United States Patent

[11] **3,580,047**

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[54] **POUR AND CLOUD POINT ANALYZER**
6 Claims, 12 Drawing Figs.

[52] **U.S. Cl.** **73/17**
[51] **Int. Cl.** **G01n 25/02**
[50] **Field of Search** 73/17, 79

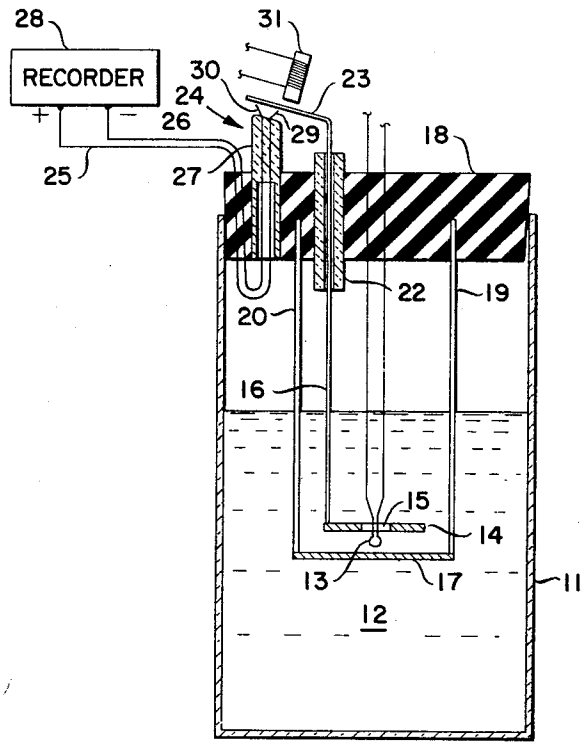
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ABSTRACT: Apparatus for determining the pour point of an oil sample by observing the temperature at which the solidified constituents in the oil sample, upon being heated, will no longer support a small object. This point is indicated by discontinuance in an automatically recorded time/temperature curve during the warning portion of the cycle. The inception point of a plateau in the time/temperature curve during the cooling portion of the cycle is the crystal point. This crystal point correlates with the ASTM cloud point. Modification of the apparatus for continuous service can be accomplished by automatically introducing the sample, then cooling, heating, removing the sample and recirculating a fresh sample.



