50-203 CROSS REFERENCE EXAMINE

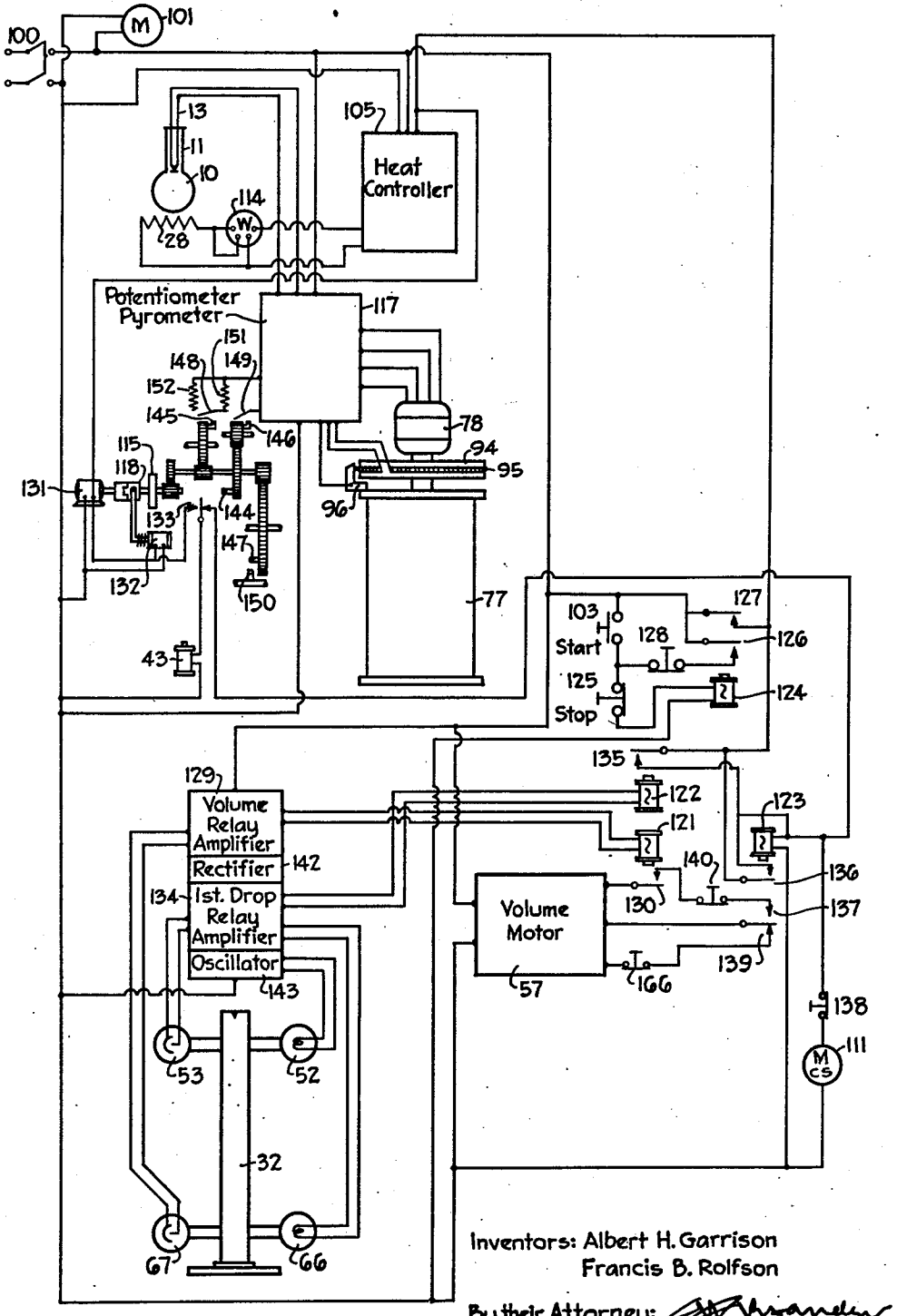
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Dec. 4, 1951 A. H. GARRISON ET AL 2,577,615

AUTOMATIC DISTILLATION APPARATUS

Filed Sept. 8, 1947 4 Sheets-Sheet 1



Inventors: Albert H. Garrison
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By their Attorney:

Fig. 1

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AUTOMATIC DISTILLATION APPARATUS

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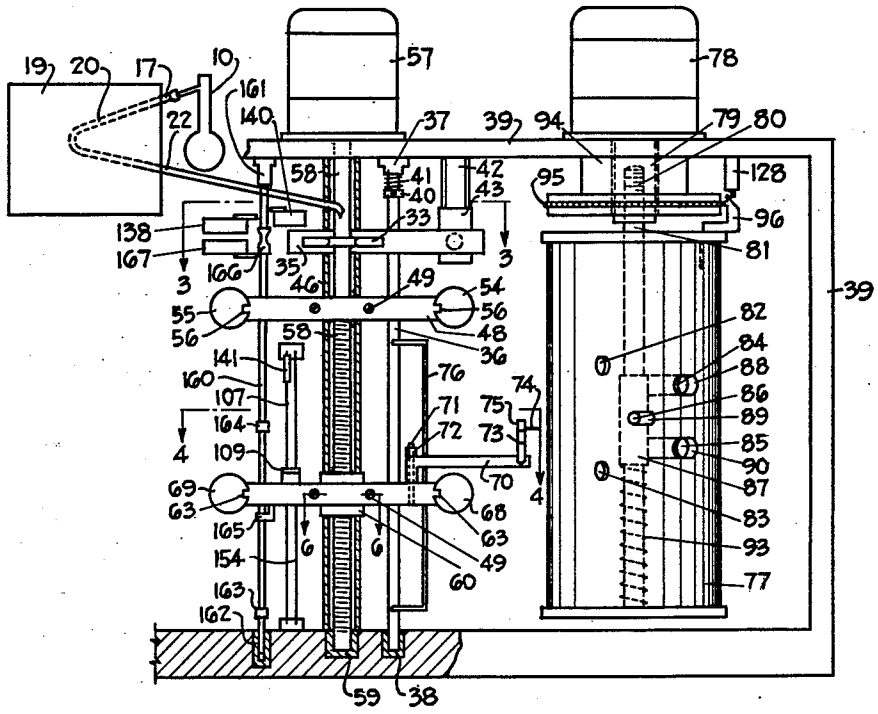


Fig. 2

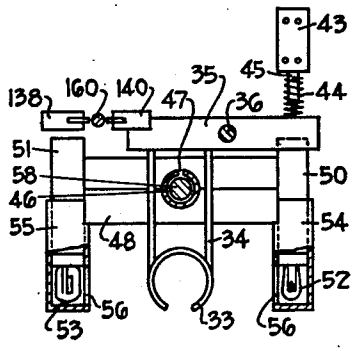


Fig. 3

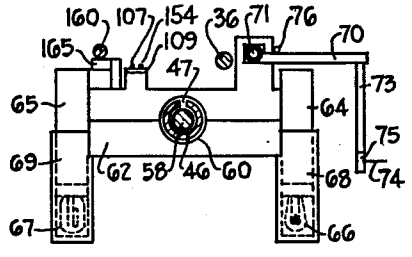


Fig. 4

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Dec. 4, 1951 A. H. GARRISON ET AL 2,577,615

