United States Patent [19]

Goolsby et al.

[11] **3,732,723**

[45] **May 15, 1973**

[54] APPARATUS FOR DETERMINING DISTILLATION CUT POINTS

[75] Inventors: **Alvin D. Goolsby**, Moraga; **Benjiman D. Stanton**, Walnut Creek, both of Calif.

[73] Assignee: **Shell Oil Company**, New York, N.Y.

[22] Filed: **Aug. 13, 1970**

[21] Appl. No.: **63,534**

[52] **U.S. Cl.** ..... **73/17 A**

[51] **Int. Cl.** **G01n 25/08**

[58] **Field of Search** 73/15, 17, 36

[56] **References Cited**

UNITED STATES PATENTS

3,135,107	6/1964	Paulik et al.	73/17
3,055,206	9/1962	Watson et al.	73/15
3,271,996	9/1966	Paulik et al.	73/15

OTHER PUBLICATIONS

Topham, "Multistream Distillation Monitors," Control Engineering, Vol. 11, No. 4, April 1964, 65–67.

DuPont Product Bulletin 950–1, Thermogravimetric Anzylzer, received February 1966, 4 pages.

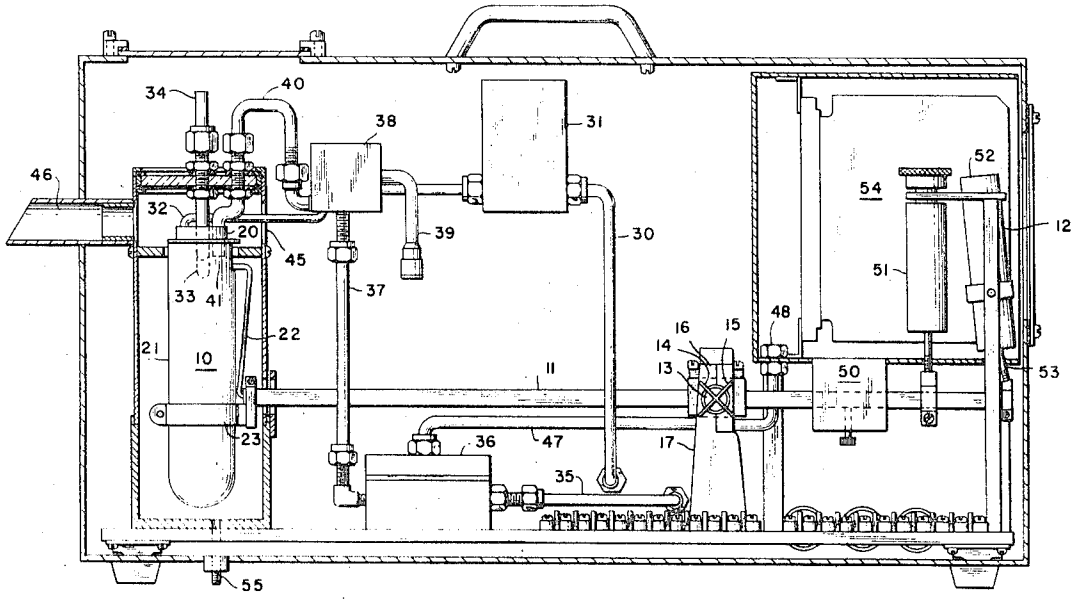
Primary Examiner—Herbert Goldstein

Attorney—T. E. Bieber and H. L. Denkler

[57] **ABSTRACT**

An apparatus for automatically performing distillation analysis of liquids wherein a sample of the liquid is vaporized while the temperature of the vapor and the weight of the sample are continuously measured and recorded. The data is supplied to a computer programmed to provide preset percent distillation points for the sample and hold circuits that can be set to trigger at any preselected boiling point or cut point.