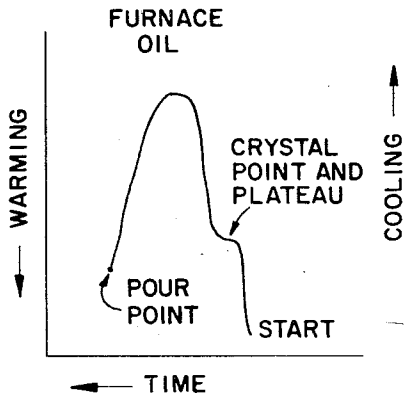


FIG. 2

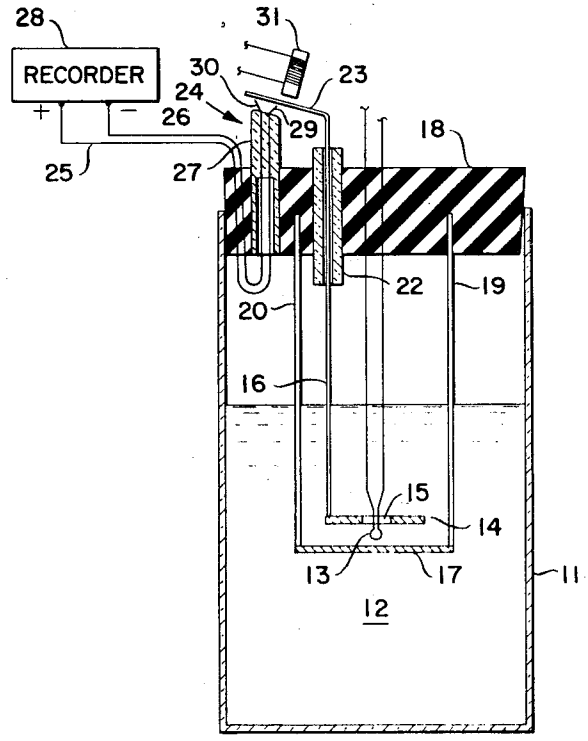


FIG. 1

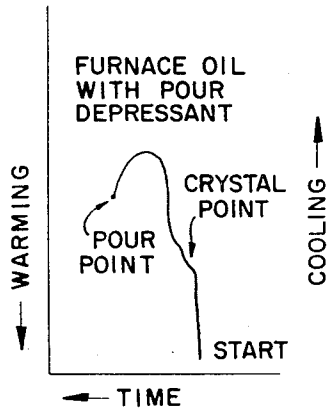


FIG. 3

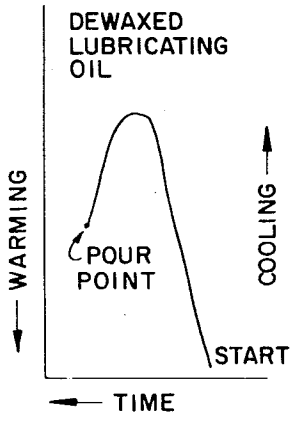


FIG. 4

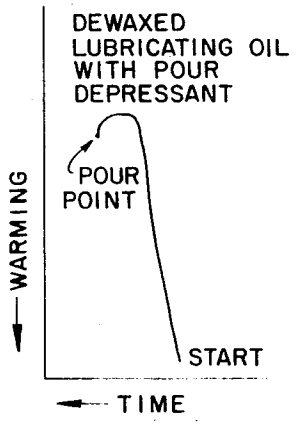


FIG. 5

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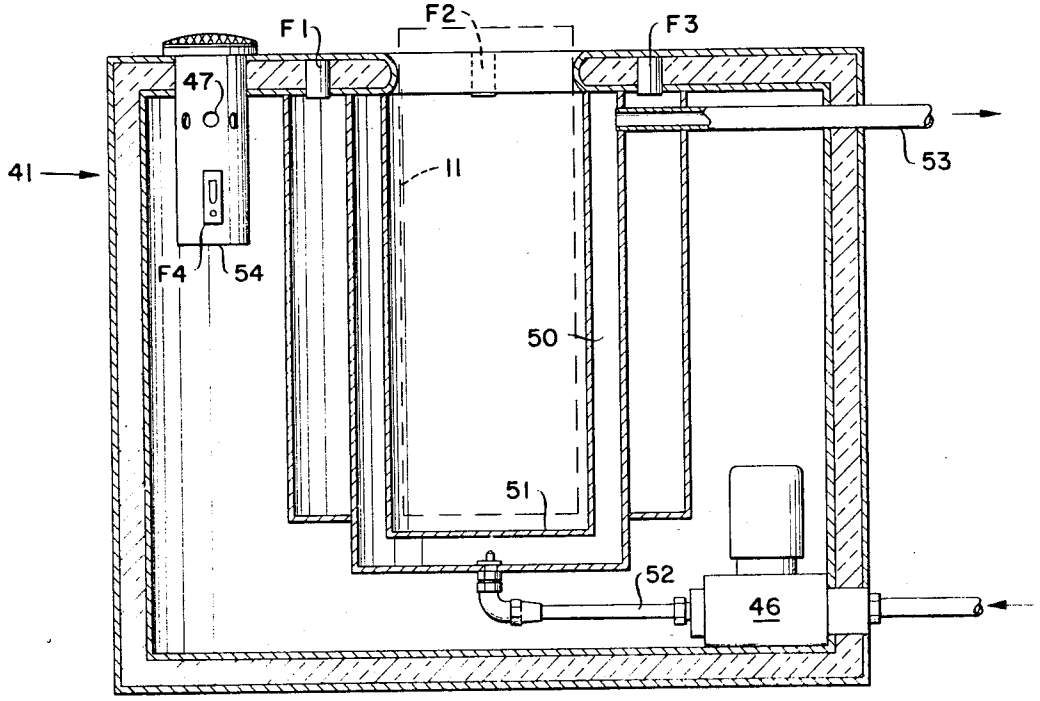


FIG. 7

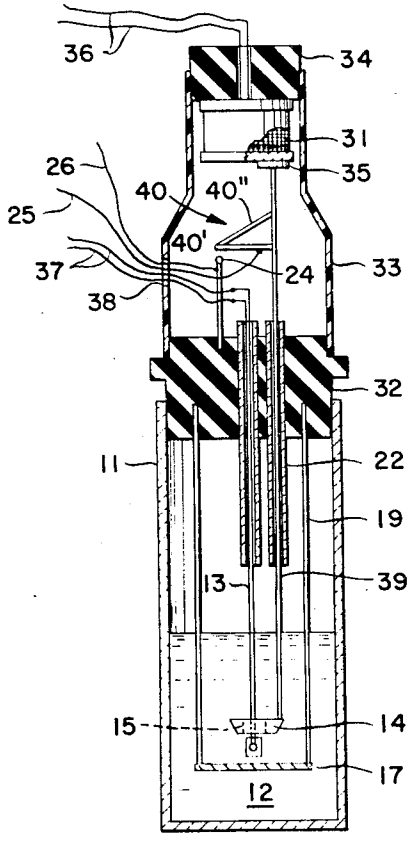


FIG. 6

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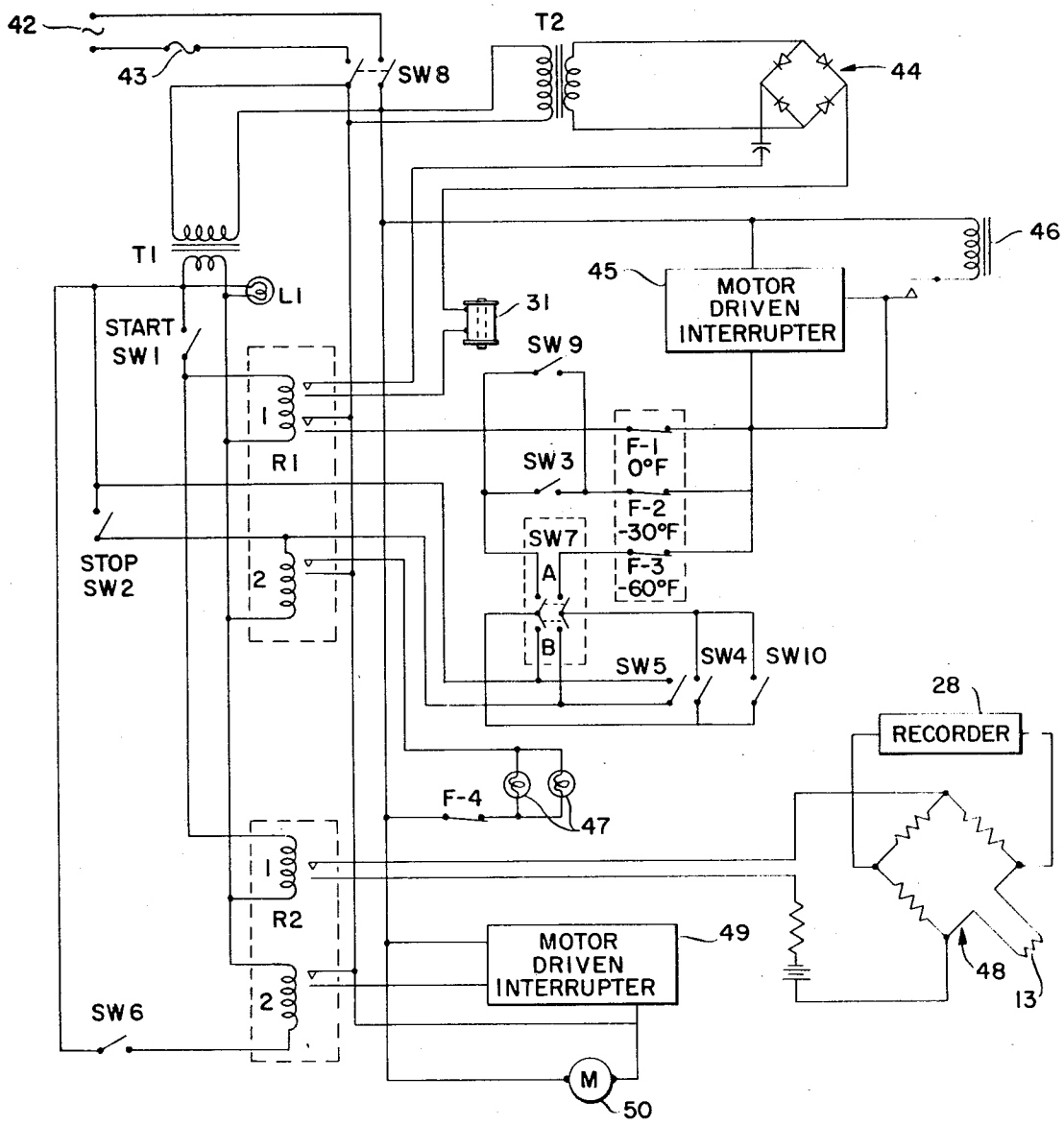