AUTOMATIC DISTILLATION APPARATUS

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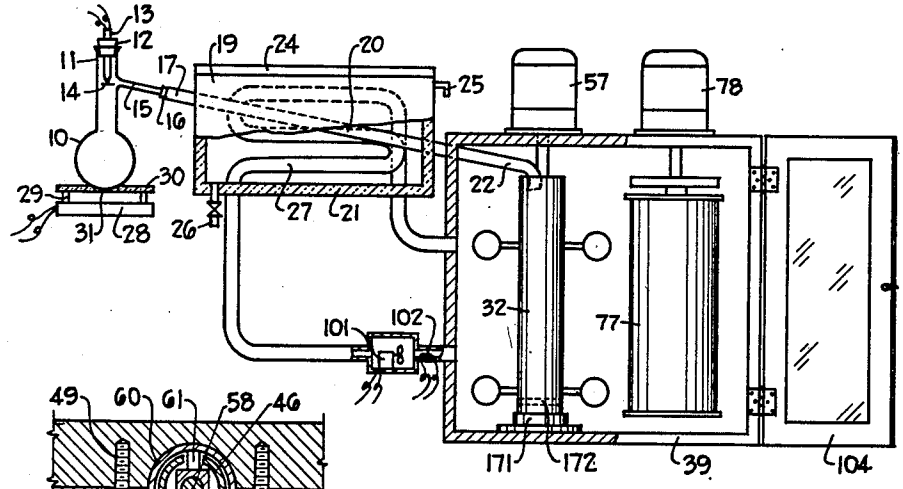


Fig. 5

Fig. 6

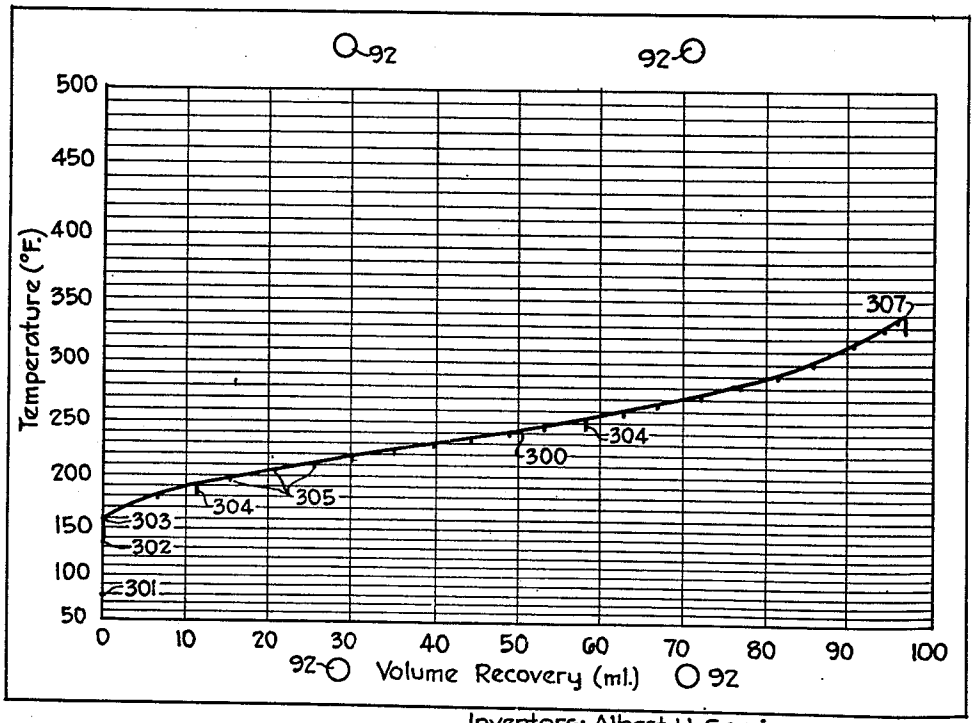


Fig. 7

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AUTOMATIC DISTILLATION APPARATUS

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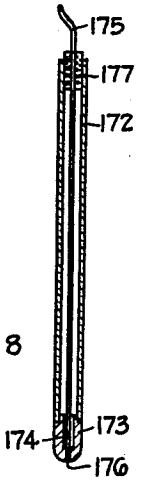


Fig. 8

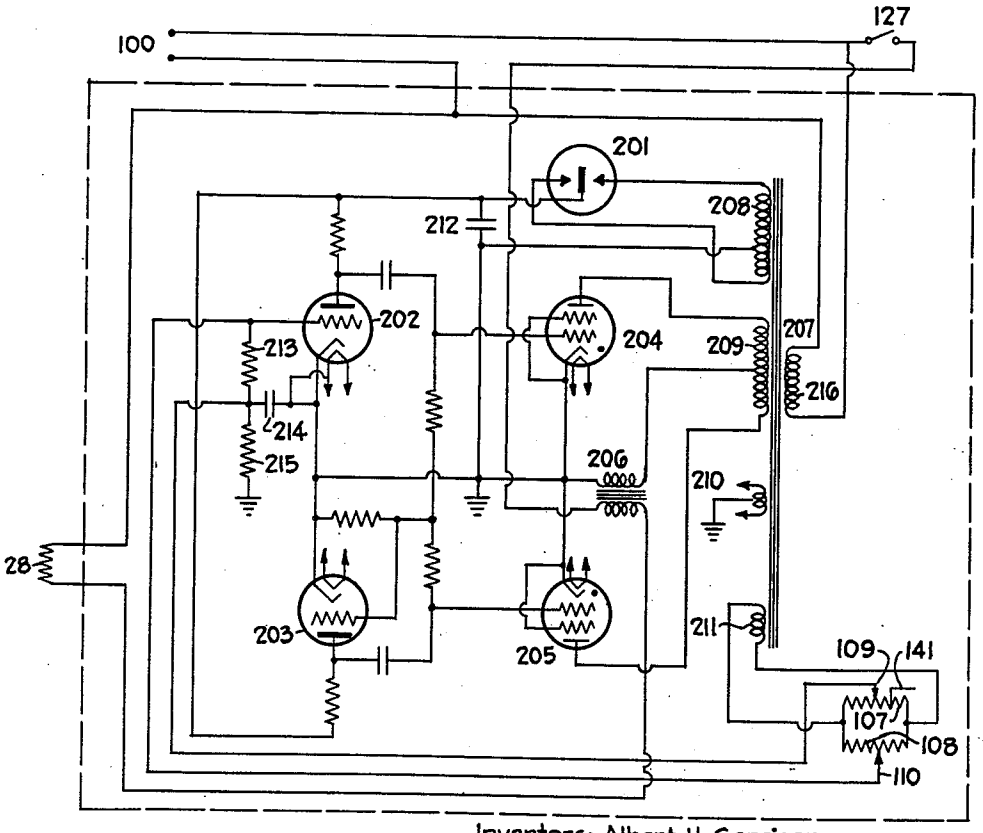


Fig. 9

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Patented Dec. 4, 1951 2,577,615

UNITED STATES PATENT OFFICE

2,577,615

AUTOMATIC DISTILLATION APPARATUS

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Application September 8, 1947, Serial No. 772,628
14 Claims. (Cl. 202—206)

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This invention relates to apparatus for automatically distilling liquids under controlled conditions and for automatically recording the volumetric and temperature distillation data.

One of the many control tests used by the chemical industry to maintain a uniform quality in products is a distillation test in which a small quantity of the liquid to be tested is distilled, the distillation temperature and the volume of recovered distillate being noted and recorded at regular time intervals. In the petroleum industry, where it is necessary to run hundreds of distillation tests daily, a standard test method, including standard apparatus to be used, has been set up, approved, and adopted by the American Petroleum Institute and the American Society for Testing Materials (ASTM).

In order to utilize fully the time used by operators in running a distillation test on a petroleum product, it is a general practice for one operator to run a series of distillations simultaneously. Such a practice may naturally lead to errors in the distillation data, as it is not always possible to control the distillations at the same rate. When the observer has to take thermometer and volumetric readings on more than one distillation apparatus at the same time, it may become necessary for him to forego the taking of one reading, thus making the data incomplete. When single distillations are run, the interval between readings is usually sufficient to allow the operator to obtain satisfactory results. However, this practice also often results in incomplete data when the operator inadvertently fails to take a reading at the prescribed time. Only with the utmost care and attention and strict adherence to details is the operator able to obtain accurate and satisfactory results.