5 Claims, 9 Drawing Figures



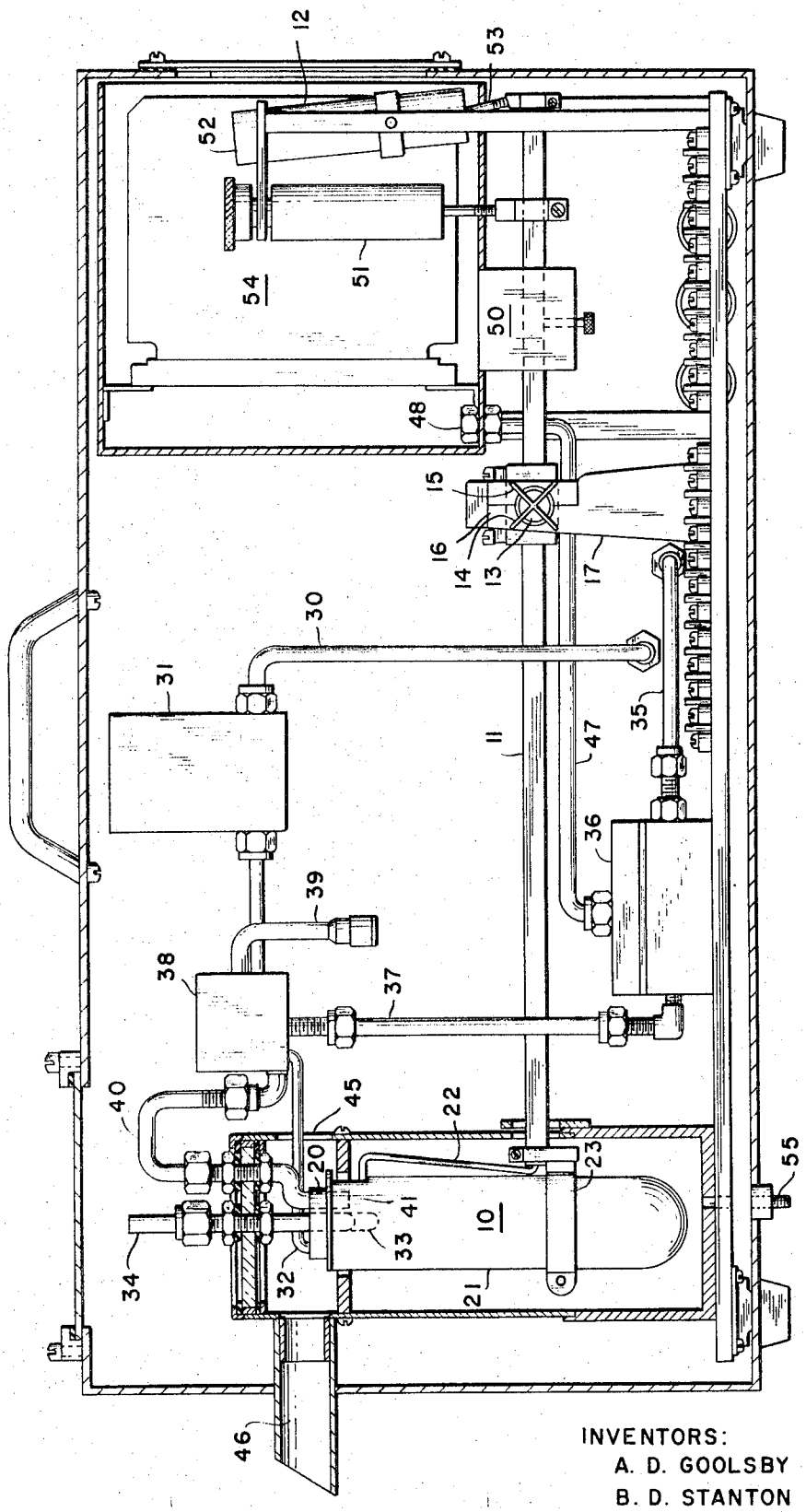


FIG. 1

INVENTORS:
A. D. GOOLSBY
B. D. STANTON
BY: Harold L. Winkler
THEIR ATTORNEY

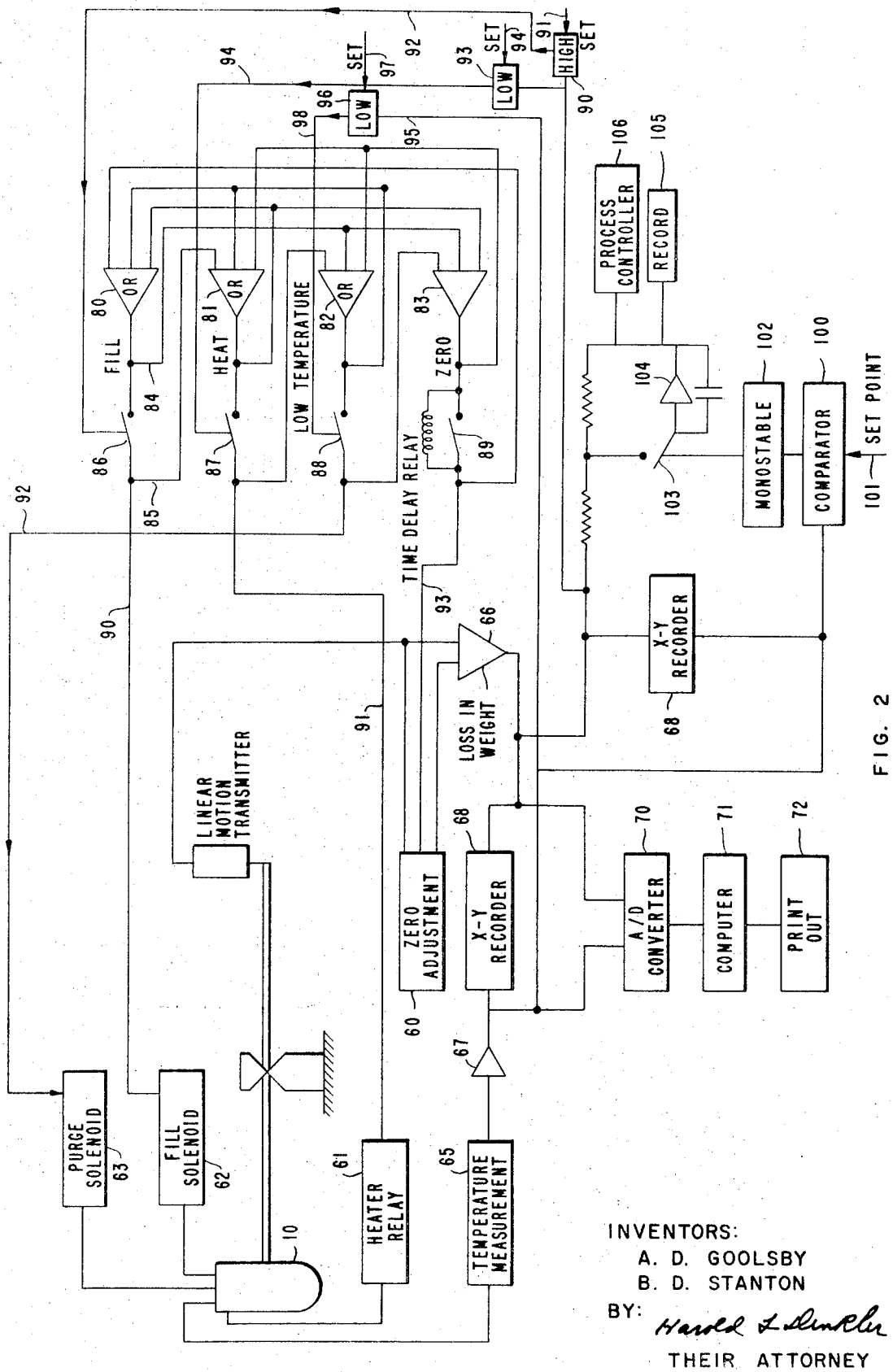


FIG. 2

INVENTORS:
A. D. GOOLSBY
B. D. STANTON
BY: Harold L. Denkler
THEIR ATTORNEY

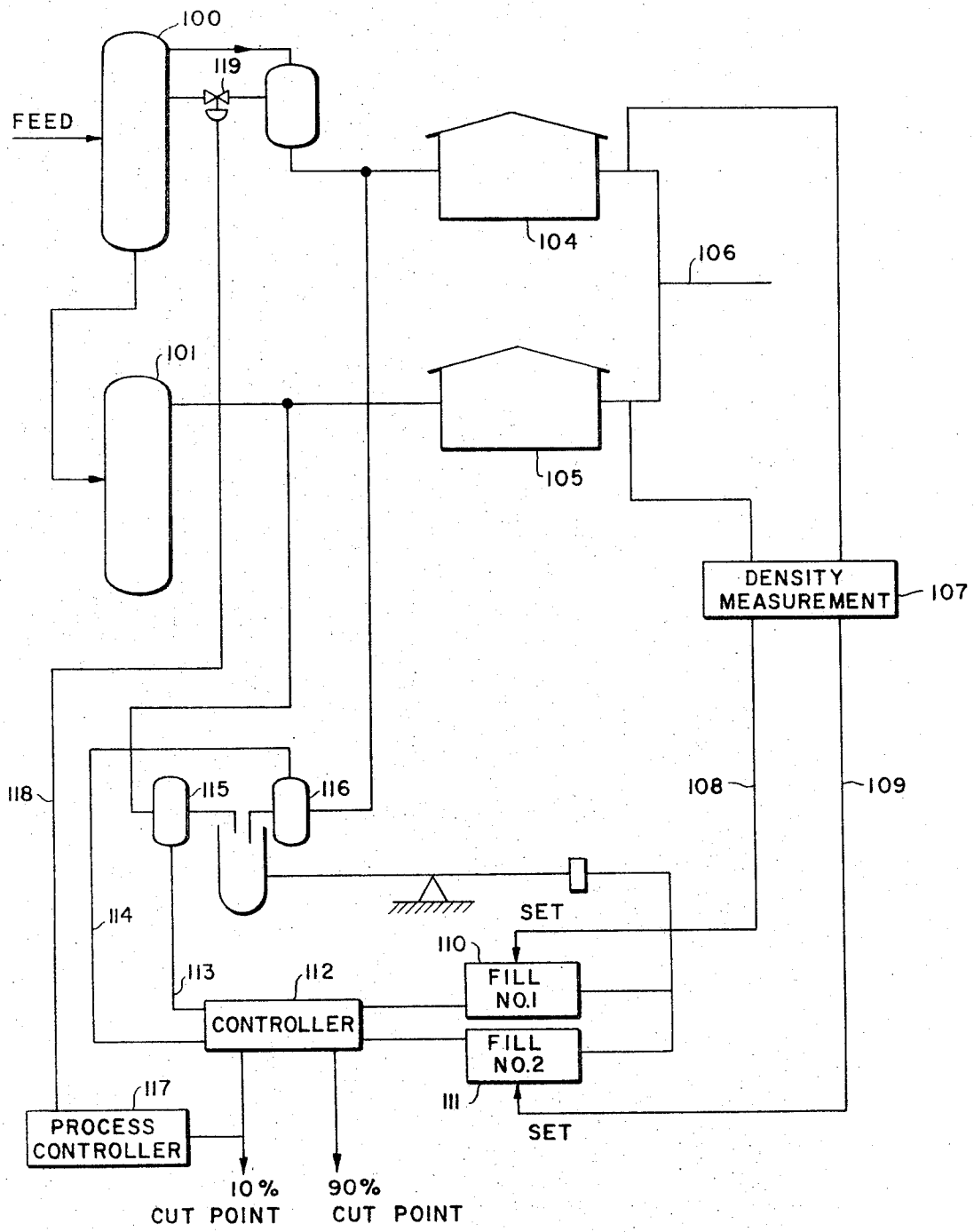


FIG. 3

INVENTORS:
A. D. GOOLSBY
B. D. STANTON
BY: Harold L Winkler
THEIR ATTORNEY

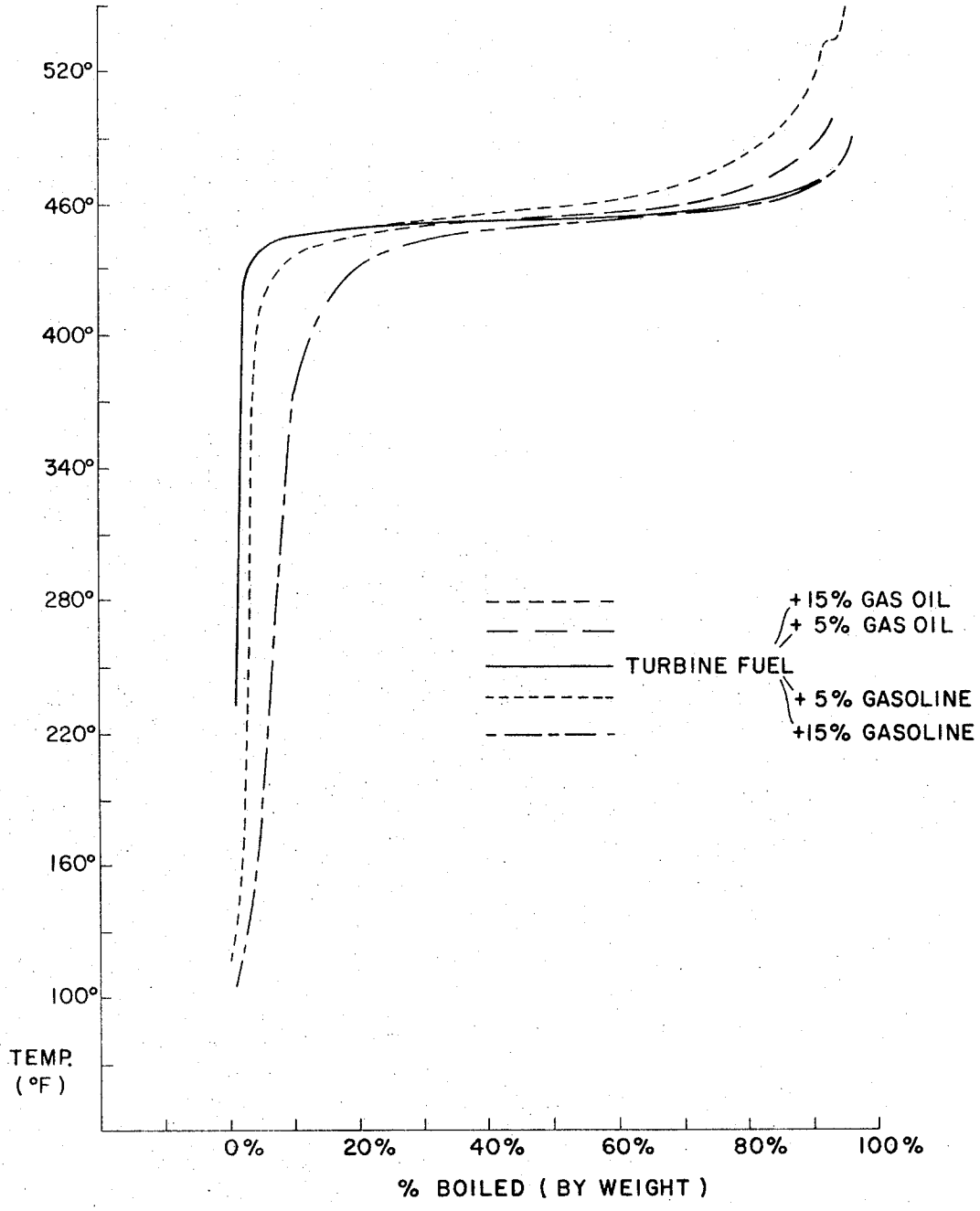


FIG. 4

INVENTORS:
A. D. GOOLSBY
B. D. STANTON
BY:
Harold L. Winkler
THEIR ATTORNEY

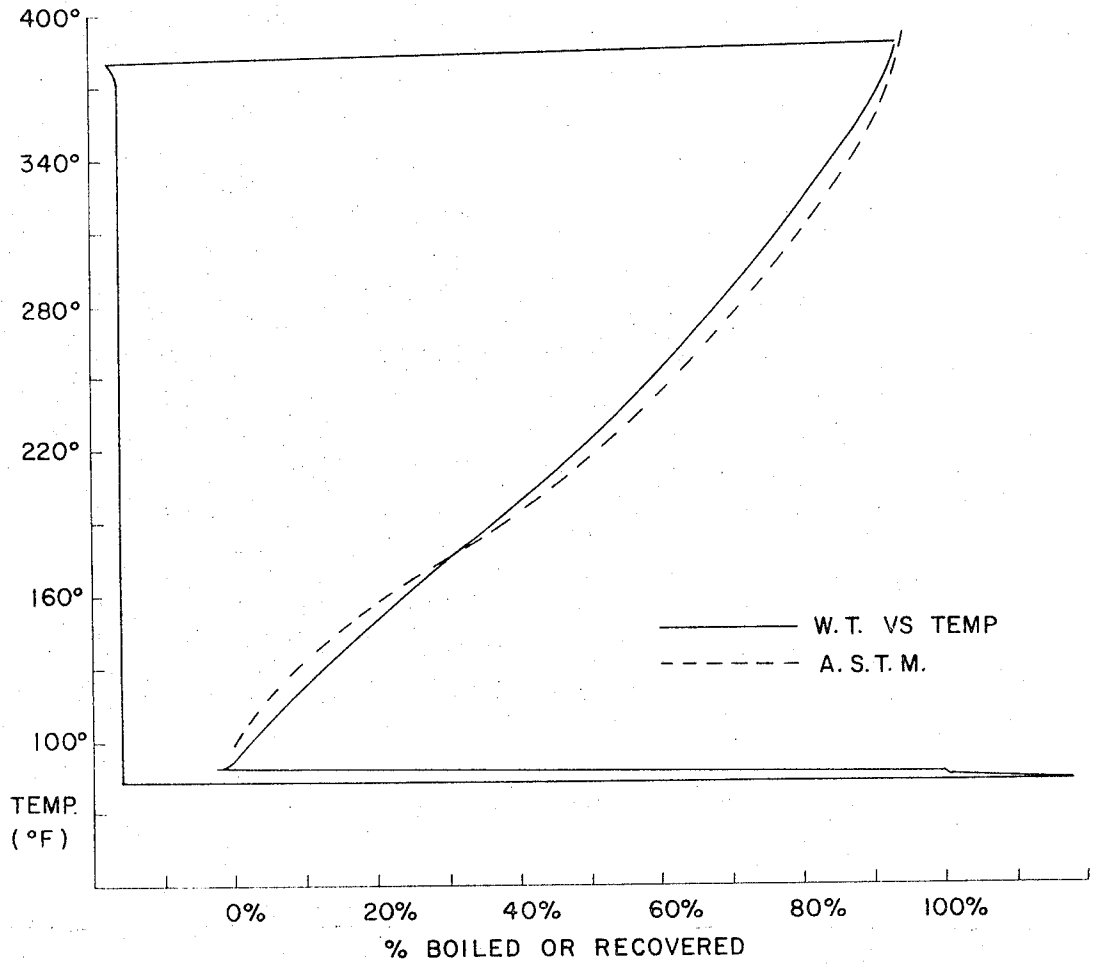


FIG. 5

INVENTORS:
A. D. GOOLSBY
B. D. STANTON
BY:
Harold L. Denkler
THEIR ATTORNEY

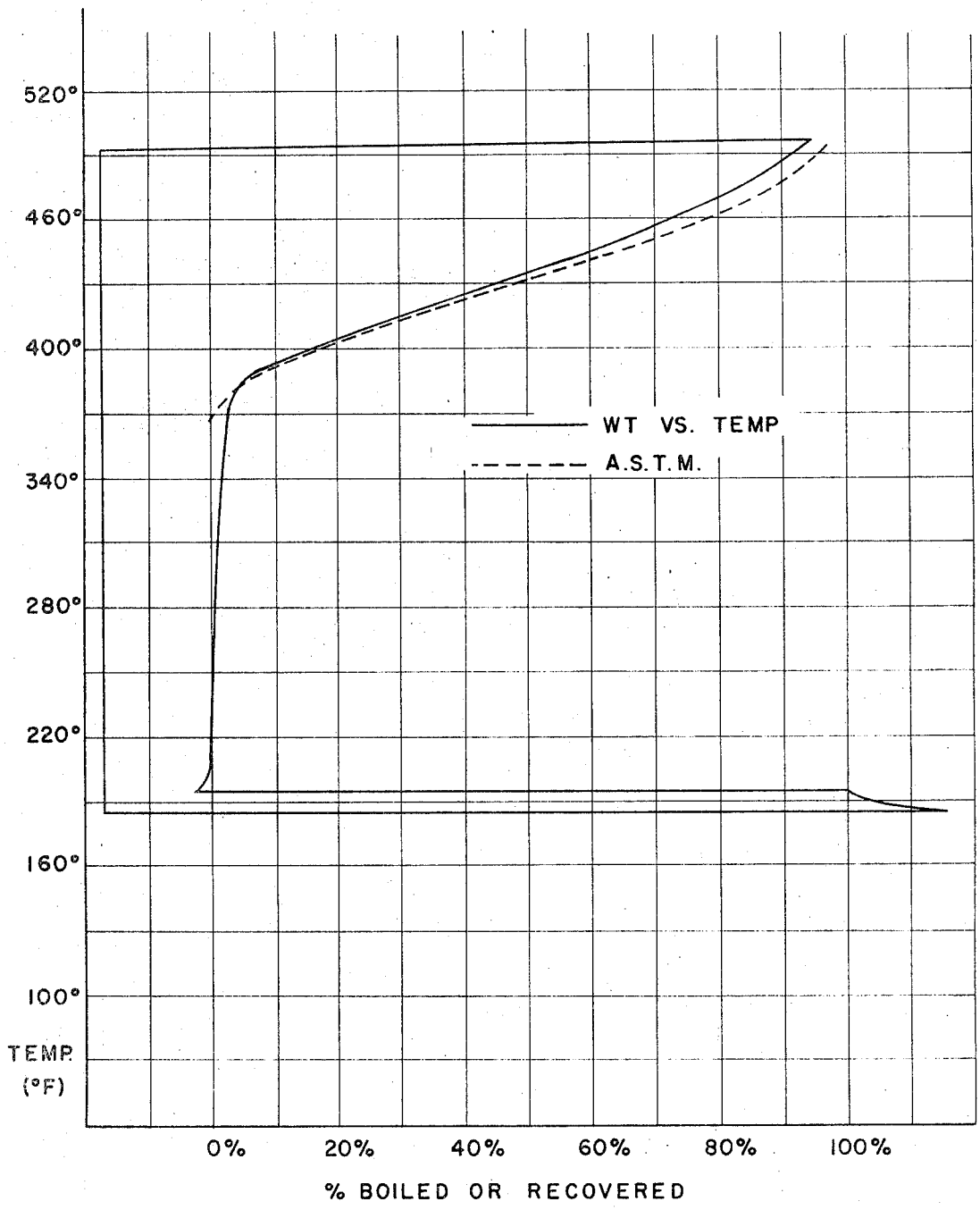


FIG. 6

INVENTORS:
A. D. GOOLSBY
B. D. STANTON
BY: Harold L Stinkler