FIG. 8

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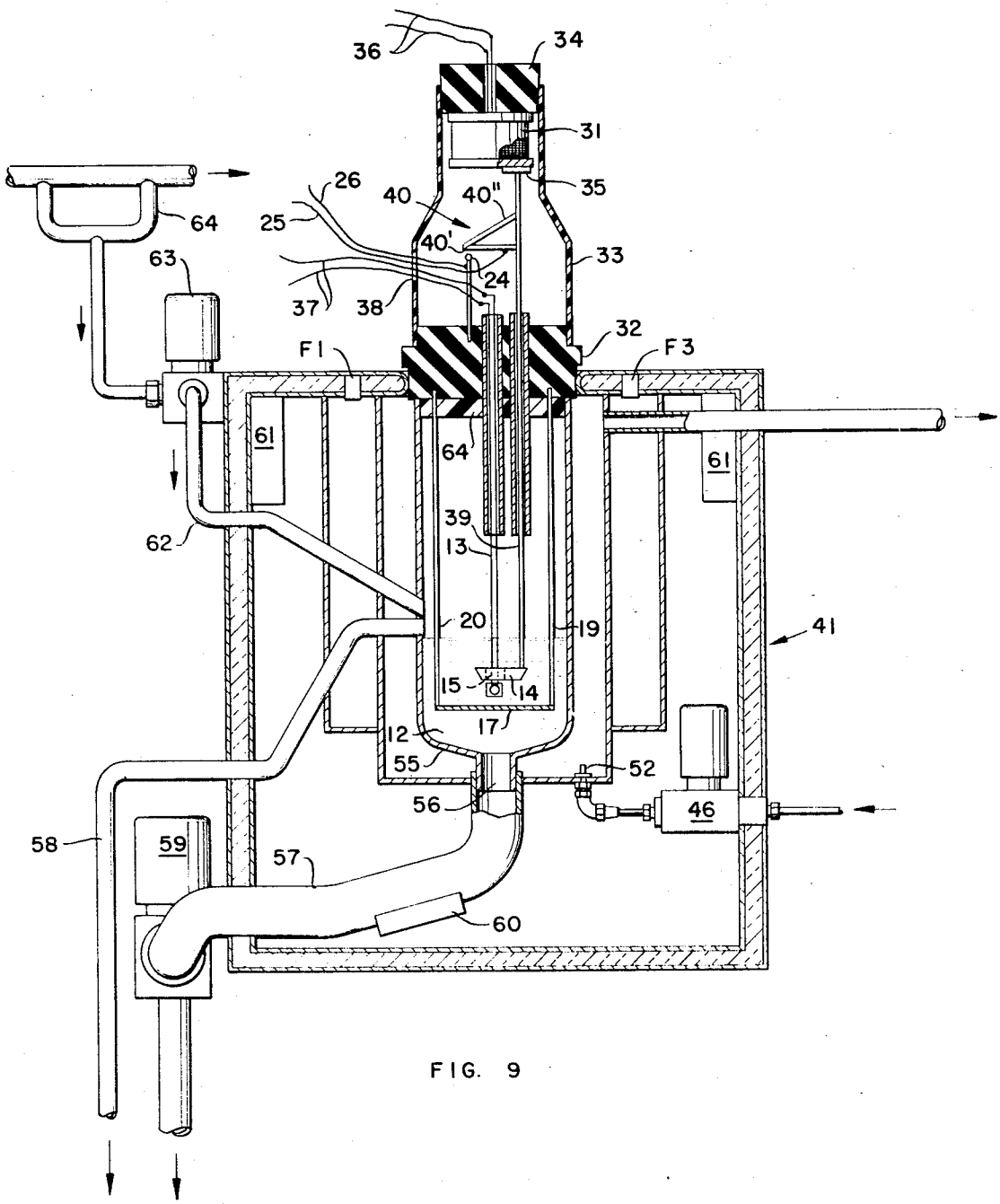