It is therefore the primary object of this invention to eliminate the source of all personal errors by providing an apparatus capable of automatically distilling, according to standard specifications, various fluids such as, for example, gasoline, naphtha, kerosene, stove oil, and similar petroleum products.

Another object of this invention is to provide a distillation apparatus adapted to maintain a desired distillation rate by controlling the heat supply.

It is a further object of the present invention to provide an automatic distillation apparatus for automatically recording volumetric and temperature distillation data.

Another object of the present invention is to provide an automatic distillation apparatus having a recording pyrometer adapted to draw a temperature-distillate volume curve from the data supplied by the apparatus.

A still further object of the present invention is to provide an apparatus adapted to automatically reset itself to zero or to its original position after completing an automatic distillation of a liquid.

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These and other objects of this invention will be understood from the following detailed description of the preferred embodiment of the invention and from the accompanying drawings, wherein:

Figure 1 is a schematic diagram of the electrical circuit of the automatic distillation apparatus of the present invention.

Figure 2 is a diagrammatic front view of the recording apparatus of the present invention.

Figure 3 is a cross sectional view taken along the line 3—3 of Figure 2.

Figure 4 is a cross sectional view taken along the line 4—4 of Figure 2.

Figure 5 is a diagrammatic view partially in cross section showing the cooling apparatus of the present invention.

Figure 6 is a cross sectional view taken along the line 6—6 of Figure 2.

Figure 7 is an illustrative example of a chart obtained by means of the recording device of the present invention.

Figure 8 is a detailed view of one embodiment of a thermocouple used in the present invention.

Figure 9 is a diagram of the circuit of the heat controller shown in Figure 1.

Although the automatic distillation apparatus of the present invention may be used for the distillation of many types of fluids, it will be described, for simplicity, in conjunction with the standard test for the distillation of petroleum products which has an ASTM designation of D86. The apparatus normally used in this distillation procedure comprises a standard 100 ml. Engler distillation flask, a 100 ml. graduate to collect distillate, a condenser tube between the flask and the graduate, an ice water bath around the condenser tube, an electric heating unit under the flask, a thermometer in the neck of the flask and means for regulating the temperature of the receiving graduate.

The ASTM procedure for the D86 test requires that

(*a*) the heater be set to deliver heat at a uniform rate and be so regulated that the first drop of distillate falls from the condenser between 5 and 10 minutes after starting the heater cold,

(*b*) the distillation thermometer be read two minutes after heat is applied,

(*c*) the receiving graduate be moved after the first drop so that the nozzle of the condenser tube touches the side of the graduate,

(*d*) the heat be regulated so that the distillation proceeds at a uniform rate of between 4 and 5 ml. per minute,

(*e*) the temperature be observed and recorded for every 10 ml. of distillate collected or the amount of distillate be observed and recorded for every 10° C. rise in temperature,

(*f*) no adjustment to the heat be made when the liquid residue in the flask is approximately 5 ml.,

(*g*) the end point maximum temperature be observed and recorded, and

(*h*) the temperature outside the collecting graduate be maintained between 55° and 65° F.

Briefly, the automatic distillation apparatus of the present invention includes a distillation flask, condenser tube, ice bath, electric heater and collecting cylinder which are built to the standard specifications for this test. In addition to the above equipment, the present automatic distillation apparatus comprises a "first drop" photocell means which detects and records the initial boiling point, means for moving the collecting cylinder against the discharge end of the condenser tube after the first drop, a recording pyrometer adapted to draw a curve of temperature v. volume of distillate, a "volume follow-up" photocell means movably mounted and adapted to follow the rising meniscus of the distillate in the collecting cylinder, a distillation rate timing motor, heat control means for adjusting the electic heater so as to maintain the prescribed distillation rate, means for marking time intervals on the distillation curve drawn on the pyrometer chart, a substantially airtight compartment enclosing the collecting cylinder and photocell units, and cooling means for maintaining the temperature of the collecting cylinder at a predetermined, substantially constant value.

Referring to Fig. 5 of the drawing, it will be seen that the apparatus includes a standard 100 ml. Engler distillation flask 10, and a thermocouple unit 13 removably seated in the neck 11 of the flask 10 by means of a stopper 12 having an axial bore therethrough. The thermocouple unit 13 depends in the middle of the neck so that the hot junction 14 of said unit is substantially on a level with the vapor outlet tube 15 at its junction with the neck of the flask 10.

The vapor outlet tube 15 is fitted with a stopper 16, which seals it in the receiving end 17 of the condenser tube 20. The condenser unit 19 comprises a condenser tube 20 passing through a heat insulated condenser jacket 21 with the receiving end 17 and the discharge end 22 of said tube extending in a fluid-tight manner for short prescribed distances through the walls of said jacket 21. The discharge end 22 of said tube 20 is curved downward and slightly backward to insure contact with the wall of the receiving graduate or cylinder 32. The fluid-tight condenser jacket 21 is equipped with a cover 24, overflow tube 25 and a valved drain port 26. The size, bore and slope of the condenser tube 20 are those prescribed by the ASTM. The condenser tube 20 may be curved back on itself to bring the discharge end out of the same side of the jacket 21 as the inlet 17, as shown in Fig. 2. Also located within the condenser jacket 21 is an air cooling conduit 27 whose ends pass through the walls of said jacket in a fluid-tight manner.

Any suitable type of an electric heater 28 may be used to heat the flask 10 and the liquid contained therein. The heater should be of a type having low heat lag so that it possesses a rapid response to changes in the automatic controls. The heater may be positioned in any suitable manner, as by spacing rods 29, a short distance below an asbestos board 30. This board is from ⅛ to ¼ inch thick and has a circular hole 31 in the center thereof over which the distillation flask 10 is placed.

Positioned under the discharge end 22 of the condenser tube 20 is a collecting cylinder or graduate 32 in which the liquid distillate is received and stored during the distillation run. The collecting or receiving cylinder 32 is made of light-transmitting material having a uniform bore. The cylinder must be capable of holding the 100 ml. of liquid used in the test and may be graduated. In order to observe the first drop of distillate falling into the cylinder 32, said cylinder is securely mounted in a clamp 33 (Figs. 2 and 3) which centers the cylinder 32 under discharge end 22 of the condenser tube.

After the first drop of distillate has been observed dropping from the condenser tube, a substantially level, undisturbed, slowly-rising meniscus of the distillate is desired in order to obtain accurate observations of the volume of distillate in the cylinder. In order to secure the required meniscus, the collecting cylinder 32 must be moved against the tip of the discharge end 22 of the condenser tube 20 so that the liquid distillate runs down the inside surface of cylinder 32. The extending arms 34 of the ring clamp 33 are securely attached to one side of a cross bar 35 which pivots on a vertical rod 36 mounted pivotally between upper 37 and lower 38 bearings attached to the top and bottom, respectively, of the air-tight compartment 39 enclosing the collecting cylinder 32. An adjustable collar 40 (Fig. 2) is located near one end of the rod 36, having a torsion spring 41 attached to it and to an adjacent bearing 37, or other support. The spring 41 supplies a slight amount of torque to rod 36, tending to rotate it so as to move the attached cross bar 35 and ring clamp 33, whereby the collecting cylinder 32 is drawn and held against the tip of the discharge end 22 of the condenser tube 20.

Attached to the inside of the compartment 39 in a rigid manner, as by supports 42, is a solenoid 43 having a compression spring 44 (Fig. 3) attached to the projecting portion of the solenoid plunger 45. The spring 44 normally exerts sufficient force against the cross bar 35, which it contacts, to prevent said bar 35 and attached ring clamp 33 from rotating. Thus in the normal initial position the cylinder 32 is held by the clamp 33 so that the cylinder walls do not touch the discharge end 22 of the condenser tube 20. When the solenoid 43 is energized, its plunger 45 and the spring 44 attached thereto are drawn away from the cross bar 35 a short distance allowing the torsion spring 41 to rotate the rod 36, cross bar 35 and attached ring clamp 33 thus moving the cylinder 32 so that its inside wall touches the tip of the condenser tube 20. The purpose of the spring means 44 between the cross bar 35 and the plunger 45 is to cushion the movement of said bar 45 thus preventing the glass cylinder 32 from making any sharp contact with the condenser tube 20 which might break it.