THEIR ATTORNEY

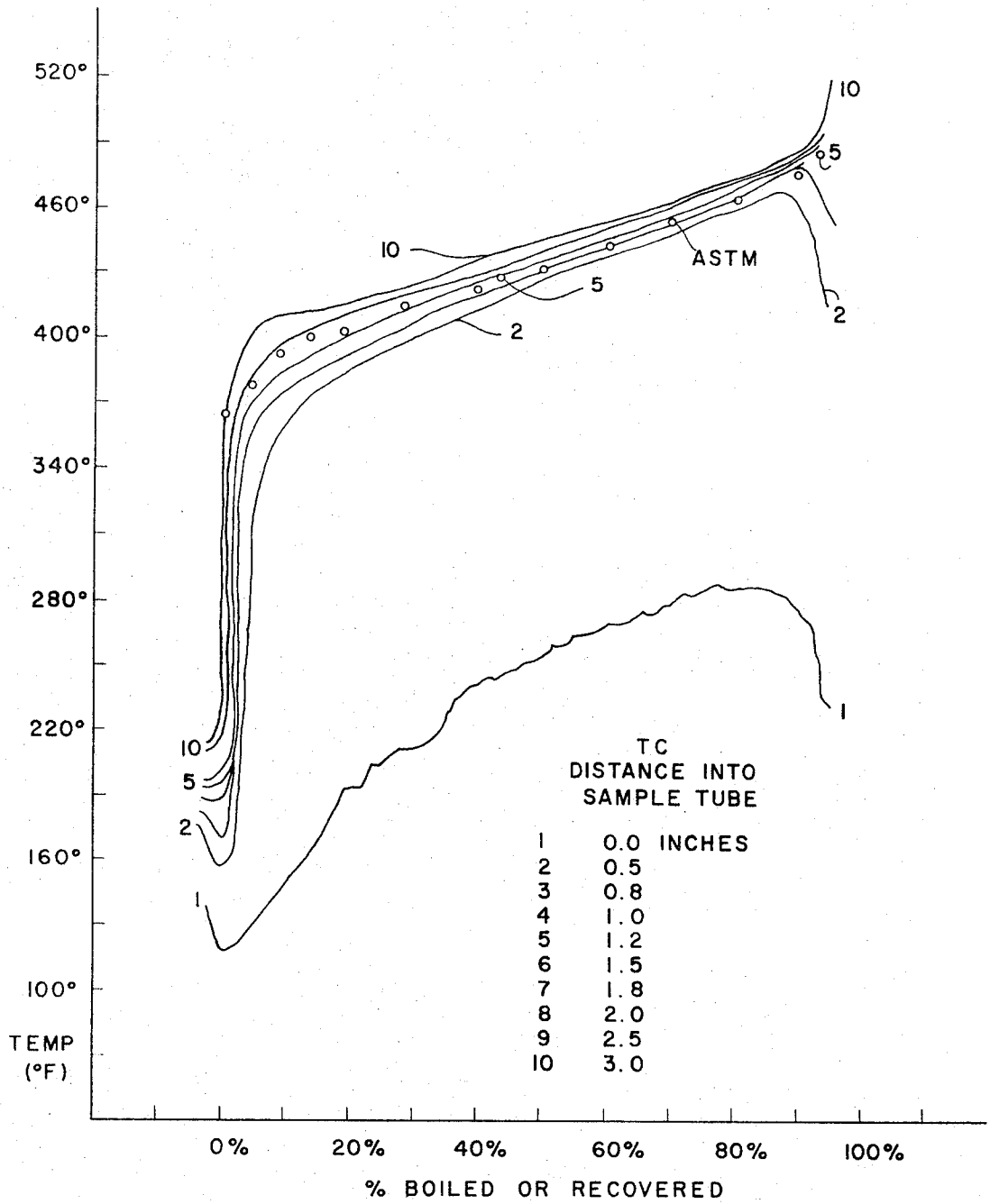


FIG. 7

INVENTORS:
A. D. GOOLSBY
B. D. STANTON
BY: Harold L Dinkler
THEIR ATTORNEY

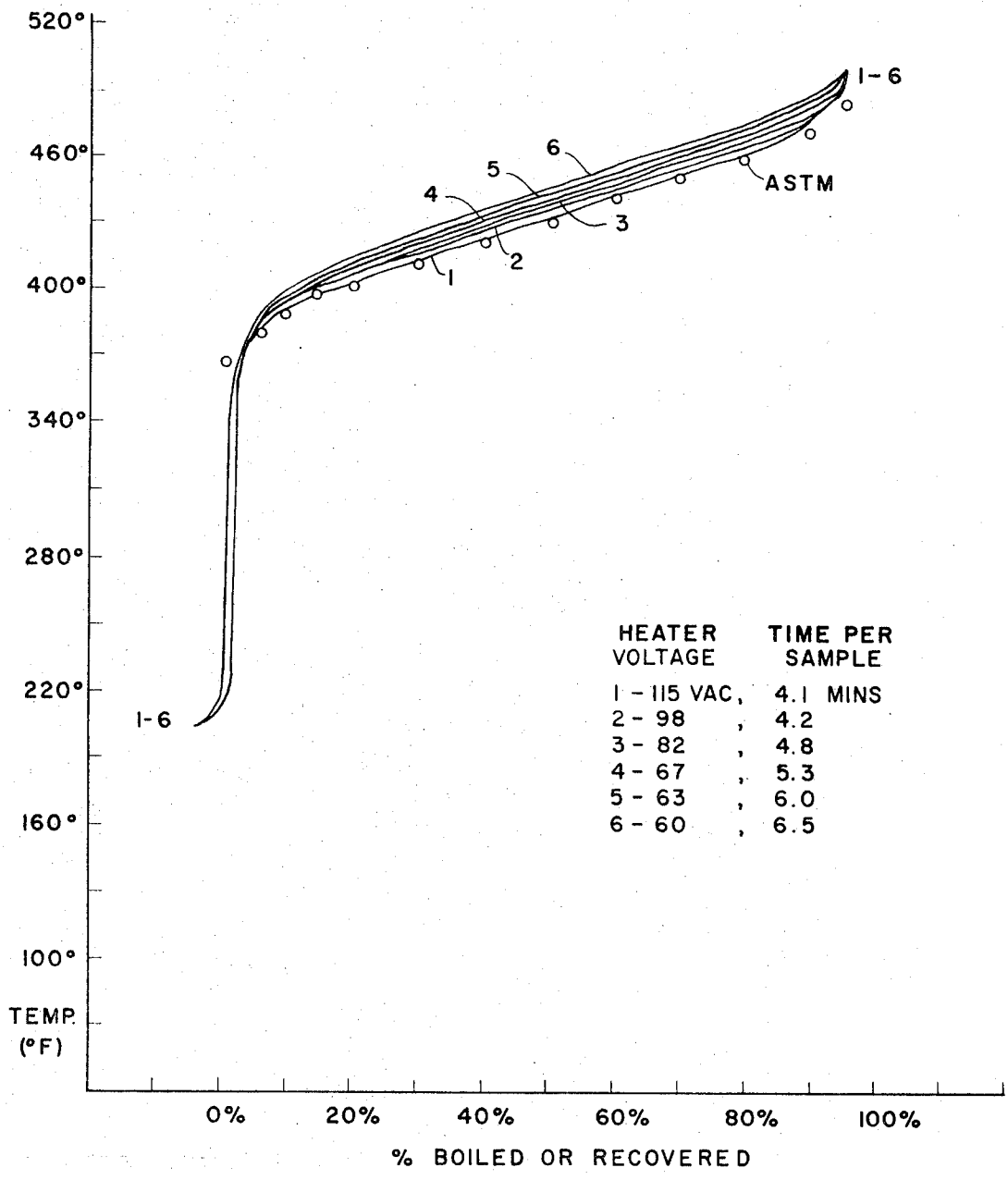


FIG. 8

INVENTORS:
A. D. GOOLSBY
B. D. STANTON
BY: Harold L. Simpler
THEIR ATTORNEY

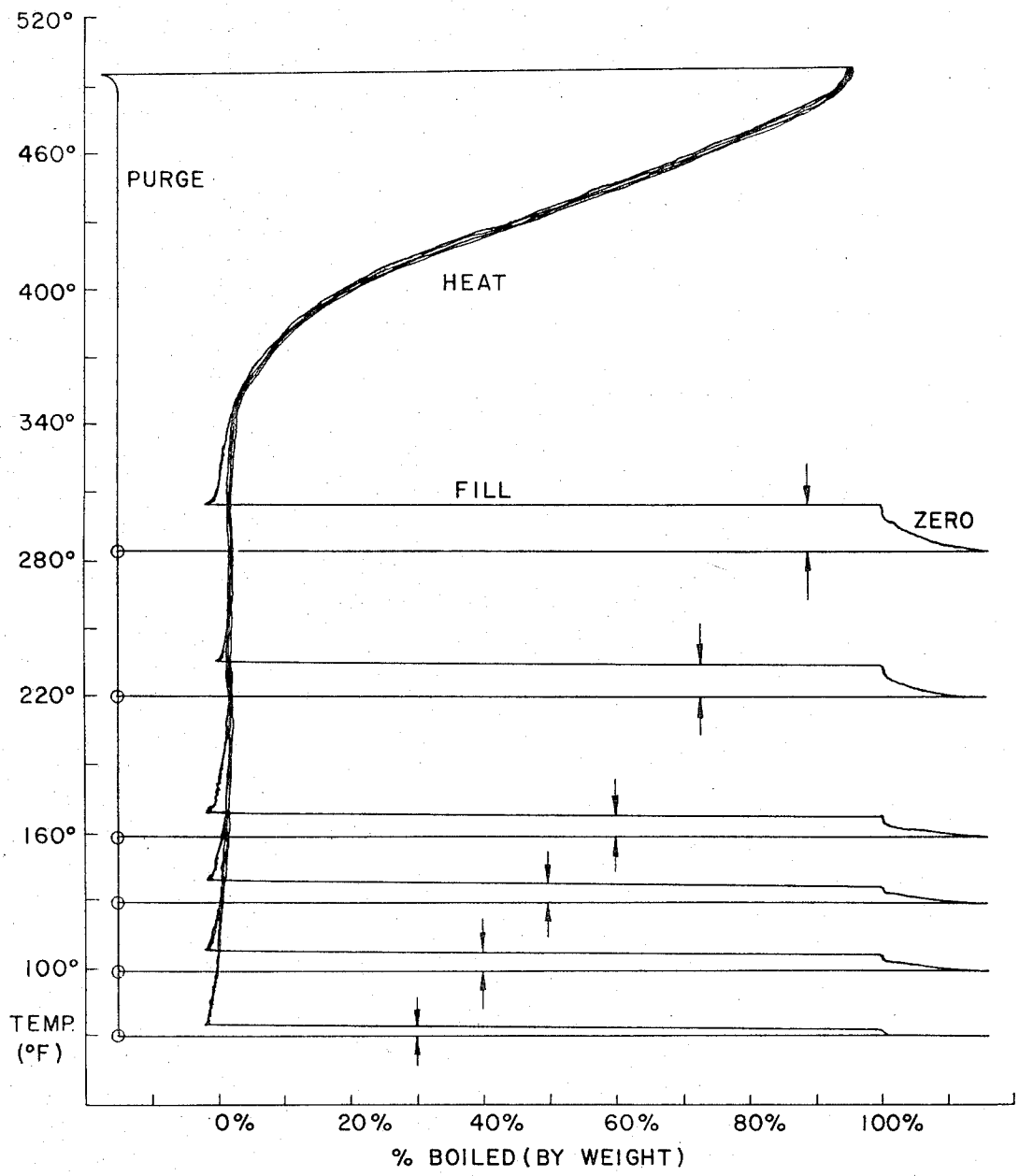


FIG. 9

INVENTORS:
A. D. GOOLSBY
B. D. STANTON
BY: Harold L Hinkle
THEIR ATTORNEY

APPARATUS FOR DETERMINING DISTILLATION CUT POINTS

BACKGROUND OF THE INVENTION

The present invention relates to a method and apparatus for determining the distillation cut and boiling points of materials and particularly liquids. The problem arises in all process industries, particularly chemical and petroleum industries, of determining the distillation cut and boiling points of liquid samples. In the past a recognized method known as an ASTM method (D86) has been developed for determining distillation cut points. This method comprises placing a measured amount of the liquid in a container and heating the liquid to vaporize it with the vapor being collected and condensed. The various cut points are determined by measuring the amount of liquid as it is condensed and recording the temperature. It requires a skilled operator and 45 minutes to perform a complete distillation analysis of a liquid by this method.

The results obtained with the ASTM method depend to a large extent on the skill of the operator and his attention to the process. Even when the operator is skilled and attentive the method still suffers from incomplete condensation of the sample. When unskilled operators are used the sample size, initial sample temperature and sample recovery may all vary to produce inaccuracies and variations in the results.

Various apparatus have been developed to automate or at least partially automate the above method to eliminate the dependence on the operator. These apparatus have comprised the boiling of a known amount of liquid and collecting the vapors to determine the various distillation cut points of the liquid. These methods while eliminating the need for a skilled operator are time consuming since they substantially duplicate the steps of the ASTM method. Also, the automated methods require equipment to condense and measure the quantity of condensed liquid to determine the various distillation cut-points.

