be made for either one or the other of said qualities rather than for both. Generally, though not exclusively, in the art of refining natural liquid hydro-carbons to produce fuels, lubricants, etc., the production of a product of definite volatility is of major importance, and it is to the provision of means for insuring the continuous production of a product of constant volatility that the present invention particularly relates.

In the apparatus shown, the aforesaid operating conditions are seen to be chiefly controlled by the supply valve 10. The heating-coil valve 14, the condenser coil valve 28, the back-pressure valve 30 in the duct 31, and the return-reflux valve 34, all of said valves being constantly open to some degree. Assuming the system operatively balanced, a change in the setting of any one of the aforesaid valves will change the volatilities and compositions of all three products by caus-

ing a new pressure-temperature balance in the apparatus. It will be clear, therefore, that if, during the operation of the apparatus, the volatility of one of the liquid products should change, an appropriate adjustment of one or more of said valves is required to bring the volatility of said product back to its desired value.

For effecting the aforesaid regulation of a fractionator system, a control unit 41 is provided, said unit is arranged to have a continuous liquid sample from some part of the apparatus flow therethrough, and to measure and/or utilize the vapor pressure of the sample as a basis and/or means for appropriately setting a control valve of the apparatus to maintain a desired degree of volatility for a product. Essentially, and as is particularly brought out in the detail view of Figure 3, the control unit 41 comprises a closed evaporation cup 42 through which a sample stream is arranged to continuously flow at a constant rate while it is maintained at a constant temperature, and indicating and/or control means operated by the vapor pressure in the cup.

As particularly illustrated, the cup 42 is disposed centrally in a liquid bath 43 provided in a cylindrical heat-insulated vessel 44 having a cover 45. The bath 43 must be kept at a constant temperature while supplying any heat of vaporization needed in the cup or absorbing any heat of condensation emanating therefrom on account of a volatility change in the control sample. A further function of the bath 43 is to provide the sample to the cup at a constant temperature, and to this end the incoming sample is caused to flow through a coil 46 immersed in the bath and coaxially surrounding and discharging into the cup at its top. The cup and coil are preferably formed of heat-conducting material for facilitating any heat transfers required for maintaining a constant temperature of the sample portion in the cup.

The control sample enters the coil from a duct 47 extending into the vessel 44 and having a flow-control valve 48 therein; as illustrated, the valve 48 is of a differential diaphragm motor type and is controlled from an orifice plate 48' whereby to automatically insure a fixed flow rate of the sample through the coil 46 and cup 42. A float-controlled valve 49 in the cup 42 operates to maintain a fixed liquid level in the cup, and discharges into a duct 51 for conveying the tested sample where desired. A recording thermometer 52 has its bulb immersed in the sample stream in a well 52' provided in the coil 46 adjacent its point of discharge into the cup whereby the temperature conditions of the supplied sample are both indicated and recorded for reference in adjusting the control unit.

Means are provided for automatically controlling the temperature of the bath 43 as may be required, said means comprising an electric heater 53, a motor-driven stirrer 54, and a circuit-closing thermostat 55, all being operatively immersed in the bath. The heater 53 is arranged to derive power from a suitable source through a suitable relay switch 56 which is controlled by the thermostat 55 in such a manner that the heater is energized when, and only when, the bath temperature is below a certain predetermined value. The stirrer is constantly operated by an electric motor 57. As shown, the motor 57 is mounted on the cover 45, and the heater 53 and stirrer 54 and control thermostat 55 all depend from said cover into the bath.

It will be noted that the bath 43 may be of any suitable liquid, and would be suitably maintained at a proper depth in the vessel 44. Since it is a general practice in the art to test volatility at a temperature of 100 degrees Fahrenheit, the various control factors for the bath 43 may be so adjusted that the sample portion in the cup is automatically maintained at the aforesaid temperature; at such a temperature either water or an oil will be found satisfactory as a bath liquid for present purposes.

As is previously indicated, the vapor pressure generated and produced in the cup 42 is utilized for control purposes, and to this end the top of the cup cavity is connected through a duct 61 with an expansion, or diaphragm, chamber 62 forming part of the structure of a pressure recording meter 63. As is usual in such meters, a movable wall 64 of the chamber 62 is arranged to control the disposal of a push rod 65, which rod in turn is arranged to constantly engage the wall 64 with a constant degree of pressure and, through suitable connecting mechanism such as that shown generally in Figures 2 and 3, controls the positioning of the meter hand 66 with respect to a rotary record dial 67.