Securely mounted between the top and bottom of the compartment 39 is a tubular member 46 having a longitudinal guide slot 47 cut in the wall thereof (Fig. 3). The tube 46 serves as support means for a "first drop" photocell unit which comprises a split frame 48 rigidly clamped in a fixed position on the tube 46 by means of screws 49 (Fig. 6), forwardly extending end members 50 and 51 supporting respectively a light source 52 and a photocell 53 which are housed in removable cylindrical housings 54 and 55 having slits 56 therein. The end members 50 and 51 extend forward in such a manner that the light source 52 and photocell 53 held by them are positioned on either side of the cylinder 32 substantially opposite the axial line thereof. The shields or housings 54 and 55 covering the light

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source 52 and photocell 53 are turned towards each other so that a light beam from the source 52 continuously falls on the photocell 53 through the slits 56. As the first drop of distillate falls into the cylinder 32 and momentarily crosses the light beam, its shadow registers on the photocell 53, which is electrically connected to the solenoid 43 so as to energize said solenoid and to cause the collecting cylinder 32 to be moved over against the condenser tube 20.

Rigidly secured to the top of the compartment 39 is a reversible motor 57, preferably of the induction type, to whose shaft is attached a lead screw 58 which extends through the top of the compartment 39 and down through the tubular member 46 therein. The lower end of said lead screw 58 rotates in a bearing 59. Slidably mounted on the tubular member 46 is a collar 60 having attached to the inner wall thereof or formed integrally therewith, a radially extending member 61 having a threaded axial bore therethrough, said bore being in screw-threaded engagement with the lead screw 58 as shown in Fig. 6. Rotation of the lead screw 58 causes the collar to rise on the screw as rotation of the collar is prevented by the radial member which rides in the longitudinal guide slot 47 in the wall of the tube 46 in which the lead screw 58 is encased.

The split frame 62 of the lower or "meniscus following" photocell unit is rigidly clamped in a fixed position on the collar 60 by means of screws 49. This meniscus following or follow-up photocell unit is similar to the "first drop" photocell unit attached to frame 48 and has forwardly extending end members 64 and 65 attached to the split frame 62 for holding a light source 66 and a photocell 67 respectively, and removable cylindrical housings or shields 68 and 69 having slits 63 therein and covering said light 66 and photocell 67 respectively. The end members 64 and 65 extend forward sufficiently so that the light source 66 and photocell 67 held by them are positioned on either side of the cylinder 32 substantially opposite the axial line thereof. The shields 68 and 69 are turned towards each other so that a light beam from the source 66 continuously falls on the photocell 67 through the slits 63. As a column of liquid rises in collecting cylinder 32 and intercepts the beam of light from the source 66 falling on the photocell 67, the intensity of the light beam falling on the photocell is changed. This change of the light beam registers on the photocell 67, which is electrically connected to the motor 57 and energizes said motor causing the attached lead screw 58 to raise the follow-up photocell unit.

Pivotally carried on the rear portion of the split frame 62 (Fig. 4) of the follow-up photocell unit is a laterally extending arm 70 mounted on a pivot 71. A torsion-spring loaded collar unit 72 (Fig. 2) attached to said arm 70 and pivot pin 71 tends to cause said arm 70 to rotate in a counter-clockwise direction. Attached to said arm 70 is a pen arm 73 at the end of which is a stylus, such as a pen 74 and ink reservoir 75. Fixedly secured to the vertical, rotatable rod 36, and in parallel spaced relationship therewith, is a pen controlling bar 76 which is in contact with the horizontal pen arm 70 at all times. Before any liquid is received in the collecting cylinder 32, the pen 74 is held away from the recording drum 77 by the pen-controlling bar 76 pressing on the pen arm 70 against the action of the spring loaded collar 72. When the first drop of liquid falls past the "first drop" photocell 53, the

solenoid 43 is energized, the plunger 45 is withdrawn, and the vertical rod 36 together with the attached pen lifting bar 76 are rotated by spring 41 a slight distance in a counter-clockwise direction, which allows the pen arm 70 to be rotated in the same direction by the spring loaded collar 72, thus causing the pen 74 to contact the recording drum 77.

As shown in Fig. 5, the recording drum 77, collecting cylinder 32, discharge end 22 of the condenser tube 20 and the "first drop" and "volume follow-up" photocell units are enclosed in an air-tight compartment 39. The compartment 39 is equipped with a door 104 through which the collecting cylinder 32 and recording charts may be removed. The compartment 39 is preferably located adjacent the condenser unit 19 containing the air cooling conduit 27, both ends of which are in communication with the inside of the compartment 39. A fan or blower 101 circulates the air from the compartment 39, through the cooling conduit 27 and back to the compartment. A thermostat 102 positioned in the cooling coil 27 adjacent the fan controls the operation of said fan to maintain the receiving compartment 39 at a desired temperature, for example, between 55° and 65° F. as prescribed by the ASTM.

Securely mounted atop the compartment 39 (Fig. 2) is a two phase induction motor 78 having a shaft 79 extending through a hole in the top of the compartment 39 into said compartment. The shaft 79 is drilled and tapped for threaded connection with the threaded end 80 of axial rod 81, said rod being rigidly mounted in and extending through the upper end of the drum 77. A chart 91 (Fig. 7) is secured in any suitable manner to the vertical surface of the depending drum 77. For example, the desired alignment of the chart 91 on the drum 77 may be secured by fitting punched holes 92 in said chart over four knobs 82, 83, 84 and 85 protruding outwardly from the surface of the drum 77. Two of the knobs 82 and 83 are rigidly secured to the surface of the drum. The other two knobs 84 and 85, along with a control knob 86, are movable within slots 88, 89 and 90 in the walls of the drum, being attached to the ends of three radially extending fingers of a spring loaded rotatable collar 87 mounted on the axial rod 81 of the drum 77. Thus when a chart is positioned on the drum, the holes 92 on one side of the chart are fitted over the knobs 82 and 83, the chart is fitted around the drum and the knob 86 is moved in its slot 89 thus causing the collar 87 to rotate moving knobs 84 and 85 in their slots 88 and 90 respectively until they are positioned so that the other two holes 92 in the chart 91 may be fitted over them. Release of knob 86 allows spring 93, attached to the collar 87, to rotate said collar 87 and knobs 84, 85 and 86 in a clockwise manner, said knobs 84 and 85 furnishing adequate tension to stretch the chart tightly on the drum. The holes at either end of the chart are differently spaced so as to preclude the inadvertent placing of the chart on the drum in an incorrect manner, i. e. upside down.

Located inside the compartment 39 and secured to the top thereof is a cylindrical block 94 having an axial bore therethrough adapted to allow the rotation of the shaft 79 of the motor 78 therein. Secured to the outer periphery of the block is a manganin slide wire 95 wound on any suitable mandrel, such as a length of enameled copper wire, said slide wire being electrically connected to the pyrometer circuit of the present apparatus

and being part of the slide wire potentiometer arrangement therein. The sliding contact 96 for this slide wire 95 is fixedly mounted on the rotatable drum 77. Positioned adjacent the rotatable drum 77 and secured to the inside top of compartment 39 is an end point switch 128 which is opened by the sliding contact 96 a short time after the end point or maximum temperature has been reached and the drum 77 and attached contact 96 reverse and start rotating in the opposite direction, as will be described hereinbelow. This switch 128 is normally closed and is not actuated by the passing contact 96 when the drum 77 is rotating in the original direction with rising temperature as the moving contact 96 bends the flexible contact of switch 128 towards its closed position as it passes it the first time.