In addition to the above problems with available apparatus they are substantially batched-type apparatus and relatively slow. The slow operation produces a time lag between taking of a sample and the obtaining of the distillation points. The time lag prevents the use of present systems with process controls since the information they supply is out-of-date and not related to the present state or operating levels of the process.

BRIEF DESCRIPTION OF THE INVENTION

The present invention solves the above problems by providing a method and apparatus which is completely automatic and provides a complete distillation analysis of the sample in a short time. The method measures the loss-in-weight of a sample of material as it is heated to determine the various percentage cut-points of the material. As the various percentage cut-points of the material are determined the temperature of the vapor which is being continuously measured, is recorded. The cut-points and temperature can be made to correspond closely to the cut-points obtained by the ASTM method. The analyzer can be adjusted to operate either as a cut-point analyzer or a boiling point analyzer.

The apparatus comprises a sample holder for holding a sample of material with the sample holder being attached to one end of a beam balance. A weight detector is positioned at the opposite end of the beam balance to measure the loss-in-weight of the material as it is heated. The apparatus includes controls for supplying a preset amount of sample to the sample holder and controlling the heating of the sample to obtain the desired rate of evaporation of the sample. After the sample is evaporated the sample holder is purged to both cool it and remove the remaining traces of the sample. The system is automatically zeroed before each new sample is placed in the sample holder. This is done to compensate for build-up of residue in various portions of the sample holder and low frequency drift of the electronic circuits.

The weighing means may be either a device which actually weighs the sample and sample holder or a device which determines the displacement of the end of the beam. The signals representing the temperature and the loss-in-weight of the sample can be recorded in correlation so that the various percentage cut-points and corresponding temperatures may be determined. These variables can be sampled at preset positions to give output data on specific cut-points and/or temperatures. The signals from the temperature and weighing means may also be supplied to an analog-to-ditigal converter for conversion to digital numbers. The digital numbers can then be supplied to a computer which will compute the various percentage cut-points and print out the corresponding temperatures. Of course, the computer can also be used to control the process utilizing the information from the apparatus.

The system requires a relatively short time to perform a complete distillation analysis, for example, a 10cc sample can be evaporated in approximately 5 minutes to obtain the same degree of accuracy that is obtained using a skilled operator and following the ASTM method. This time can be further reduced to a minimum of approximately 3 minutes if a smaller sample is used. When a small sample is used the accuracy is not as good as the ASTM method but is normally satisfactory for most control purposes.

The system can also be used directly to control a process. For example, when two streams of product from distillation columns are blended to form a finish product having a specified boiling or cut-point the system can monitor the column streams and control the columns. This is accomplished by supplying the sample holder with a ratio of the two column streams, the ratio being determined by the finished product. If the analyzer determines the ratio it does not supply a finished product having the desired characteristics, then its signal can be used to vary the column streams to obtain a more accurate ratio.

BRIEF DESCRIPTION OF THE DRAWINGS

The present invention will be more easily understood from the following detailed description of a preferred embodiment when taken in conjunction with the attached drawings in which:

FIG. 1 is an elevation view of an instrument constructed according to this invention;

FIG. 2 is a schematic diagram in block form of the control circuits of this invention;

FIG. 3 is a block diagram of the system used to control the blending of two column streams;

FIG. 4 is a curve of the distillation cut-points of a turbine fuel containing various additives;

FIG. 5 is a comparison of the cut-points for gasoline obtained using the processes of the invention with those obtained with the ASTM method;

FIG. 6 is a similar comparison for kerosene;

FIG. 7 illustrates the effect of thermocouple position on the cut-points for kerosene;

FIG. 8 illustrates the effect of heating rate on the cut-points for kerosene; and

FIG. 9 illustrates the effect of varying the temperature of the sample holder on the cut-points of kerosene.

DESCRIPTION OF THE PREFERRED EMBODIMENT

Referring to FIG. 1 there is shown an elevation view of a complete instrument constructed according to this invention. The instrument utilizes loss-in-weight to determine the percentage distillation cut-points of a liquid sample. A preset quantity of sample is placed in a sample holder **10** which is attached to one end of the beam **11.** The beam is flexurally pivoted at its center **13,** for example Bendix and Rex pivots, and the movement of the opposite end of the beam is measured by means of a linear displacement device **12** (LVDT).

The sample holder comprises a sample tube **20** which holds the liquid sample and a heating section or outer housing **21.** The heating section contains a suitable heating means, for example a resistance heater which is coupled by means of a lead **22** to a source of power. The leads **22** pass through the center of the beam and are taken out near the flexure pivot to eliminate the possibility of the leads creating a load on the beam and destroying the accuracy of the measurement.

The liquid sample is supplied through a line **30** to a solenoid valve **31** that controls the flow of the sample to the sample tube **20.** The outlet of the solenoid valve is coupled by means of a line **32** to the top of the sample tube. Also disposed in the top of the sample tube are thermocouple **33** and an outlet **41** for the purge line. All of the elements **32, 33** and **41** should be positioned so that they do not contact the sides of the sample tube or interfere with the free movement of the beam and the sample holder.

Purging air is supplied by means of a line **35** to a solenoid valve **36** that controls the flow of the purging air to the sample tube in response to signals received from the control system described below. The discharge from the purge control solenoid is coupled by means of a line **37** to a vortex tube **38** with the hot end **39** of the vortex tube discharging to the atmosphere while the cold discharge from the vortex tube is connected by a lead **40** to the purge inlet **41** in the top of the sample tube. The use of the vortex tube provides cold air for both purging and rapidly cooling the sample holder so that all samples may be run under the same conditions.

The vapors which are evolved from the sample as it is heated are swept from the top of the sample tube by means of a flow of air through the inlet opening **45** and the exhaust opening **46.** This flow of air may be supplied from the same external source as the purge air. The inlet **35** can be coupled directly to a line **47** that discharges from its open end **48** into the housing **54** containing the electronic circuits. The air escapes from the housing **54** at the right hand end and flows out the opening **45-46** to sweep the vapors from the housing. This air flow insures that vapors do not flow into the electronic circuits and at the same time cools the circuits. A counter-weight **50** is provided on beam **11** to counter-balance the weight of the sample holder. The placement of the counter-balance is not critical since the actual displacement of the beam is measured by means of the device **12.** An adjustable dashpot member **51** is coupled to the beam to limit or control sudden movements of the beam and for stabilizing the beam. The linear measuring device may be a linear displacement device that supplies an electrical signal proportional to the displacement of its core member **53** that is coupled to the end of the beam by a suitable clamp. A Schaevitz motion transmitter manufactured by Schaevitz Engineering of Pennsauken, N. J., may be used for the linear displacement measuring device. A drain **55** provides for disposal of sample resulting from overflow or leakage.

The control circuit for the apparatus is shown in FIG. 2 in a block diagram form. The control system utilizes a series of solenoid valves and relays **61-63** to control the flow of the sample to the sample tube, the flow of purging air for purging the sample tube and the flow of electrical current supplied to the heater element of the sample container. The position of the solenoid valves is controlled by a controller in response to various conditions existing in the device as explained below. The control system also utilizes a zero adjustment circuit **60** which adjusts the signal from the position-measuring device to a zero level after the sample holder has been purged. This compensates for residues remaining in the system, the build-up of foreign matter and low frequency drift of the circuits.

The linear motion transmitter is coupled to a loss-in-weight measuring circuit comprising an amplifier **66.** The zero adjustment circuit is also connected to the amplifier **66** to adjust the amplifier to a zero output after the sample tube has been purged. The thermocouple is coupled to a temperature measuring circuit also consisting of an amplifier **67.** The signals from amplifiers **66** and **67** representing the loss-in-weight of the sample and the temperature of the vapor are supplied to an X-Y recorder **68** that records the loss-in-weight of the sample in correlation with the temperature of the vapor. The two signals are also supplied to an analog-to-digital converter **70** whose output is supplied to a computer **71** programmed as set forth below so that it will supply a print-out indicating the percentage distillation cut-points and the corresponding temperatures as selected.