FIG. 9

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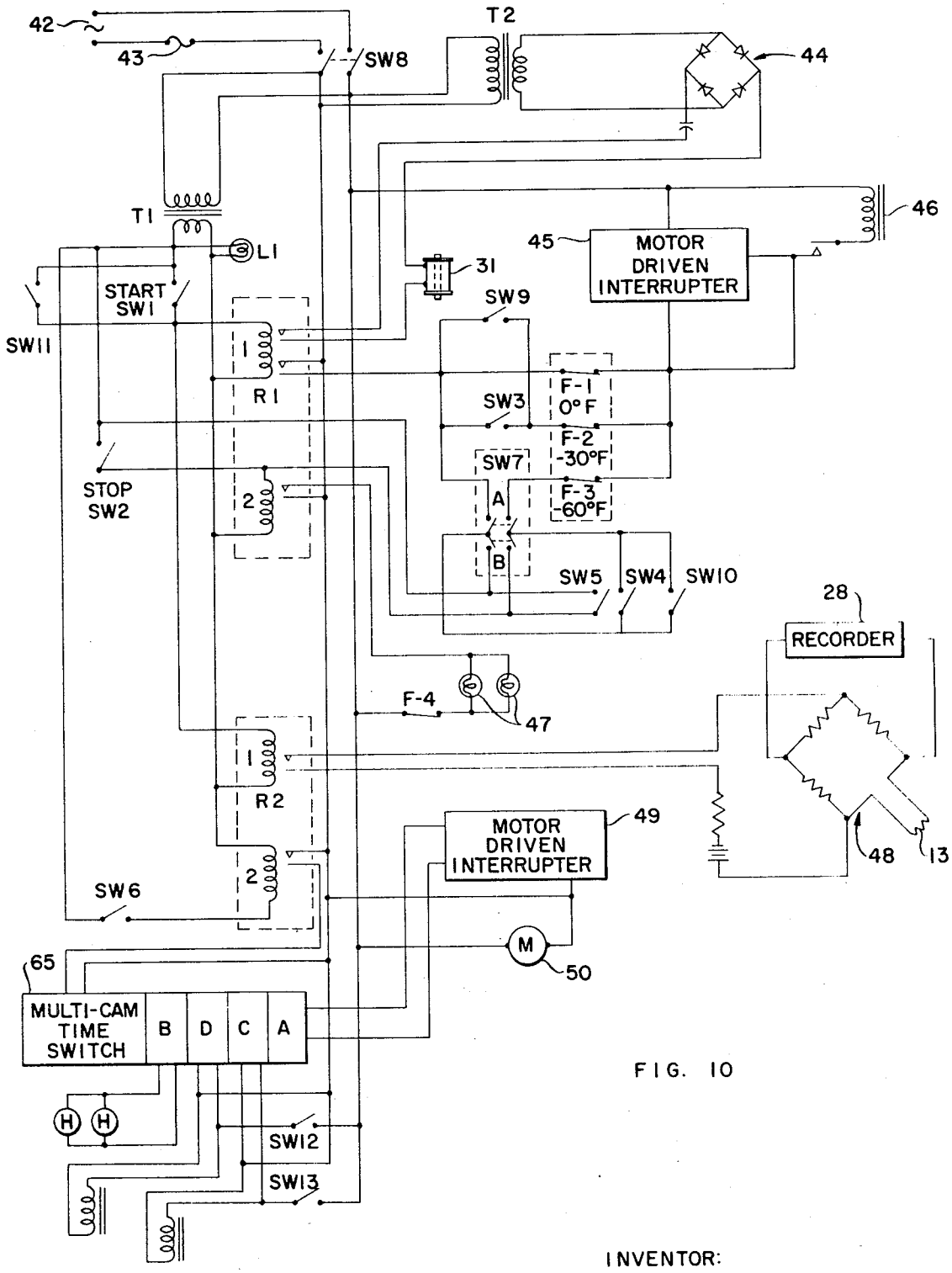


FIG. 10

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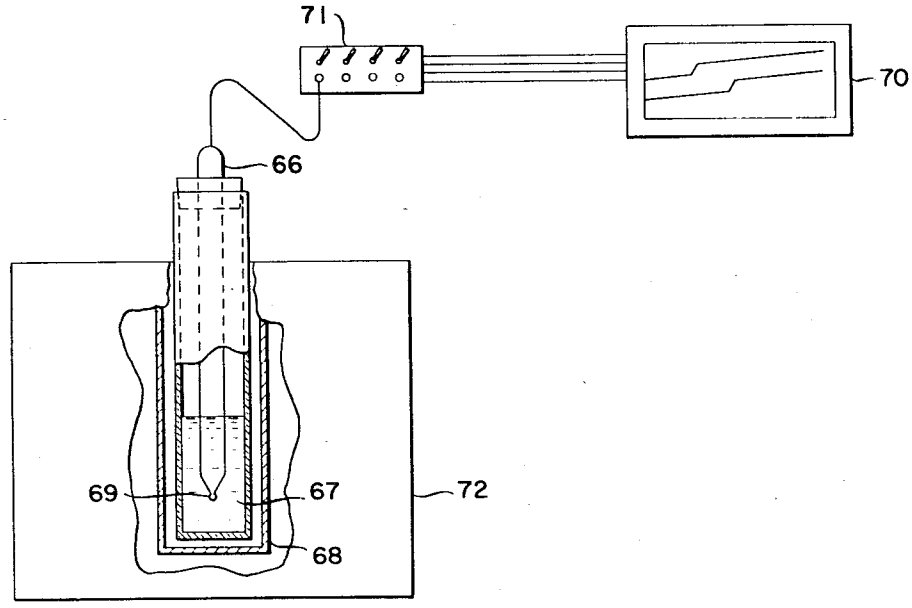


FIG. 11

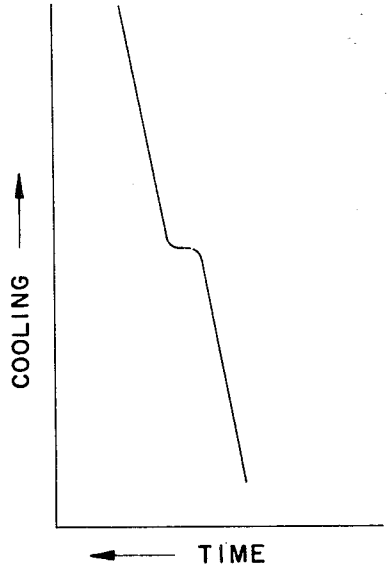


FIG. 12

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POUR AND CLOUD POINT ANALYZER

BACKGROUND OF THE INVENTION

1. Field of the Invention

This invention relates to apparatus for determining the pour and cloud points of liquid compositions. In particular, the invention relates to apparatus for determining our points by heating a cooled sample of a liquid composition until a plate supported solely by the solidified constituents in the cooled sample moves downwardly.

2. Description of the Prior Art

When liquid petroleum products are cooled, a point is reached at which some of the constituents begin to solidify; and if cooling is continued, the oil eventually possesses a viscosity at which it would not flow. The temperature at which this occurs under certain conditions is recorded as the setting or solid point and the pour point is defined at the temperature 5° F. above it. One conventional method to determine the pour point of a liquid composition is to heat a sample in a cylindrical glass jar with a flat bottom to 115° F. or a temperature 15° F. above the expected pour point if this is above 90° F. The sample is then cooled at a specified rate until there is no apparent movement of the surface of the sample when the jar is held horizontally for 5 seconds. This is the setting point; and the pour point, as discussed above, is 5° F. above it. In modern day practice in oil refineries, hundreds of such pour point tests are carried out daily more or less manually and require the constant presence of an operator whose judgment and skill are relied upon for a determination of the pour points. Manipulations are generally made by hand and there is a high variance from sample to sample and from operator to operator so that, for any particular oil under test, the results obtained by several operators may vary as much as 15° F.

SUMMARY OF THE INVENTION

It is an object of this invention to provide more accurate apparatus for determining the pour point of a liquid composition, such as a lubricating oil.