A switch-actuating rod 160 (Fig. 2) is mounted for vertical sliding movement between bearings 161 and 162. Carried by this rod are two collars 163 and 164 which are contacted by an arm 165 attached to and projecting from the split frame 62 as it is raised and lowered. When 95% of the liquid has been distilled and the frame 62 is correspondingly raised, arm 165 contacts the upper collar 164 forcing collar 164, rod 160 and a switch-actuating collar 166 upward. Switch actuating collar 166 passes a zero limit switch 167 and opens the switch 138 which stops a timing motor 111 when 95% of the fluid has left the distillation flask 10. If the volume motor 57 accidentally continues to raise the split frame 62 above a predetermined limit of the instrument, the switch actuating collar 164 will be forced upward until it contacts an upper limit switch 140 which will cause the volume motor to stop. After a distillation has been completed, the follow-up photocell 67 is returned to its original or zero position, during which operation the arm 165 contacts the collar 163 to force the switch-actuating rod 160 and collar 166 downward, opening the normally closed zero limit switch 167 and thus shutting off the elevator motor 57.

A special collecting cylinder or graduate 32, as shown in Fig. 5, is used with the system of the present invention, as difficulties are usually encountered at the zero level of ordinary glass graduates. The errors encountered in using ordinary glass graduates may be due to various reasons: first, the walls of the graduate are usually thicker at the zero (ml.) mark, which changes the intensification of the light at this point; second, the inside bottom of the graduate is usually slightly concave causing light beams directed thereon to be reflected at various angles therefrom, which affects the rising photocell in an adverse manner; and lastly, the thickness of graduate bases generally varies so as to place the zero marks of the graduates at varying heights above the bottom of the base or surface upon which the graduates rest, thus necessitating the recalibration of the instrument to a new zero point every time a new graduate is used. In order to preclude the above sources of error a special graduate 32 may be made from glass tubing having uniform bore and wall thickness. A flat glass disc 172 may be sealed inside the tubing which may be then mounted or sealed in a metal base 171 so that the top of the disc 172 is above the top of said base 171. It is important that the distance between the top of the disc 172 and the bottom of the metal base 171 be the same on all graduates used with the same apparatus in order to obviate any resetting of the lower photocell 67. To eliminate or reduce possible reflections from the disc a thin Pyrex glass disc may be used having rough-ground surfaces with the bottom surface painted black. To eliminate optical distortion the polished edges of the disc 172 may be carefully ring-sealed to the tube. Since a direct pen connection 74 is used, it is apparent that the height of the graduated portion of the receiving cylinder 32 must be the same as the length of the volume axis of the distillation chart. To obtain accurate volume recordings on the chart it is preferable to use precision bore tubing in fabricating the graduate. According to ASTM specifications, 100 ml. of distillate must be contained in 7 to 8 inches of the tubing. The chart is then drawn using the height of 100 ml. of liquid in the cylinder as the length of the volume axis of the chart.

In starting the operation of the above-described apparatus, the main power switch 100 (as shown in Fig. 1) is closed, which causes the blower or fan 101 (Fig. 5) to circulate air from the air cooling coil 27 through the receiving compartment 39. If the volume "follow-up" photocell 67 is at any position other than its lowermost or zero position on the lead screw 58, the switches of the system will remain in the necessary positions from the previous distillation run, so that the motor 57 will automatically start and return the photocell and its split frame 62 and light source 66 to the zero position, after which the motor 57 will be shut off. A sample of fluid is placed in the distillation flask 10 whose outlet tube 15 is connected to the upper end of the condenser tube 20 by means of a rubber or cork stopper 16. The thermocouple unit 13 and attached stopper 12 is secured in a fluid-tight manner in the neck 11 of the flask 10. The collecting cylinder 32 is placed in its holding clamp 33, a chart is attached to the drum 77 and the door 104 of the receiving compartment is closed.

The distillation "start" switch 103 (Fig. 1) is then momentarily closed, which energizes coil 124 through stop switch 125, causing the normally open contacts 126 and 127 of coil 124 to close. After the switch 103 is released, the coil 124 remains energized through contact 126, end point switch 128 and "stop" switch 125. When the contact 127 is closed, current is furnished through the heat controller 105 to the heater 28, a time marking motor 131 and a magnetic clutch solenoid 132 for said motor.

Since the operator knows the type of liquid (i. e. its approximate initial boiling point) being tested or distilled, he also experimentally knows the amount of heat needed to cause the first drop of distillate to fall into the collecting cylinder 32 in 5 to 10 minutes after the heater 28 is turned on. Therefore the amount of heat being supplied to the heater 28 is controlled by adjusting the timing potentiometer 108 of the heat controller 105 until a suitable instrument, such as a wattmeter 114, reads the proper value as determined by previous experience.

As shown in Fig. 9, the heat controller 105 comprises a "volume" potentiometer 107 whose sliding contact 109 moves with the rise of the meniscus level in the receiving cylinder 32, contact 109 being directly attached in an electrically insulated manner to split frame 62 carrying photocell 67 and recording pen 74, a "timing" potentiometer 108 whose sliding contact 110 is driven at a constant rate being adjustably geared to a synchronous motor 111 (Fig. 1); and an electronic circuit which is responsive to the

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above-mentioned potentiometers and regulates the amount of current going to the heater 28 so as to produce the desired distillation rate. The contact 110 may be connected to the motor 111 by a friction clutch (not shown) which will permit manual setting of potentiometer contact 110 so as to adjust the initial heat at the start of the distillation.

The potentiometers 107 and 108 consist of resistances connected in parallel to an alternating voltage furnished by the secondary 211 of a transformer 207, whose primary 216 is energized through switch 100. Thus it can be seen that the phase and magnitude of the voltage difference between the two sliding contacts 109 and 110 will depend on the relative position of the two contacts on their respective resistances, the value of said voltage difference being zero, or nearly so, whenever the sliding contacts are in the same relative positions. During a distillation, the timing contact 110 is driven by a synchronous motor 111, shown in Fig. 1, adjusted so that it traverses the entire resistance 108 in the time (20 to 25 minutes) required for the distillation (100 ml. at 4 to 5 ml. per min.). When the sliding contact 109 of the volume potentiometer 107 is advancing at the desired rate, i. e. at the same rate as that of the timing potentiometer 108, the two sliding contacts 109 and 110 have the same relative position and no voltage appears between them. At such a time a constant amount of current is being supplied to the heater 28 which in turn supplies a constant amount of heat to the distillation flask 10. However, if the distillation rate is either too fast or too slow, a voltage difference will appear between the contacts, the phase and magnitude of said voltage or error signal being an indication of the required correction to the volume potentiometer.

This voltage or error signal will appear across a resistor 213 where a quadrature phase component is superimposed upon it by a capacitor 214 and a resistor 215 connected to one of the heater circuit terminals. The sum of these two voltages is a voltage whose phase (with respect to that of the line voltage) shifts smoothly as the phase of the error signal changes abruptly, this being the condition required for phase shift control of the two thyratron tubes 204 and 205. The secondary winding 209 of the transformer 207 supplies the anode voltage to the thyratrons 204 and 205 which, acting as grid controlled rectifiers, furnish controlled direct current to the saturable reactor 206 which in turn has sufficient capacity to control the current to the heater 28. A rectifier tube 201 furnishes plate voltage to the two tubes 202 and 203 which act as an amplifier and a phase inverter, respectively, to provide push pull voltage of sufficient magnitude to control the thyratrons 204 and 205. A secondary winding 208 of transformer 207, along with rectifier tube 201 and condenser 212 constitute the direct current plate power supply to tubes 202 and 203. Heater current is supplied to all tubes by secondary winding 210 while secondary winding 211 furnishes alternating voltage for the timing potentiometer 108 and the volume potentiometer 107.