The controller for automatically controlling the operation cycle of the instrument is shown on the right of FIG. 2 and utilizes a series of operational amplifiers **80-83** coupled as OR gates and disposed in closed loop so that when any one conducts the remainder are latched out. In this type of circuit the output signal of one OR gate is used to latch out the remaining gates with the coupling to the next succeeding gate having a switch disposed in its circuit. For example, the output of OR gate **80** is coupled by a lead **84** to OR gates **82** and **83** and by a lead **85** to OR gate **81** with a switch **86** disposed between the take-off points of the two leads **84** and **85.** Thus, the OR gates **81-83** remained latched out as long as OR gate **80** is triggered or until relay switch **86** is opened. When relay switch **86** opens the OR gate **81** will be triggered since the output signal from OR gate **80** is no longer applied to the input of OR gate **81.** The remaining OR gates will remain latched out and OR gate **80** will be latched out when OR gate **81** is triggered. This circle of operation will continue as

the relay switches **86, 87, 88** and **89** are opened and closed. The opening of the relay switches is controlled by signals from various portions of the apparatus as explained below while the relay switches return to a normally closed position in the absence of a signal.

The relay switch **86** is controlled by output signal of the loss-in-weight circuit so that it opens whenever the weight of the sample reaches a preset high value. This is accomplished by a comparing circuit **90** that compares the weight signal with a preset value **91** and supplies a signal on lead **92** to open relay switch **86** when the weight of sample equals the preset value. In a similar manner comparing circuit **93** compares the weight signal with a preset value and supplies a signal on lead **94** to open relay switch **87** when the weight of sample reaches a preset low value. The temperature signal is supplied over a lead **95** to a comparing circuit **96** where the temperature is compared with a preset value **97.** The signal from the comparator **96** is supplied over lead **98** to open relay switch **88** when the temperature of the interior of the sample holder reaches a lower limit. The switch **89** is a time delay relay whose coil is coupled in the output lead of the OR gate **83.** The time delay is set sufficiently long to permit the weighing circuit to adjust to zero, for example 10 seconds. The output signals of the OR gates **80-83** are supplied over leads **90-93** to the fill solenoid **62,** heater relay **61,** purge solenoid **63** and zero adjustment circuit **60,** respectively The sequence of operation of the circuit is described below.

In addition to recording the temperature versus weight curve on the X-Y recorder **68** the temperature and weight signals are also supplied to a sample and hold circuit that records any preset cut-point or boiling point. The sample and hold circuit uses a comparator **100** that compares either the temperature or weight, for example the temperature as shown in FIG. **2,** with a preset value **101** to give the cut-point for the temperature. The output signal from the comparator is supplied to a monostable circuit **102** that closes the relay switch **103** to complete the input circuit to the amplifier **104.** The signal from the amplifier **104** can be recorded on the recorder **105** to give the cut-point for the preset temperature or supplied to a process controller **106.** The process controller can position the controls of a distillation column to control the column reflux and reboiling to supply the proper top and bottom products.

FIG. **3** illustrates the use of the apparatus to control the blending of two streams to produce a final product whose density and cut-points meet preset specifications. As an example the product may be gas turbine fuel blended from the top product of a column **100** and the product from a side stripper **101.** The bottom product from the column **100** is used as the feed for the side stripper. The top product is stored in a tank **104** while the product of the side stripper is stored in a tank **105.** While the storage tanks are desirable they can be eliminated if desired. The products in the two storage tanks are blended and supplied as turbine fuel through the pipeline **106.**

It is assumed that the specifications for the turbine fuel require that it have a density that falls between upper and lower limits and 10 percent and 90 percent cut-points that also fall within set limits. The density of the products from the two storage tanks are measured by a density measuring circuit **107.** The density measuring circuit **107** also computes the approximate quantity of each product and supplies voltage signals on leads **108** and **109** that can be used as set points for the fill controllers **110** and **111.** The fill controllers are identical to the controller **90** of FIG. **2** and control the operation of two OR gates in the controller **112.** An additional fill OR gate is used with the fill control **110** controlling one to supply a first percentage of the total sample and the fill control **111** controlling the second to supply the remainder of the sample. The controller **112** supplies signals over leads **113** and **114** to control the opening of the solenoid valves **115** and **116** that control the flow of two products into the sample holder. The controller **112** also determines the 10 percent and 90 percent cut-points of the sample using the same circuits as shown in FIG. **2.** The 10 percent cut-point can be processed by a controller **117** and supplied over a lead **118** to control the positioning of a valve **119** in the reflux line of the column **100.** The operation of distillation columns and various control systems are shown in the prior art, for example see U.S. Pat. No. 3,342,698. The information on the 10 percent cut-point can be used to control the reflux to vary the quantity of the light product supplied to the blend. The 90 percent cut-point can be used in a similar manner to control the quantity of the heavy product supplied to blend to vary the density of the blend.

The computer **71** may be programmed in any desired language, for example, a program language known as BASIC wherein the commands to the computer are supplied in a substantially conversational language. An example of this programming is set forth below:

List

```
 91    FOR K=1 TO 4
 92    CALL (4,K,Ø)
 93    NEXT K
 95    PRINT "STARTING"
 1ØØ   LET C=Ø
 1Ø1   LET D1=.5
 11Ø   LET K=Ø
 12Ø   CALL (7,Ø,P)
 121   IF P#Ø THEN 9999
 122   GOSUB 2ØØØ
 13Ø   CALL (4,1,Ø)
 131   IF K=2 THEN 15Ø
 132   IF K=4 THEN 15Ø
 14Ø   WAIT (1ØØØ)
 15Ø   GOTO 12Ø
 2ØØØ  LET C=C+1
 2Ø1Ø  REM 22ØØ=PURGE, 23ØØ=WAIT,
 24ØØ=FILL, 26ØØ=CHECK, 28ØØ=HEAT
 2Ø2Ø  IF K=0 THEN 22ØØ
 2Ø3Ø  IF K=1 THEN 23ØØ
 2Ø4Ø  IF K=2 THEN 24ØØ
 2Ø5Ø  IF K=3 THEN 26ØØ
 2Ø6Ø  IF K=4 THEN 28ØØ
 21ØØ  CALL (1,5,T)
 21Ø1  LET T=1ØØ+6ØØ*T
```

```
2102  RETURN
2110  CALL (1,1,W)
2112  RETURN
2200  CALL (4,2,1)
2210  GOSUB 2100
2220  CALL (1,3,T1)
2221  LET T1=T1*1000
2230  IF T<T1 THEN 2260
2240  RETURN
2260  CALL (4,2,0)
2270  LET K=1
2280  LET C1=C
2290  RETURN
2300  IF C>C1+D1 THEN 2350
2310  RETURN
2350  GOSUB 2110
2355  LET W1=W
2360  LET K=2
2365  IF W1>-.1 THEN 2375
2370  RETURN
2375  PRINT "CRUD BUILT UP"
2376  GOTO 9999
2380  RETURN
2400  CALL (4,3,1)
2420  GOSUB 2110
2340  IF W>W1+1 THEN 2450
2440  RETURN
2450  CALL (4,3,0)
2470  LET K=3
2480  LET C1=C
2490  RETURN
2600  CALL (4,3,0)
2615  GOSUB 2110
2618  IF C>C1+D1+1 THEN 2680
2620  IF C>C1+D1 THEN 2650
2630  RETURN
2650  LET W2=W
2670  RETURN
2680  IF (W-W2)/(W2-W1)>1.00000E-02
      THEN 2790
2684  LET K=4
2690  FOR S=1 TO 9
2700  LET W[S]=W2-(_e2-W1)*S/10
2710  NEXT S
2720  LET S=1
2730  RETURN
2790  PRINT "FILL DIDN'T TURN OFF"
2792  PRINT W;W1;W2
2799  RETURN
2800  CALL (4,4,1)
2810  GOSUB 2110
2815  IF S=10 THEN 2960
2820  IF W<W[S] THEN 2900
2830  RETURN
2900  LET W[S]=W
```



```
2910  LET W[S]=W
2920  GOSUB .2100
2930  LET T[S]=T
2940  LET S=S+1
2950  RETURN
2960  CALL (1,4,E1)
2968  IF W<W2-(W2-W1)*E1 THEN 2975
2970  RETURN
2975  LET K=0
2977  CALL (4,4,0)
2978  GOSUB 3000
2980  RETURN
3000  PRINT "#     % BOILED     TEMP"
3002  FOR S=1 TO 9
3005  LET W[S]=(W2-W[S]*100/(W2-W1)
3010  PRINT S;W[S];T[S]
3020  NEXT S
3025  PRINT "WEIGHTS";
3026  PRINT W2,W1
3027  PRINT "---------"
3030  RETURN
9999  END
```

READY

From the above program it can be seen that the first steps are to command the computer to recall from memory preprogrammed data and set various constants equal to various values. The computer is then told to let various constants perform various functions if various temperature and weight conditions exist in the system. The system is then operated until the steps for commanding the computer to print-out the various percentage boiling points when *w* equals a certain percentage of the initial weight. Thus, if all the steps up to 2800 have been satisfied the computer will commence to print-out the temperatures for the various percentage boiling points as they are reached. Also the signal from the computer indicating the temperature per percentage boiling point can be transmitted to a separate control system, not shown, in FIG. 1 to be used in a process control.