The rod 65 is also operative as a valve stem in a suitably formed fluid passage 68 which it traverses, said passage connecting ducts 69 and 71 of a fluid pressure line which in the present instance is arranged to carry air. A valve plug 72 is adjustably carried on the rod portion within said passage, and is operative in the passage to vary the permitted air flow therethrough as the rod is displaced. As particularly illustrated in Figure 3, the effective passage between the ducts 69 and 71 is arranged to be decreased as the meter indications are increased with an increased pressure in the cup whereby this valve is reverse-acting with respect to the meter.

In the described valve and meter unit 41, the same being known generally in the art as a recorder-controller, it will be noted that the passage 68 and the connections for the ducts 69 and 71 are provided in a member 73 which is threadedly and replaceably engaged with and between a tubular meter body portion 74 and a housing 75 for the expansion chamber 62. If the valve action is not desired, the member 73 may be omitted and the housing 75 may be directly secured to the extension 74; such an arrangement is shown in detail in Figure 2, it being noted that a shorter push rod 65' is therein utilized. As is particularly shown in the present installation, a shut-off valve 70 is provided in the duct 69 for complete closing to render the valve ineffective as a control, or to adjustably limit the air supply to the member 73.

For most dependable results with the evaporation cup 42, it is desirable that the vapor space provided by the cup 42 and the duct 61 and the expansion chamber 62 contain a minimum of air. Accordingly, when the unit 41 is first connected in a line to be sampled, a normally open valve 50 in the cup discharge line 51 is fully closed and the said vapor space is opened as high therein as is practically possible to permit an initial filling of the space with the liquid sample. As particularly shown, that portion of the expansion chamber housing 75 which engages the upper end of the duct 61 and mounts the expansion chamber 62 is provided with a passage extending radially from said space and normally closed by a screw plug 76, the removal of said plug permitting the aforesaid flow of liquid into the vapor space

to expel substantially all of the air therefrom. When the vapor space is filled with the sample liquid, the plug 76 is replaced, and a subsequent opening of the valve 50 permits a lowering of the liquid to its normal level in the cup as controlled by the float valve 49. It will be understood that the valve 50 is normally open to a constant degree whereby the discharge pressure in the line 51 may remain constant, said pressure being necessarily less than that in the cup 42.

As is particularly shown, the control unit 41 is applied for producing the bottom fraction to have a constant volatility through control of the valve 14 for the heating coil 12 in direct accordance with the volatility of a sample taken from a bubble tray 7. In the present arrangement, the supply valve 10 is of a differential diaphragm motor type and is controlled from an orifice plate 10' in the supply duct 9 whereby to insure a constant flow rate through the valve 10 even though the supply pressure should vary. The heating-coil valve 14 is a diaphragm valve which is arranged to be operated by air pressure for controlling the heat supply to the coil 12. The condenser coil valve 29 may be an ordinary manually set throttle valve, as is indicated. The return-reflux valve 34 is also arranged to maintain a constant flow rate therethrough, and, as shown, may comprise a differential diaphragm motor valve which is controlled by and from an orifice plate 34' in the duct 32.

Referring now to the application of the control unit to the described fractionating apparatus, a reference to Figure 1 discloses that the unit 41 is therein arranged to control the volatility of the discharged bottom product through utilizing a sample from the bottom bubble tray 7 as a control means for the heating coil 12. Accordingly, the duct 47 is connected through the tower at the bottom bubble tray 7 to receive a continuous sample of liquid from the tray, the valve duct 69 is connected with the compressed air main 22, and the valve duct 71 is connected to the valve 14. The duct 71 is provided with a constantly open branch 78 having a needle valve 79 operative therein; in this manner, regulation of the actuation of the valve 72 may be effected in adjusted proportion to the actuation of the meter control rod 65 and the vapor pressure which actuates it. The valve 72 never fully closes the passage 68 thereat, and, by reason of the back pressure created in the duct 71 by the action of the needle valve 79, any difference in the air pressure against the different sides of the valve 72 is not sufficient to appreciably effect the movement of the diaphragm 64 or the meter indications which are controlled by the rod 65.

The duct 13 has a hand-operated throttle valve 81 therein, and when the valve 14 is controlled from the unit 41 in the described manner, the valve 81 is left open. If the valve 14 is not to be automatically controlled, the valve 70 would be closed and the valve 81 would be manually and variably set to control the system as required, it being noted that the volatility of the sample in the cup would still be indicated and recorded at the meter 63.