Thus, from the above description it is evident that the two potentiometer contacts 109 and 110 must vary their relative positions to produce the error signal which controls the output to the heater 28. For instance, if more heat is required to maintain the prescribed distillation rate, the contact 110 of the timing potentiometer 108 advances slightly with respect to the contact 109 of the volume potentiometer 107. It will then maintain this lead so long as the heat demand remains the same. If, on the other hand, the distillation rate is too high, the volume potentiometer 107 advances in its relative position to the timing potentiometer 108, decreasing the differential voltage which in turn lowers the heat until the distillation rate is again correct.

As shown in Figs. 2 and 4, the sliding contact 109 of the volume potentiometer 107 is mounted at the rear of the split frame 62 of the lower photocell 67 unit. The potentiometer 107 is mounted in a vertical position adjacent thereto so that it is contacted by the sliding contact 109 at all times. To eliminate the use of a cumbersome lead attached to the sliding contact 109, a trolley wire 154 is mounted parallel to the potentiometer 107, and, being in contact with sliding contact 109 at all times, serves as a current lead thereto.

Since the pen 74 and the contact 109 of the potentiometer 107 are both secured to the split frame 62, the length of the slide wire of the volume potentiometer 107 from the zero position of the contact to a 95% sleeve 141 (described hereinbelow) is exactly the same as the height of 95 ml. of distillate in the collecting cylinder 32. As the distillation must be carried out at a predetermined rate, for example, 4 to 5 ml. per minute, it follows that it takes a predetermined time, for example, 21.1 minutes for the first 95 ml. of distillate to be collected. Therefore the timing potentiometer 108 is preferably designed so that its contact 110, driven by the motor 111, traverses its length in the same amount of time. The actual length and resistance of the timing potentiometer 108 is immaterial as long as its time of travel is synchronized with that of the volume potentiometer.

When heat is applied to the distillation flask 10, the temperature of the liquid in the flask starts to build up, but as the temperature of the vapor in the flask is not required at this point, it is not recorded on the drum 77 by the pen 74, said pen 74 being held off the drum 77 by the pen-controlling bar 76. Two minutes after the heat is applied, cam 144 (Fig. 1) driven by the time marking motor 131 operates a switch 133 which momentarily furnishes current to the solenoid 43 whose plunger 45 is drawn inward allowing rod 36 and attached pen-controlling bar 76 to rotate so that the pen 74 momentarily contacts the chart. The pen at this time places a mark 301 on the chart 91 (Fig. 7) indicating the temperature in the flask two minutes after the heat has been applied. Immediately thereafter the pen is withdrawn from the chart to its original position.

Continued heating of the flask 10 causes the liquid therein to vaporize with the vapors flowing out of the flask into the condenser tube 20 where they are condensed, the first drop of condensate emerging from said tube 20 and falling into the collecting cylinder 32 within 5 to 10 minutes after the heat was applied. As the first drop of distillate falls past the "first drop" photocell 53 and partially intercepts the beam from the light source 52 which is focused on photocell 53, the sensitive first drop amplifier 134, which is used between photocell 53 and relay 122, amplifies and lengthens the pulse from the photocell 53, causing relay 122 to close its contact long enough to allow relay 123 to close and lock its

contact 136 which energizes the solenoid 43. At the same time as relay 123 closes, the "up" contact switch 137 of the volume motor 57 is closed so that the motor will be in condition to cause the volume follow-up photocell 67 unit to rise whenever the contact 130 of relay 121 is closed. The first drop amplifier used between the photocell 53 and relay 122 should preferably be quite sensitive because the change in light caused by the falling drop is very small and of short duration. The relatively high sensitivity of the photocell and amplifier circuit makes the use of alternating current of 60 cycles frequency on the light source 52 undesirable because of the 4 or 5% modulation of the light beam due to said frequency, might render the system inoperative. Therefore a radio frequency oscillator 143 is provided to furnish high frequency current for this light source 52. A rectifier 142, of any suitable type, is connected into the system for converting the alternating current to direct current. The rectifier 142 supplies the necessary direct current needed by the electronic equipment used in the volume relay amplifier 129, the first drop amplifier 134 and the oscillator 143, and also supplies the direct current needed by the photocells 53 and 67.

The moment the solenoid 43 is energized, the spring-loaded rod 36 slightly rotates the attached pen-controlling bar 76, allowing the pen 74 to contact the chart on the drum 77. At the same time, the cross bar 35, mounted on the rotatable rod 36, is rotated slightly, moving the attached cylinder clamp 33 so that the collecting cylinder 32 is moved against the discharge tip 22 of the condenser tube 20. With the collecting cylinder in this position, the condensate runs down the inner surface of the cylinder so that the rising meniscus of the distillate is substantially undisturbed. As previously mentioned, the sliding contact 109 of the volume potentiometer 107 in the heat controller 105 is mounted on the split frame carrying the volume follow-up photocell 67 and the recording pen 74. As the distillation continues, a column of distillate collects in the bottom of the collecting cylinder 32 and intercepts the light beam between light source 66 and the volume follow-up photocell 67. In a preferred embodiment, the operation of the volume photocell unit 67 is based on the fact that the liquid-filled glass cylinder 32 focuses the light beam from the source 66 so that the light falling on the photocell 67 is intensified. Thus, the photocell receives a relatively intense light from the space below the meniscus; at the meniscus the intensity is greatly reduced, while above the meniscus an intermediate intensity is obtained. It is, however, obvious that by a suitable adjustment of the photocell circuit, the present system may be adapted to be operated under conditions where accumulation of the liquid in the cylinder would decrease rather than increase the amount of light reaching the photocell, as, for example, in the case of liquids giving an opaque distillate.

As the intensity of the light beam falling on the photocell 67 is varied by the rising liquid column when its meniscus rises above the light beam, the increased current output of the photocell is amplified by the volume relay amplifier 129 and causes contact 130 of relay 121 to close and the volume motor 57 to start driving lead screw 58 so that the volume follow-up photocell 67 is raised a slight amount until it is level with the meniscus which then shadows the photocell 67. This immediately causes relay 121 to open, stopping motor 57. As the meniscus of the rising liquid column again rises above the light beam to the photocell 67, the above-described operation is repeated until the distillation is complete. The sensitivity of the volume photocell-amplifier system is such that the instrument follows changes in level of the order of a few thousandths of an inch, whereby a smooth curve 300 is drawn on the chart, as shown in Fig. 7, thus recording the temperature of the distillation flask 10 and the volume of distillate collected. The straight portion of the curve between 302 and 303 indicates a rise in temperature without any distillate being collected. This is the initial increase in temperature when the distillate is flowing down the inside surface of the collecting cylinder 32. After point 303 on the curve the distillate has begun to collect in the bottom of the cylinder, so that it intercepts the light beam to photocell 67.

The pyrometer portion of the present system comprises a thermocouple 13 adapted to be positioned in the distillation flask 10, a suitable potentiometer pyrometer, and the rotatable drum 77 to which the recording chart can be attached. Any suitable type of thermocouple and mounting may be used but the preferred type of thermocouple mounting used is one whose performance closely duplicates the thermal performance of a standard ASTM distillation thermometer. A preferred form of thermocouple mounting, as shown in Fig. 8, comprises a glass tube 172 of sufficient length and size so that its heat conductivity and capacity is substantially equal to that of the glass stem of the ASTM thermometer. Mounted at one end of said glass tube 172 is a metallic plug or tip 173 having a heat capacity substantially equal to that of the mercury and glass ASTM thermometer bulb and being capable of being fused to glass. The metallic plug 173 has an axial bore 174 therethrough so that the thermocouple wires 175 may be passed through it and soldered or brazed in place at the end 176 of the bore 174. The wires 175 may be also sealed by means of an insulating plug 177 at the other end of the tube 172. If an all metal thermocouple unit is desired, one having a tube 172 of stainless steel and a tip 173 of brass may be used. Such a metallic mounting may be sometimes preferred to a more fragile glass mounting.