It is a further object to provide means for automatically measuring the pour point of lubricating oil base stocks and other oils containing chemical pour depressants. Presently known automatic pour point instruments are not usable on most lubricating oils.

It is a still further object of the invention to provide a continuous automatic instrument for process control or for lubricating compounding.

The teachings of the invention are carried out by disposing a cooled sample of a normally liquid composition, having constituents which solidify upon cooling, into a container. A thermistor probe is then inserted into the container and below the level of the sample in order to measure the temperature of the sample. Means for recording changes in temperature of the sample are connected to the probe. A movable plate is disposed within the container and is supported solely by the solidified constituents within the sample. The plate has an opening to accommodate the lower end of the probe. The liquid constituents directly below the plate are supported within the container by means of a second plate rigidly fastened in the container. Movement indicating and recording means are attached to the first-named plate for detecting any downward movement when the sample is heated until the softening point in the vicinity of the probe is reached and the plate moved downwardly since it can no longer be supported by the solidified constituents.

The information derived from the apparatus of this invention can be further used to determine the cloud point of the liquid sample since the crystal point of the sample correlates directly with the ASTM cloud point. The thermistor crystal point derived at by the preferred method has been found to be the most accurate tool to predict the low temperature characteristics of fuel oil.

The apparatus described herein is suitable for use with any liquid composition which contains solidified constituents when cooled. However, it is particularly applicable to oils, including synthetic lubricating oils, mineral hydrocarbon oils, and fuel oils, since the pour and cloud points of these oils is an important aspect of quality evaluation in the oil industry.

These and other advantages will be further apparent from the following description of a preferred embodiment, taken in connection with the attached drawings, which form a part of this specification.

BRIEF DESCRIPTION OF THE DRAWING

FIG. **1** is a partly diagrammatic vertical sectional view of an arrangement of apparatus for carrying out the invention;

FIG. **2** through **5** are graphs showing typical cooling-warming curves of various oils utilizing the apparatus of FIG. **1**;

FIG. **6** is a diagrammatic view, partly in section, showing the apparatus of FIG. **1** adapted to an automatic assembly;

FIG. **7** is a diagrammatic view, partly in section, of a cooling bath assembly to be utilized with the automatic apparatus of FIG. **6**;

FIG. **8** is a wiring diagram for the automatic apparatus of FIG. **6**;

FIG. **9** is a partly diagrammatic vertical sectional view of an automatic-filling and -discharging assembly to be used with the apparatus of FIG. **6**;

FIG. **10** is a wiring diagram for the automatic-filling and discharging assembly of FIG. **9**;

FIG. **11** is a schematic view of a simplified method and apparatus for estimating automatically certain properties of an oil sample; and

FIG. **12** is a typical cooling curve of the oil sample of FIG. **11**.

DESCRIPTION OF THE PREFERRED EMBODIMENT

Turning to the drawings, FIG. **1** shows a standard pour and cloud point test jar **11**, preferably of glass, for automatically measuring the pour point of an oil sample **12** by observing the temperature at which the solidified constituents in the oil sample **12**, after being heated, will no longer support a small object. A measuring device, such as a thermistor probe **13**, for making temperature measurements at a point source is disposed in jar **11** and below the level of sample **12**. A thermistor probe is a very small solid-state semiconductor with a negative coefficient of resistivity which is used to sense temperature by measurement of resistance. The probe **13** is coupled to both a bridge circuit and a strip chart recorder for making profiles of time versus temperature in a manner to be discussed more fully hereinbelow.

A small, preferably circular, movable plate **14** having a hole or opening **15** in its center is placed around the lower end of probe **13**, as can be seen in FIG. **1**. A wire adjusting rod **16** is attached to plate **14** to indicate any slight downward movement of plate **14** as will be discussed in more detail below. A supporting device such as a plate **17** is rigidly supported below the lower end of probe **13**. Plate **17** supports any solidified constituents in the cooled oil sample immediately surrounding and directly below the probe end of thermistor probe **13**. This support plate **17** is necessary to prevent the solidified constituents surrounding plate **14** and the lower end of probe **13** from sinking into the warmer surrounding liquid of sample **12** when the sample is being heated, thereby indicating downward movement of plate **14** before the pour point of sample **12** is reached at the lower end of probe **13**. In other words, the property being measured is the temperature when plate **14** sinks in sample **12** through the solidified constituents and not the temperature when the solidified constituents begin to liquefy and sink within the surrounding liquid of sample **12**.

Jar **11** is closed by a resilient stopper **18** which has a plurality of openings extending therethrough to allow the various components of the apparatus to pass. Supporting plate **17** is preferably rigidly mounted in jar **11** by a pair of rigid means such as wires **19**, **20**. Wires **19** and **20** are preferably attached at one end to stopper **18** and at their free end to supporting

plate 17. Wire rod 16 is freely slidable in glass bushing 22 which bushing is fixed in stopper 18. The upper portion of wire rod 16 extends beyond stopper 18 and is angled at 23 for a purpose to be described hereinbelow. A switch 24 consisting of a pair of wires 25 and 26 is rigidly mounted in a glass housing 27 fixed in stopper 18. One end of wire 25 is connected to the positive terminal of a strip chart recorder 28 and passes through stopper 18 and out of the top of glass housing 27 where it is angled at 29 to form one-half of a seat for angled portion 23 of wire rod 16. One end of wire 26 is connected to the negative terminal of strip recorder 28 and passes through stopper 18 and out of the top of glass housing 27 where it is oppositely angled at 30 to angled portion 29 of wire 25 to form the other half of a seat for angled portion 23 of rod 16 as can be seen in FIG. 1. The purpose of wires 25 and 26 is to short out strip recorder 28 when contact is made with the adjusting wire rod 16 which moves downwardly whenever plate 14 is sinking in sample 12. The wires 25 and 26, at least adjacent angled portions 29 and 30, thus must necessarily be extremely close to each other so that angled portion 29 can readily bridge the gap between them.

In a preferred embodiment of the invention, an electromagnet 31 automatically lifts and holds the angled portion 23 of rod 16 in spaced relation from switch 24 until the sample 12 is cooled beyond its solid point. When this point is reached, the sample 12 in its jar 11 is moved to a warmer bath, the electromagnet 31 is removed and the sample 12 is allowed to soften while being heated. The electromagnet 31 is supported by any known means and can be removed either manually or automatically as will be discussed hereinbelow.