If desired, the duct 47 which conveys the sample to the test unit 41 may have a suitable heat-exchanger 82 interposed therein for cooling the sample from the tower to a temperature slightly below that at which the sample is to be tested, whereby said test temperature may be the usual testing temperature of 100 degrees Fahrenheit. As shown, the heat-exchanger 82 is disposed between the tower and the valve 48 whereby the

cooled liquid may pass through the orifice plate 48' and the valve. Furthermore, the opening of the orifice plate 48' is preferably of such a size that the discharge pressure for the sample flowing into the cup is very much less than the tower pressure and only moderately exceeds the maximum vapor pressure for the cup and is constant, it being noted that the tower pressure is held constant through the automatic operation of the valve 30 and that the sample need comprise but a small fraction of the liquid passing through the bubble tray 7.

Since a vapor pressure will be set up in the constant-temperature sample in the cup 42 in direct accordance with the volatility of the sample portion in the cup, said pressure will change if the volatility of the sample changes. Momentary changes in the volatility of the sample will, of course, produce but a negligible effect on the cup pressure, but sustained changes in the volatility of the sample will effect the cup pressure in accordance therewith. Since the volatility of a liquid changes with the temperature thereof, the automatic maintenance of a constant sample temperature, as by the previously described operation of the control for the bath temperature, is obviously an essential feature of the device.

Means are provided for effecting a rapid change in the vapor pressure in the cup 42 as the sample varies, whereby to increase the sensitiveness of the testing unit, said means being provided in connection with the discharge of the continuous sample into the cup. As is shown in Figure 3, a portion of the top of the cup is offset upwardly of the point of connection of the pressure pipe 61 from the cup to provide a space 84 through which a nozzle assembly extends downwardly into the cup. In its present form, the nozzle assembly includes a tubular member 85 which is sealedly engaged through the top cup wall above the space 84, provides a connection for the discharge end of the coil 46, and supports nozzle tubes of the assembly.

A spray nozzle tube 86 extends axially and downwardly from the member 85, said tube receiving the inflowing sample and discharging the same through a more or less constricted orifice 87 at its free end whereby it may be operative to atomize the discharging liquid and is thereby operative as a spray jet for the sample stream. The member 85 also carries a tube 88 which encloses the jet tube 86 in coaxial relation therewith to define a free space of annular cross-section about the jet tube. The tube 88 extends somewhat into the liquid in the cup and is provided with openings 89 opposite the tube 86 and at the space 84 whereby vapor from the cup may enter the tube 88 only at its top. The jet tube 86 terminates substantially at the constant float-controlled level of the liquid in the cup.

The present nozzle assembly is essentially such that the spray discharge of the liquid sample from the jet tube 86 is arranged to force the cup liquid downwardly within the tube 88 substantially to its bottom while the discharging spray entrains the cup vapors in the tube 88. One result of this action is that the discharged sample carries an excess of vapor into the cup liquid, which vapor must escape into the vapor space of the cup by bubbling upwardly through the liquid outside of the tube 88. If the incoming sample is more or less volatile than the liquid in the cup, a corresponding increase or decrease in vapor pressure in the cup quickly results; if the sample and cup liquid have the same volatility, the pres-

sure balance is maintained. Another important result of the described nozzle action is the production of a constant circulation of the vapor in the cup whereby the sensitiveness of the testing unit to volatility changes in the sample is further increased.

The aforesaid vapor pressure in the sample cup is arranged to control the valve 14 in such a manner that a change in the volatility of the sample will cause such a change in the kettle heat as will bring the volatility of the controlling sample and the controlled product back to their desired values. Thus, should the volatility of the sample increase to produce an increased pressure in the cup 42, the valve 14 would be opened to increase the supply of heat to the kettle, and thereby decrease the volatility of the bottom product.

Since it is the volatility of a product which is to be constant, either a top or bottom product must be continuously tested for volatility during the operation of the unit 41, it being noted that a sample not a product might well have a different volatility than the product to be controlled. In the present arrangement, it is the bottom product which is to have a constant volatility, and a continuous sample is accordingly taken from a point of the duct 15 between the heat-exchanger 16 and the valve 17 and delivered to a testing unit 41' through a duct 91 which has an automatic flow control valve 92 operative therein. The valve 92 is operative as the valve 48, and the sample is at a uniform temperature as it leaves the duct 15 by reason of the action of the heat-exchanger 16.