The potentiometer pyrometer 117 preferably comprises a standard continuous self-balancing potentiometer recorder employing cold junction compensation of a conventional type, and an amplifier for converting the minute direct voltage from thermocouple 13 to an A. C. voltage of proper phase and magnitude to drive the drum motor 78, which forms part of this recorder. The sliding contact 96 of the potentiometer resistor wire 95 is mounted on the drum 77. The resistor values in the potentiometer bridge circuit are selected to obtain the desired range for the instrument. Since a recorder of this type uses a balancing motor for an automatic adjustment of its potentiometer circuit, the rotatable drum 77, when connected to the motor 78 of this recorder is rotated in one direction or the other as the temperature rises and falls.

The temperatures recorded during an ASTM distillation are not the true temperatures, but are in error by the amount of emergent stem error of the thermometer which varies according to the temperature. Thus, in calibrating the

recording mechanism it is necessary to compensate for this. The emergent stem error of an ASTM distillation thermometer varies from 0° error at 32° F. to 25.4° error at 580° F. Since the error is non-linear over this range, the preferred method of compensating for the errors is to make the correction to the chart by distorting the temperature graduations thereon. Instead of a standard thermocouple E. M. F. chart, a new chart is plotted having unequal divisions in the temperature scale. Thus on the chart 91 (Fig. 7), the spacings between successive 50° abscissas are made increasingly larger to compensate for the increasing temperature error due to the emergent stem of a thermometer. By using a chart of this type no correction need be made to the data recorded on the chart in order to obtain temperature readings equivalent to those obtained by an ASTM thermometer.

The pen 74 that is attached to the split frame 62 of the photocell 67 contacts the paper and records the volume of distillate in the cylinder 32 as the volume photocell 67 follows the rising meniscus. At the same time the recording drum is rotated by the motor 78 to record changes in temperature. The remaining data to be recorded on the chart, and preferably on the curve, are time marks or intervals to indicate time elapsed at any point during the distillation. As seen from Fig. 7, the time marking system is adapted to actuate the recording drum 77 so that the recording pen 74 draws marks 305 on the distillation curve 300 every minute after the distillate starts collecting in the cylinder 32. The system also causes a longer mark 304 to be recorded every ten minutes after the heat has been applied to the flask 10. Thus, the temperature at any minute during the distillation is known. The first ten-minute mark 304 shows that the drop of distillate dropped in the cylinder 32 within ten minutes after the distillation had started as prescribed by ASTM.

Referring to Fig. 1, the time marking system may comprise a time marking motor 131 and a suitable gear system having cams 144, 145, 146 and 147 attached thereto. The rotating cam 146 closes switch 149 momentarily every minute to shunt a high resistance 151, such for example as 2 megohms across the cold junction compensating network of the potentiometer pyrometer system, thus disturbing the bridge balance and causing the bridge to momentarily seek a new balance point. This in turn causes the motor 78 and connected drum 77 to give a momentary deflection under pen 74, thus making a short mark 305 on the curve 300 as shown in Fig. 7. Every ten minutes, a second actuating cam 145 closes switch 148 simultaneously with switch 149, thus putting another resistance 152 (e. g. one megohm) in parallel with resistance 151, which results in a greater disturbance to the bridge circuit and in a longer mark 304 on the curve 300. A magnetic clutch 118 (and its actuating solenoid 132) connected in parallel with the time marking motor 131 permits the resetting of the time marking cam and gear system to zero at the completion of the distillation. Resetting is done by the unwinding action of a coil spring 115 attached to the gear system which is wound up by motor 131 during the distillation run. A cam 147 contacts a stop 150 when the gear system returns to zero, thus assuring the correct positioning of the cams 144, 145 and 146 in readiness for the next distillation run.

The distillation is carried on under automatic heat control until approximately 5 ml. of liquid remain in the distillation flask 10 as prescribed by the ASTM operating procedure. In actual practice, this point is said to have been reached when a volume of 93 to 94 ml. has been collected in the cylinder 32, as 1 to 2 ml. are assumed to be flowing through the condenser tube 20. Heat applied to the distillation flask 10 must be maintained at a constant rate from the time the 95% point is reached until the end point is reached, as prescribed by ASTM. The extending arm 165 on the rising photocell split frame 62 contacts the collar 164 on the switch-actuating rod 160, which raises said rod 160 and opens the switch 138, thus stopping the timing potentiometer motor 111. At the same time, the sliding contact 109 on the volume potentiometer 107 slides on a sleeve 141 mounted on the upper end thereof but electrically connected thereto at the 95% mark so that even though the contact 109 continues to rise it remains at the same potential. In effect, no further changes in the settings of both potentiometers 107 and 108 are thus permitted, and heat is generated at a constant rate for the rest of the distillation.

When the end point or maximum temperature of the distillation is reached and the temperature starts to fall, the chart drum 77 which has been rotating in one direction automatically stops and rotates in the other direction as the balancing motor 78 of the recording potentiometer reverses. After the temperature has fallen and the drum 77 has rotated backwards a predetermined amount, e. g. an amount corresponding to 15 or 20° F., any suitable switch-actuating means, such as the potentiometer contact 96 attached to the drum, contacts and opens the end point switch 128, which in turn opens contacts 126 and 127 of coil 124, and contacts 136 and 137 of coil 123. When this takes place, the heater 28 is turned off, the time marking motor 131 stops, and the magnetic clutch solenoid 132, attached thereto, is de-energized allowing the spring-loaded time marks gear and cam system to return to zero. The solenoid 43 is de-energized allowing the spring-loaded rod 36 and attached pen-lifting bar 76 to lift the pen 74 off the chart, and the "down" contact 139 is closed which reverses the volume follow-up motor 57 and causes the volume follow-up photocell 67 to be lowered until it reaches its original or zero position, at which time the zero limit switch 166 is opened, shutting off the motor 57. About two minutes is required for the volume motor 57 to reset photocell 67 to its original position. During this time the operator sets the equipment up for another run.

We claim as our invention:

1. An automatic system for controlling and recording the distillation of a liquid, comprising a flask adapted to hold said liquid, electric heater means for said flask, a transparent receiver of uniform cross section, condenser means for transferring distillate from said flask to said receiver, thermocouple means in said flask responsive to temperatures therein, first photoelectric means mounted for vertical movement adjacent said transparent receiver, said movement being responsive to the rate of change of volume of the distillate transferred thereto by the condenser, timing means, potentiometer means comprising first and second potentiometers connected in parallel for continuously varying the electric current supplied to said heater means, said first potentiometer being responsive to the movement of said photoelectric means, said second po-

tentiometer having a sliding contact actuated by said timing means at a constant predetermined rate, second photoelectric means mounted adjacent said transparent receiver above said first photoelectric means and responsive to the initial discharge of distillate into said transparent receiver, and recorder means responsive to said thermocouple means and said first and second photoelectric means for recording a temperature-volume distillation curve of said liquid, said first and said second photoelectric means comprising a photocell, a light source, a shield covering said light source having a slot therein and a carriage, said photocell and said light source being supported by said carriage in a horizontal plane on opposite sides of the transparent receiver, said slot in the shield covering the light source being positioned to direct a horizontal light beam from said light source through said transparent receiver onto said photocell.