Alternately, a small section of wire insulation (not shown) can be placed on angled portion 23 of rod 16 and removed when desired.

Accordingly, after actuating strip recorder 28 to record changes in the temperature of the sample 12, the solidified constituents in sample 12 will support the adjusting rod 16 and plate 14 until the softening point of the sample 12 in the immediate vicinity of thermistor probe 13 is reached. At the softening point, the plate 14 and adjusting rod 16 will begin to move slowly downwardly. Experiments have shown this rate of movement to be approximately 1 millimeter per degree Fahrenheit i.e., the rate of downward movement of plate 14 and rod 16 is for a distance of approximately 1 millimeter for each degree Fahrenheit of temperature change. After approximately 1 millimeter of downward movement, the angled portion 23 of adjusting rod 16 comes into contact with switch 24 thereby shorting and thus stopping the strip recorder 28 at the temperature shown experimentally to correspond to the pour point of the oil sample 12. Comparative data have demonstrated the disclosed method to give more accurate pour point values than the conventional manual method. The preferred method is not influenced by tester bias, nor are the determinations restricted to products which do not contain pour point depressants since the test is made on a static sample.

FIGS. 2 through 5 show typical cooling and warming curves resulting from tests performed on different types of samples as indicated using the preferred method. The pour point is indicated by discontinuance of the time versus temperature curve when the plate begins to sink in the sample. The crystal point of the furnace oils is indicated by the portions of the curves in FIGS. 2 and 3. In other words, the inflection point of a plateau in the time versus temperature curve during the cooling or chilling portion of the cycle is the crystal point. The crystal or cloud points determined here are directly equivalent to the ASTM (D-97) cloud point (i.e., the American Society for Testing and Materials standardization of cloud points) as a definite correlation exists.

FIG. 6 shows the preferred method of FIG. 1 adapted to an assembly for automatic operation. The assembly of FIGS. 6 is to be used in conjunction with the cooling bath assembly of FIG. 7. In FIG. 6, like elements refer to like parts of FIG. 1.

A resilient stopper 32, having a preferably plastic integral housing 33 on its upper end, has been substituted for the stopper 18 of FIG. 1. The electromagnet 31 of FIG. 1 is mounted in the upper portion of housing 33 and the extreme upper portion of housing 33 is closed by resilient stopper 34. A plate 35 is magnetically retained in position by electromagnet 31. The plate 35 is preferably of soft iron and covered with aluminum foil to overcome any residual magnetism and insure release by the electromagnet 31 whenever desired. Wires 36 couple the electromagnet 31 to conventional releasing means (not shown in FIG. 6). Wires 37 couple the thermistor probe 13 to both a bridge circuit and the strip recorder 28 as will be discussed more fully hereinbelow. Wires 37 and switch wire 25 and 26 extend out of an opening 38 in housing 33. An adjusting rod 39 is fixedly attached at its upper end to plate 35 and at its lower end to plate 14. Rod 39 passes through an opening in stopper 32. Thus, rod 39 has been substituted for the rod 16 in FIG. 1 embodiment and the release of electromagnet 31 allows iron plate 35, attached rod 39, and plate 14 to move downwardly as the liquid sample is heated. Rod 39 has an angled portion 40 adapted to engage switch 24 as it moves downwardly. Extension portion 40 is formed of a horizontal leg portion 40' having an end attached to vertical rod 39 and its free end attached to an angled portion 40', the free end of which is also attached to rod 39 as can be seen in FIG. 6. Both switch 34 and extension 40 are preferably of platinum and can contact each other under a force of less than 4 grams. Conventional leaf spring switches having platinum contacts may be substituted for the switches 24 and 40 if desired.

After a specified volume of sample 12 is introduced into the test jar 11, the glass lower portion (below stopper 32) is lowered into the cooling bath assembly 41 of FIG. 7. The cooling bath assembly 41 shows three adjustable thermoswitches F-1 through F-3 mounted in the upper portion of assembly 41 which have been preset to control (i.e. make and break) at temperature of 0° F., −30° F. and −60° F., respectively. A hollow chamber 50 surrounds bath portion 51 of the apparatus. This chamber 50 allows for expansion for a cooling agent, such as Freon, which is introduced through solenoid 46 as will be explained hereinbelow. Solenoid 46 is coupled to chamber 50 of bath portion 51 by any known means, such as tubing 52. Vent 53 at the upper portion of cooling bath assembly 41 and communicating chamber 50 allows for the relief of pressure of the cooling agent introduced into chamber 50. Thermoswitch F-4 is coupled to a pair of lamps 47 in lamp housing 54 and to the automatic-cooling circuit as will also be discussed below. The cooling bath assembly 41 can be programmed to first cool to 0° F., and then to −30° F., and then either stop or proceed to −60° F. (as required to simulate the ASTM (D-97) method) or it can cool directly to −60° F. without stopping at the 0° F. or −30° F. points. The use of the rapid cooling rate whenever possible is advantageous because it reduces the total testing time. Distillate fuels such as gas oils and furnace oils can be tested with a rapid cooling rate. However, pour points of some lubricating oil base stocks are affected by cooling rate when they contain chemical pour depressants. This precludes the use of the fast cooling rate of all compounded lubricating oils containing these base stocks. Also, in order to conform to the ASTM (D-97) method, prescribed cooling methods must be used.