For testing a product independently of the automatic control of my invention, only the cup and recording portions of the previously described testing and control unit 41 are utilized to provide the unit 41'. As shown, the meter portions 74 and 75 are directly connected and an appropriate rod 65' is provided in lieu of the rod 65 which is longer and carries a valve plug. The present arrangement is shown in detail in Figure 2, it being noted that a weight 94 carried by a lever of the meter mechanism is constantly operative to retain the rod against the diaphragm 64 with a uniform degree of force; the use of the weight 94 is also shown in Figure 3.

With the present control arrangement, it will be understood that if the volatility of the sample increases, the resulting increased pressure in the cup 42 is arranged to effect an opening of the valve 14 to increase the rate of heat supply from the coil 12 and so increase the heating in the kettle; in this manner the volatility of the top product will be lowered through the automatic addition thereto of intermediate fractions from the liquid in the kettle by reason of the increased kettle heat, and the volatility of the bottom product will be decreased by the removal of said intermediate fractions therefrom. The action is, of course, reversed, when the volatility of the sample decreases. The present control arrangement, both as to the action of the unit 41 and the installation thereof, is understood to be particularly sensitive and eliminates the lag which would usually result if the control should be effected through a sample of a final product.

Essentially, the invention now disclosed is understood to concern devices for automatically measuring the volatility of a mixed fraction, and to be generally usable with other types of apparatus than that particularly illustrated for bringing out the present features of invention.

From the foregoing description taken in con-

nection with the accompanying drawings, the advantages of the construction and method of operation will be readily understood by those skilled in the art to which the invention appertains, and while I have described the principle of operation, together with forms of the device which I now consider preferred embodiments thereof, I desire to have it understood that the showings are primarily illustrative, and that such changes may be made, when desired, as fall within the scope of the following claims.

I claim:

1. In apparatus for continuously testing the vapor pressure of a liquid flowing in a closed duct, a liquid heating bath, a closed evaporation cup having heat-conducting walls interposed in said duct and immersed in said bath, means automatically operative to maintain the temperature of said bath at a constant and predetermined value, and an indicating meter means actuated by and in direct accordance with the vapor pressure generated in said cup by reason of the evaporation of liquid therein.

2. In apparatus for continuously testing the vapor pressure of a liquid flowing in a closed duct, a liquid heating bath, a closed evaporation cup interposed in said duct for the continuous flow of the liquid therethrough and immersed in said bath whereby the liquid contents thereof may acquire the temperature of the bath and be partially evaporated at said temperature, means automatically operative to maintain the temperature of said bath at a constant value of the order of one hundred degrees Fahrenheit, a meter, and means directly utilizing the pressure in said cup to control the indications of said meter.

3. In apparatus for continuously testing the volatility of a liquid flowing in a closed duct, a closed evaporation cup interposed in said duct, means automatically operative to maintain a fixed liquid level in said cup, means to maintain the temperature of the cup contents at a fixed value, means to measure the vapor pressure in said cup, and means automatically operative to

introduce vapor from the cup into the liquid as it is discharged into the cup.

4. In apparatus for continuously testing the volatility of a liquid flowing in a closed duct, a closed evaporation cup interposed in said duct, means automatically operative to maintain a fixed liquid level in said cup, means to maintain the temperature of the cup contents at a fixed value, means to measure the vapor pressure in said cup, a jet nozzle operative to deliver the liquid into the cup, and means utilizing the jet from said nozzle to circulate the vapor in the cup.

5. In apparatus for continuously testing the volatility of a liquid flowing in a closed duct, a closed evaporation cup interposed in said duct, means automatically operative to maintain a fixed temperature in and at said cup, means to measure the vapor pressure in said cup, a jet nozzle operative to deliver the liquid from the duct to the liquid in the cup, and means whereby the jet is arranged to entrain vapor from the cup as it is discharged into the liquid in the cup.

6. In apparatus for continuously testing the volatility of a liquid flowing in a closed duct, a closed evaporation cup interposed in said duct, means automatically operative to maintain a fixed temperature in and at said cup, means whereby the liquid is arranged to enter the cup at said fixed temperature, an expansion chamber connected with a vapor space of the cup, a pressure meter, and means utilizing a movable wall of said chamber to actuate said meter.

7. In apparatus for continuously testing the vapor pressure of a liquid flowing in a closed duct, a closed evaporation cup interposed in said duct and arranged for the partial evaporation of the liquid in the cup, temperature control means for the cup contents, means automatically operative by and in accordance with a change in the temperature of the cup contents to actuate said temperature control means for restoring the temperature of the cup contents to its initial value, and means to measure the vapor pressure in the cup.

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