2. An automatic system for controlling and recording the distillation of a liquid, comprising a flask adapted to hold said liquid, electric heater means for said flask, a transparent receiver of uniform cross section, condenser means for transferring distillate from said flask to said receiver, thermocouple means in said flask responsive to temperatures therein, first photoelectric means mounted for vertical movement adjacent said transparent receiver, said movement being responsive to the rate of change of volume of the distillate transferred thereto by the condenser, timing means, potentiometer means comprising first and second potentiometers for continuously varying the electric current supplied to said heater means, said first potentiometer being responsive to the movement of said photoelectric means, said second potentiometer being actuated by said timing means at a constant predetermined rate, second photoelectric means mounted adjacent said transparent receiver above said first photoelectric means and responsive to the initial discharge of distillate into said transparent receiver, and recorder means comprising marker means, a chart, and a movable chart support adapted to maintain said chart in contact with said marker means during the distillation, said recorder means being responsive to said thermocouple means and said first and second photoelectric means for recording a temperature-volume distillation curve of said liquid, said first and said second photoelectric means comprising a photocell, a light source, a shield for said light source having a slot therein, and a carriage, said photocell and said light source being supported by said carriage in a horizontal plane on opposite sides of the transparent receiver, said slot in the shield for the light source being positioned to direct a light beam from said light source through said transparent receiver onto said photocell.

3. A system for automatically recording the distillation curve of a liquid, comprising a flask holding said liquid, heater means for said flask, thermocouple means in said flask responsive to distillation temperatures therein, a transparent receiver of uniform cross section, condenser means in fluid communication between said flask and receiver, a carriage member arranged for vertical motion adjacent the receiver, a first light source and a first photocell supported on said carriage member on opposite sides of the receiver, whereby the amount of light falling on the photocell from the light source through the transparent receiver is varied by the distillate delivered thereto by the condenser, first electrical prime mover means energized by the output of the first photocell to actuate said carriage, whereby the motion of the carriage along the receiver follows the rise of the distillate accumulating therein, a second light source and a second photocell fixedly positioned on opposite sides and near the upper portion of said transparent receiver, whereby the amount of light falling on the second photocell from the second light source through the transparent receiver is varied by the first drop of distillate delivered thereto by the condenser, marker means carried by the carriage, a movable chart support adapted to maintain a chart in contact with the marker means during the recording operation, and second electrical prime mover means energized by the output of the thermocouple means for moving said chart support, whereby a distillation curve is traced on said chart by said marker means as a function of the relative motion of the carriage and chart support with regard to each other.

4. A device according to claim 3, including auxiliary marking means for marking time intervals on said chart during the distillation, said auxiliary marking means comprising prime mover means, a gear system, cam means driven by said prime mover means through said gear system, switch means periodically actuated by said cam means, and resistance means in the potentiometer bridge circuit energized upon the closing of said switch means, whereby the response of the recorder means to the thermocouple means is momentarily altered, causing a slight movement of said chart holder under said marker means to produce a time mark on the chart.

5. An automatic system for controlling the distillation of a liquid and recording a temperature-volume distillation curve thereof, comprising a flask adapted to hold said liquid, electric heater means for said flask, a transparent receiver of uniform cross section, condenser means for condensing and transferring distillate from said flask to said receiver, thermocouple means in said flask responsive to the temperatures above the liquid therein, a carriage member arranged for vertical motion adjacent the receiver, a first light source and a first photocell supported on said carriage member on opposite sides of the receiver, whereby the amount of light falling on said first photocell from said first light source through said transparent receiver is varied by the distillate delivered thereto by the condenser, a second light source and a second photocell positioned adjacent the receiver on opposite sides thereof and above said first light source and first photocell, whereby the amount of light falling on said second photocell from said second light source through said transparent receiver is varied by the first drop of distillate delivered thereto by the condenser, electrical prime mover means energized by the output of the first photocell to actuate said carriage, whereby the motion of the carriage along the receiver follows the rise of the distillate accumulating therein, timing means adjustable to run at a constant pre-set rate, potentiometer means comprising first and second potentiometers connected in parallel for varying the electric current supplied to said heater means, said first potentiometer being responsive to said first photocell output, said second potentiometer being responsive to said timing means, and recorder means responsive to said thermocouple means and said first photocell for recording a temperature-volume distillation curve of said liquid.

6. A device according to claim 5 wherein the recorder means comprises marker means carried by the carriage, a movable chart support adapted to maintain a chart in contact with said marker means during the recording operation, and second electrical prime mover means energized by the output of the thermocouple means for moving said chart support, whereby a distillation curve is traced on said chart by said marker means as a function of the relative motion of the carriage and the chart support with regard to each other.

7. A device according to claim 6 wherein the marker means comprises a spring-loaded stylus normally contacting said chart, a stylus-lifting bar means, and solenoid means associated with said bar means for holding the stylus off the chart prior to the beginning of the distillation, said solenoid means being responsive to said second photocell to allow the stylus to contact said chart when the first drop of distillate enters said receiver.

8. A device according to claim 5 including electrically operated receiver-moving means for positioning said receiver under the discharge end of the condenser means so that the first drop of distillate falls freely within said receiver, and solenoid means associated with said receiver-moving means and responsive to said second photocell, whereby the solenoid means actuates said receiver-moving means to position the inner wall of said receiver against the discharge end of said condenser means after the first drop of distillate falls into said receiver.

9. A device according to claim 5 including variable cooling means for maintaining the temperature adjacent the outside of said receiver at a substantially constant predetermined temperature.

10. A device according to claim 5 wherein the first and second potentiometers comprise first and second slide wires connected in parallel, a first contact actuated by said photocell carriage to slide on said first slide wire, and a second contact actuated by said timing means to slide on said second slide wire.

11. A device according to claim 10 including potentiometer-setting means for fixing the settings of the contacts on their respective slide wires after a substantial portion of the distillation has been completed, whereby a constant amount of current is supplied to said heater for the remainder of the distillation.

12. A device according to claim 5 wherein the thermocouple means comprises a tubular glass body member, a metallic tip fixedly secured to one end of said body member, and thermocouple wires within said tubular body member, said wires being fixedly attached at one end to said metallic tip.

13. An automatic system for controlling and recording the distillation of a liquid, comprising a flask adapted to hold said liquid, electric heater means for said flask, a transparent receiver, condenser means for transferring distillate from said flask to said receiver, thermocouple means in said flask responsive to temperatures therein, first photoelectric means mounted for vertical movement adjacent said transparent receiver and responsive to the change of volume of the distillate transferred thereto by the condenser, timing means, potentiometer means responsive to the movement of said first photoelectric means and said timing means for continuously varying the electric current supplied to said heater means to increase the distillation temperature at a substantially constant rate, potentiometer-setting means for fixing the settings of the contacts on their respective slide wires after a substantial portion of the distillation has been completed, whereby a constant amount of current is supplied to said heater for the remainder of the distillation, second photoelectric means mounted adjacent said transparent receiver above said first photoelectric means and responsive to the initial discharge of distillate into said transparent receiver, and recorder means responsive to said thermocouple means and said first and second photoelectric means for recording a temperature-volume distillation curve of said liquid.

14. An automatic system for controlling and recording the distillation of a liquid, comprising a flask adapted to hold said liquid, electric heater means for said flask, a transparent receiver, condenser means for transferring distillate from said flask to said receiver, thermocouple means in said flask responsive to temperatures therein, first photoelectric means mounted for vertical movement adjacent said transparent receiver and responsive to the change of volume of the distillate transferred thereto by the condenser, timing means, potentiometer means responsive to the movement of said first photoelectric means and said timing means for continuously varying the electric current supplied to said heater means to increase the distillation temperature at a substantially constant rate, second photoelectric means mounted adjacent said transparent receiver above said first photoelectric means and responsive to the initial discharge of distillate into said transparent receiver, and recorder means responsive to said thermocouple means and said first and second photoelectric means for recording a temperature-volume distillation curve of said liquid, said recorder means comprising a movable chart holder having a relative movement which is a function of the distillation temperature, marker means actuated by the motion of said first photoelectric means which are movable as a function of the volume of distillate recovered in the receiver, a chart carried and said chart holder, and means for momentarily changing the relative position of the marker means and the chart holder for periodically recording time-interval marks on the distillation curve drawn on said chart.

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