The following are four distillation cut-point analyses as measured by the instrument and computed by the computer following the above instructions.

#	% BOILED	TEMPERATURE	
1	10.0321	381.845	
2	20.209	396.493	
3	30.2904	408.789	
4	40.1336	421.094	
5	50.0243	432.227	#11
6	60.0104	442.773	
7	70.3781	453.32	
8	80.0291	463.574	
9	90.0142	477.344	
WEIGHTS	.595215		-.426743
#	% BOILED	TEMPERATURE	
1	10.1763	381.259	
2	20.018	396.786	
3	30.0038	408.496	
4	40.0383	421.094	
5	50.0244	432.812	#12
6	60.1064	443.066	
7	70.0435	453.32	
8	80.0773	463.867	
9	90.0143	479.687	

WEIGHTS	.585938		-.431137
#	% BOILED	TEMPERATURE	
1	10.1277	380.38	
2	20.1135	395.322	
3	30.0515	407.324	
4	40.0859	419.922	
5	50.0243	432.227	#13
6	60.2493	443.066	
7	70.2826	452.734	
8	80.0291	463.281	
9	90.1098	475.879	
WEIGHTS	.59668		-.425278
#	% BOILED	TEMPERATURE	
1	10.3733	381.259	
2	20.0757	395.029	
3	30.0662	407.91	
4	40.0575	419.629	
5	50.2886	432.52	#14
6	60.0392	443.945	
7	70.3653	453.613	
8	80.1157	464.453	
9	90.0095	477.93	
WEIGHTS	.585449		-.431137

OPERATION

As explained, the system operation is substantially automatic and once the quantity of sample to be used and the heating rate have been set no further adjustments are required. Of course, a supply of sample under suitable pressure, 5–100 psig and a source of compressed air for purging the device and ventilating air for removing the vapors as they are evolved from the sample are all essential. With necessary services coupled the instrument can be started by the controller. The first step performed by the controller is the zeroing of the sample holder. This can be done by applying a temporary large voltage signal to the heating circuit which trigger the OR circuit **81.** When the large voltage signal is removed the relay switch **87** will open since the sample holder is empty. The opening of the relay switch **87** will trigger purge OR gate **82** and the relay switch **88** will open when the purge air has cooled the sample holder. When the relay switch **88** opens the zero OR gate **83** will trigger and zero the loss in weight amplifier **66.** When the time delay relay **89** times out the switch **89** will open and trigger the fill OR gate **80.** The quantity of sample supplied depends on the level of the set point **91** that controls the time that OR gate **80** remains triggered. After the sample has been supplied the relay switch **86** will open and trigger the heating OR gate **81** to supply a predetermined voltage to the heating element. By selecting the voltage in relation to the quantity of sample supplied one can obtain the desired heating rate of the sample. The sample will then be evolved as the weight of the sample is continuously measured by the linear displacement device **12.** In the sample system the weight of the sample will be recorded in relation to the temperature of the sample and various percentage cut-points can be determined by either inspecting the record or the sample and hold circuit. After the sample has been evaporated the relay switch **87** will open and trigger the purge OR gate **82** to open the purge solenoid and supply purge air to the system. When the temperature of the sample holder (as measured by the thermocouple) reaches a preset level the relay switch **88** opens closing the purge solenoid and triggering the zero OR gate **83.** The system will repeat the above sequence of operation and supply distillation analysis of the sample stream connected to the system.

The sample stream connected to the system may be a stream from a process or may consist of previously collected samples which are supplied individually to the system. In the case of a process suitable timing devices can be coupled to the instrument so that it only analyzes the sample at set time intervals or the instrument can be set to continuously analyze the process to provide data for controlling the process.

FIG. 4 shows the distillation cut-points for a turbine fuel that is diluted with various percentages of impurities. From FIG. 3 it is seen that if the turbine fuel is blended with a heavy gas oil the temperature of the higher cut-points is raised while if the turbine fuel is diluted with a lighter hydrocarbon as for example, gasoline, its initial cut-points are lower. Thus, the instrument responds to compositional changes in the same manner as the ASTM method.

FIGS. 5 and 6 show a comparison between the cut-points obtained using the present instrument and the ASTM method where the amount of liquid evaporated is measured and the temperatures at which it evaporated recorded. From a comparison of these two chart records it is seen that the present instrument provides results which are within approximately five percent of the ASTM recordings. This is sufficiently close for all practical purposes and can be used in control processes.

Referring to FIG. 7 there is shown a comparison of results obtained using various distances between the thermocouple and the top of the sample tube. Also shown is an ASTM distillation analysis for the same sample. From the information shown it can be seen that if the thermocouple projects approximately 1.2 inches into the same tube wherein the sample tube comprises a tube of 1.1 inch diameter and 4.5 inch length containing 10 cc. of sample; the results obtained will be substantially equal to the ASTM method. The same results would be obtained regardless of the size of the sample tube provided the thermocouple projects into the tube at least a distance equal to approximately 25 percent of the overall length of the tube. This insures that the temperature measured by the thermocouple is substantially the true temperature of the vapor evolved from the sample.

Referring to FIG. 8 there is shown a comparison between an ASTM distillation curve and the data obtained with the apparatus of this invention utilizing various heating rates. From the data it can be seen that if the heating rate is set to completely evaporate a 10 cc. of sample in approximately four minutes the results substantially equal the results obtained on the ASTM method.

Referring now to FIG. 9 there is shown a comparison between the temperature at which the purging air was shut off and the filling of the sample tube commenced. It can be seen that regardless of the temperature at which the purging air was shut off and the sample tube filled one obtained substantially the same distillation curve. Thus, the final temperature of the sample tube after purging is not critical and can vary over a relatively wide range.

We claim as our invention:

1. An apparatus for automatically conducting distillation analysis of liquids comprising:

a sample tube;

a support beam flexurally mounted at a point between its ends, said sample tube being attached to one end of said beam;

heating means disposed to heat said sample tube to vaporize a liquid sample disposed therein;

purge means disposed to purge said sample tube after said liquid sample is vaporized;

temperature measuring means disposed to measure the temperature of the vapor evolved from said sample tube and supply a signal related thereto;

weighing means disposed to determine the weight of the sample tube and sample contained therein and supply a signal related thereto;

fill means disposed to supply a sample of said liquid to said sample tube;

a control circuit, said control circuit being responsive to said temperature measuring means and said weighing means to operate said fill means to fill said sample tube with a preset quantity of a liquid sample, energize said heating means, energize said purge means after said liquid sample is vaporized and adjust said weighing means for the weight of the sample tube after said sample is vaporized; and

recording means, said temperature measuring means and said weighing means being coupled to said recording means to record said related signals in a correlatable manner.

2. The apparatus of claim **1** wherein said weighing means comprises linear displacement measuring means disposed to measure the movement of said beam as the sample is vaporized.

3. The apparatus of claim **1** wherein said weighing means comprises a strain gauge weigh cell disposed to weigh said sample tube and sample.

4. The apparatus of claim **1** wherein said control circuit includes means for setting said pre-set quantity of sample supplied to said sample tube.

5. The apparatus of claim **1** wherein one of said temperature measuring means and weighing means is coupled to comparing circuit means, said comparing circuit means comparing the temperature or weight of the sample with a set joint and supplying the other of said temperature or weight measurements of the sample to a process control means.

* * * * *