Referring now to FIG. 8, current from any preferred source, such as AC source 42, to the circuit is controlled by main power switch SW-8 and fuse 43. A transformer T1 energizes light source 11 and also energizes latching relays R1 and R2, relays R1 and R2 being controlled by starting switch SW-1 and stopping switch SW-2. Power source 42 also energizes transformer T2 which supplies a rectifier bridge 44 for the voltage supply to electromagnet 31 which is in circuit with the latching coil 1 of relay R1. Switches SW-9, SW-3, and SW-7, respectively, control thermoswitches F-1 through F-3 through the primary windings 1 of relay R-1. Switch SW-9 is preferably mounted on the control box (not shown), switch SW-3 is coupled to strip chart recorder 28 and is preferably preadjusted to be actuated at +20° F., and switch SW-7 is a

double-pole, double-throw switch for bypassing the lower limit switch and stopping the cooling cycle at −10° F. as will be explained in detail later. Switches F–1 through F–3 are thermoswitches which are normally closed at above control temperature and are adjustable. F–1 is preferably normally preset to control at a temperature of 0° F., F–2 at −30° F., and F–3 at 60° F. A motor driven interrupter **45** is in circuit with thermoswitches F–1 through F–3 and is controlled by solenoid **46**, an electrically operated valve which opens on signal from the device and admits the cooling agent to initiate the cooling cycle as will be explained hereinbelow. Lamps **47** in the cooling chamber are energized by a small thermoswitch F–4, preadjusted to +40° F., through the delatching coil **2** of relay R–1. The latching coil **1** of relay R–2 energizes the temperature-measuring bridge circuit **48** coupled to both strip recorder **28** and thermistor probe **13**. The delatching coil **2** of relay R–2 is coupled to a motor driven interrupter **49** for the balancing motor **50** of recorder **28**. SW–6 is preferably of platinum and is a contact switch energized by the secondary winding **2** of relay R–2 and refers to the probe contacts (for example, **23** and **24** in FIG. **1**) that close when the pour point is reached.

The programming control of the wiring diagram of FIG. **8** is thus accomplished in the following manner. When the start switch SW–1 is pressed, it energized the two latching relays R–1 and R–2 which in turn energize the separate circuits that control the adjusting rod **16** and plate **14**, the temperature-measuring bridge circuit **48**, and the cooling cycle. The cooling cycle is initiated when the start switch SW–1 energizes thermoswitch F–1 which is preferably preset to a control at a temperature of 0° F. through the latching relay R–1. Recorder switches SW–3, SW–4, and SW–5, coupled to the strip chart recorder **28**, are normally open and are adjusted to close when the recorded sample temperature reaches +20° F., −10° F., and −40° F., respectively. Recorder switches SW–3, and SW–4, when closed, will complete the circuit to thermoswitches F–2 and F–3 which are preferably preset to control at −30° F. and −60° F., respectively. Switch SW–5 ends the cooling cycle by deenergizing relay R–1 when the recorded sample temperature reaches −40° F. Double pole, double throw switch SW–7 has two positions. In the "B" position, it will end the cooling cycle by deenergizing relay R–1 when the sample temperature reaches −10° F. In the "A" position, switch SW–7 allows the cooling cycle to proceed to a sample temperature of −40° F. This cooling sequence which conforms to the ASTM (D–97) method can be summarized as follows:

ASTM COOLING CONTROL SEQUENCE

Temperature, ° F.		Switch positions							
Bath	Sample	SW–1	SW–3	SW–4	SW–5	F–1	F–2	F–3	SW–7
Ambient	Ambient	O [1]	O	O	O	C	C	C	
0	Approaching +20	O	O	O	O	C/O	C	C	
0	20	O	C	O	O	C/O	C	C	
−30	Approaching −10	O	C	O	O	O	C/O	C	
−30	−10	O	C	C	O	O	C/O	C	B position (end of cooling cycle).
−60	Approaching −40	O	C	C	O	O	O	C/O	
−60	−40	O	C	C	C	O	O	C/O	A position (end of cooling cycle).

[1] Closed momentarily to energize latching relays R–1 and R–2.
NOTE: C denotes closed switch position; O denotes open switch position; C/O denotes controlling point.

For rapid cooling, switches S–9 and S–10 on the control box (not shown) are closed and switch SW–7 is set in the "A" position, thus allowing the bath to immediately cool to −60° F. The bath will remain at −60° F. unless the operator depresses the stop switch SW–2, which terminates the cooling cycle at any point regardless of the remaining switch settings.

A sample **12** is generally cooled until it is at least 10° F. below the expected solid point. When the programmed switching point for stopping the cooling cycle or the manually controlled stopping point is reached, the completion of the circuit through coil **2** of R–1 deenergizes the relay R–1 and allows the adjusting rod **16** and plate **14** to rest freely on the solidified constituents of sample **12**, stops the cooling cycle and turns on thermoswitch-controlled heating lamps to speed the melting or softening cycle. When the sample **12** softens in the immediate vicinity of the temperature sensing thermistor probe **13**, the plate **14** and adjusting rod **16** drop and close the probe contacts (SW–6) in the circuit. This energizes coil **2** of relay R–2 which turns off the bridge circuit **48** and balancing motor of the recorder **28**, tracing a straight line on the strip recorder **28** at the pour temperature.

A continuous chart advance is used to record the temperature in this embodiment; however, the use of a continuous recorder is not necessary where only the pour point is to be measured. In these cases, a simple modification would allow the chart drive motor to be actuated after recording the pour point. This feature would be more desirable on a process instrument because it presents graphically essential points for easy surveillance by the operator.

A sample introduction and removal system for converting the apparatus of FIG. **6** to a continuous processing apparatus is shown in FIG. **9** wherein like numerals refer to like parts of FIG. **6**. Here, glass jar **11** has been replaced by a housing **55**, preferably of copper, having a funnel portion **56** at its lower end communicating with a sample discharge tubing **57**. In other words, the test jar **11** of FIGS. **1** and **6** has been eliminated and the metal wall of the cooling bath itself performs this function. In this embodiment of the invention, if desired, stopper **32** may be eliminated and housing **33** may be connected directly to assembly **41**. The method of programming the cooling cycles will be the same as in the apparatus of FIG. **6**. If necessary, a heat exchanger can be used to precool the materials before charging to the test cell through the solenoid valve **46**. Pipe **58** communicates with the sample **12** in housing **55** and drains off any overflow to a drain (not shown) common to discharge tubing **57** as shown in FIG. **9**. A sample discharge solenoid **59** coacts with sample discharge tubing **57** for purposes to be explained below. A small heater **60** coacts with discharge pipe **57** to heat the drained fluid sample **12**. A pair of heaters **61** are used to heat the sample **12** in housing **55**. The sample **12** is introduced into housing **55** through pipe **62** which communicates with both housing **55** and a sample introduction solenoid **63** which cooperates with a continuous sample bypass **64** for storing sample **12** prior to introducing it in the assembly. A plate **64**, of suitable material that will not be attacked by aromatics present in certain oils, such as Teflon, is preferably attached to the underside of resilient stopper **32** and retains the elements **13**, **19**, **20**, and **39** in place in housing **55**.

The wiring circuit for the continuous processing apparatus of FIG. **9** is shown in FIG. **10** where again like numerals refer to like elements of the circuit of FIG. **8**. The circuit of FIG. **10** is essentially the same as the circuit of FIG. **8** with addition of the continuous processing feature. Initial flushing and filling of the sample cell **55** will be accomplished manually by closing both inlet solenoid switch SW–12 and discharge solenoid switch SW–13 which are coupled to the power supply **42** and the recorder **28** as can be seen in FIG. **10**. This will energize both the sample introduction solenoid valve **63** and the sample discharge solenoid valve **59**. After approximately 1 minute, the sample discharge solenoid valve **59** will then be closed by

opening switch SW–12 to trap the initial sample **12**. Depressing the switch SW–1 will start the cooling cycle and the apparatus will automatically initiate and proceed through repeated cycles of sample analyses.

The functioning of the continuous processing instrument will be identical to the apparatus of FIG. **6** until the pour point is reached. At this point, coil **2** of relay R–2 (see FIG. **10**) will be energized which in turn starts a multicam timer **65**. Four contacts, numbered A through D in FIG. **10**, control four circuits: A interrupts power to the recorder balancing motor **50**, B energized the heater **60** located on the sample discharge tube **57** and the heaters **61** located between the concentric walls of the cooling chamber for a period of time sufficient to assure sample fluidity, C energizes the sample discharge solenoid valve **59** for a period of time sufficient to drain off the sample **12**, and D energizes the sample introduction solenoid valve **63** for a period of time sufficient for flushing and filling the sample housing **55**. This sequence of opening and closing the valves **59** and **63** will drain and refill housing **55** with a fresh sample **12**. Subsequently, the recorder motor **50** will be energized and the recorder **28** will proceed to the warm temperature end of the chart. When the recorder **28** reaches this point, microswitch SW–11 coupled to recorder **28** will energize latching relays R–1 and R–2 again initiating the analyses cycle.

A further embodiment of the invention involves an apparatus and technique for automatically making precise temperature versus time cooling curve measurements from which a very accurate estimate of ASTM cloud point, solid point (i.e., the quasi ASTM solid point used to define the ASTM pour point), and ASTM freeze point of certain solvents such as paraffin-containing hydrocarbons can easily be made.

The technique involves the use of a thermistor probe **66** (FIG. **11**) which is placed in approximately the center of a sample **67** of a solvent such as paraffin-containing distillates, disposed in container **68**. The thermistor probe **66** measures the temperature of the sample **67** as it is lowered at a constant rate. Wax crystals eventually begin to form as the temperature is lowered. When these crystals begin to form around the lower end **69** of thermistor probe **66**, heat is liberated and detected as a plateau on a time versus temperature curve. This plateau correlates with the ASTM cloud point, solid point (i.e., the quasi ASTM solid point), and the ASTM freeze point properties of the distillates. The apparatus illustrated in FIG. **11** for automatically measuring these properties thus consists of a strip recorder **70**, a bridge circuit **71**, the end **69** of thermistor probe **66**, test jar or container **68**, and a cooling bath **72**. The sample **67** is disposed in a standard cloud and pour point test jar or container **68**. Thermistor probe **66** is coupled to a bridge circuit **71** which gives a millivolt signal within the span of a multipoint strip chart recorder **70**. The thermistor probe **66** is inserted into the center of the sample **67**. The test container **68**, thermistor probe **66**, and sample **67** are then placed in a low temperature cooling bath **72**. The temperature is lowered at a constant rate by any conventional means and when wax crystals begin to form around the end **69** of thermistor probe **66**, a plateau appears on the time versus temperature curve recorded by the strip chart recorder **70** as shown graphically in FIG. **12**. The apparatus disclosed is capable of running a plurality of samples simultaneously. It has found use in controlling the cut point in the fractionation of catalytic cracked and straight run gas oils and in controlling the cloud point of finished furnace oils. This apparatus provides for more precise measurements and allows closer regulation of processing to maximize yield without sacrificing quality. It eliminates continual handling of the sample as required by present ASTM methods. The technique and apparatus disclosed in the embodiment of FIG. **11** can be easily adaptable to a process stream analyzer, such as the process disclosed above in FIG. **9**.

From the foregoing description of preferred embodiments of the invention, various alterations and modifications in the details of the method and apparatus may become apparent to one having ordinary skill in the art, and it is to be understood that such modifications and alterations fall within the spirit and scope of the appended claims.

I claim:

1. Apparatus for determining the pour and cloud points of a normally liquid composition having constituents which solidify upon cooling comprising:

a casing adapted to contain a cooled sample of said liquid composition containing solidified components;

temperature-measuring means disposed within said casing for measuring the temperature of said sample as a function of time;

temperature-indicating means connected to said temperature-measuring means for indicating the temperature of said sample;

a vertically movable plate means carried within said casing and adapted to be supported therein solely by said solidified constituents disposed within said casing and surrounding the lower end of said temperature-measuring means;

movement-indicating means attached to said plate means and connected to said temperature-indicating means for indicating any downward movement of said plate;

sample supporting means rigidly carried by said casing below the lower end of said temperature-measuring means to support the solidified constituents of said sample immediately surrounding and directly below said temperature-measuring means;

heating means operatively engaging said casing for heating said sample;

cooling means operatively engaging said casing for cooling said sample;

retention means connected to said casing for maintaining the movement-indicating means in spaced relation from said temperature-indicating means; and

means responsive to a predetermined temperature cooperating with said retention means for releasing said movement-indicating means after the sample has solidified.

2. Apparatus as in claim **1** wherein said plate means has an opening therein to accommodate the lower end of the temperature-measuring means.

3. Apparatus as in claim **1** wherein the weight of said plate means is approximately 4 grams.

4. Apparatus as in claim **1** wherein said cooling means comprises electrically operated solenoid valve means connected to said casing adapted to open to admit a colling medium therein to initiate the cooling cycle of said sample.

5. Apparatus as in claim **4** wherein said cooling means includes electrical circuit means connected to said valve means for actuating said cooling means.

6. Apparatus as in claim **1** including sample refilling means connected to said casing for draining any sample within said casing, flushing said casing after draining and refilling said casing with a fresh sample.