April 29, 1952 F. B. ROLFSON 2,594,683

BOILING-POINT APPARATUS

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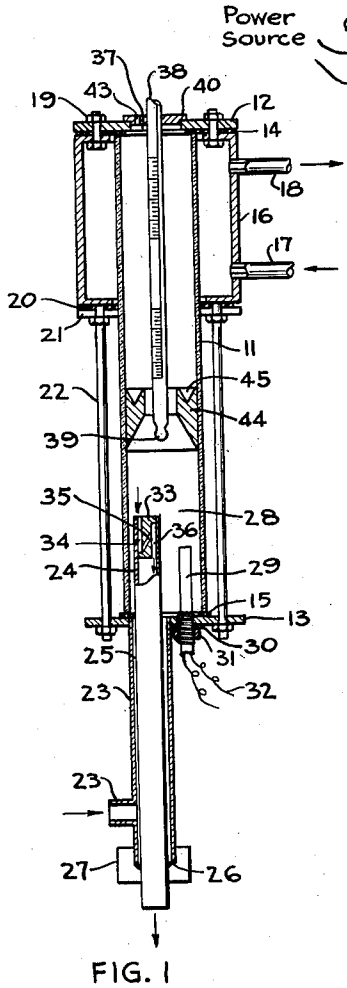


FIG. 1

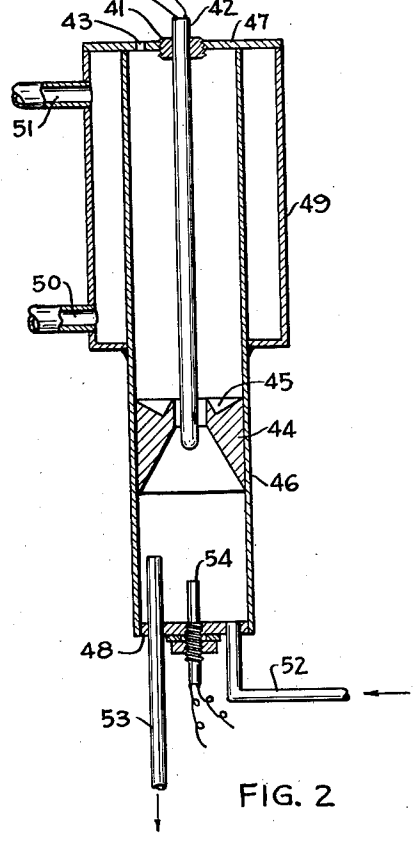


FIG. 2

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BOILING-POINT APPARATUS

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5 Claims. (Cl. 73—17)

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This invention relates to an apparatus for determining the boiling point temperature of a liquid mixture, and pertains more particularly to an apparatus for automatically and continuously determining the initial boiling point temperature of a hydrocarbon mixture, or the distillation temperature of any fraction of said mixture.

For simplicity, the present invention will be described with reference to the distillation of petroleum products, it being understood that it applies equally well to the distillation of any mixture of liquid fractions or components having different boiling points, said fractions or components being of any origin whatsoever.

During the refining or distillation of petroleum, various products such as gasoline, kerosene, naphtha, etc., may be obtained. The refining operations are normally carried out in such a manner that one or more of the resultant petroleum products meet certain boiling point temperature specifications which are generally determined by a standard ASTM distillation test. In the ASTM distillation test, a 100 ml. sample of the petroleum product is distilled through standard test equipment in a prescribed manner, and the initial boiling point of the product is observed and recorded, as well as the temperatures reached when subsequent 10 ml. or 10% portions of the product have been distilled off.

Throughout the manufacturing process of a petroleum product a rough test may be made for control purposes by running initial boiling point tests intermittently on portions of the fluid taken from the product flow stream. Since it has been found that the initial boiling point temperatures of different portions of the same product may vary greatly while the overall analysis of the product may remain substantially constant, a better test for control purposes is known as the "10% boiling point test" which comprises determining the temperature of a boiling sample of the product under test when 10 per cent (or 10 ml. of a 100 ml. sample) has been distilled off.

It is therefore the primary object of this invention to provide an apparatus for continuously distilling a portion of a product flow stream whereby the initial boiling point, the 10% boiling point, or the boiling point of any fraction of said product may be readily determined.

It is also an object of this invention to provide an apparatus for continuously and automatically determining and recording any changes in a boiling point of the liquid in a product flow stream from a petroleum distillation unit whereby the

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output from said unit may be controlled by said apparatus to deliver a substantially uniform product.

These and other objects of this invention will be understood from the following description of the invention as shown in the accompanying drawing, wherein:

Figures 1 and 2 are views, partly in cross section, of two embodiments of the present apparatus.

Referring to Figure 1 of the drawing, the present apparatus comprises a vertically-positioned tubular boiler or distillation chamber 11 having its upper and lower ends closed in a fluidtight manner by plate members 12 and 13, respectively. If necessary, gaskets 14 and 15 may be interposed between said plates 12 and 13 and the ends of said boiler 11 to form a fluidtight seal. The tubular boiler 11 may be made of a thin metallic material but is preferably made of glass or a suitable transparent and heat-resistant plastic. Continued boiling of a petroleum product within the boiler 11 usually results in the deposition of gums or other residues on the inner wall of the boiler which may affect the accurate operation of the apparatus. If a transparent boiler 11 is used, any gum deposition may be readily noted and the boiler may then be cleaned.

The upper portion of the tubular boiler 11 is surrounded by a condenser 16 having inlet and outlet ports 17 and 18 respectively for receiving and discharging cooling water. The condenser may be secured at its upper end to plate 12 by bolts 19, the gasket 14 forming a fluidtight seal. Another gasket 20 between the lower end of the condenser 16 and a ring member 21 forms a fluidtight seal between the boiler 11 and condenser 16 at the bottom of the latter. The lower end of the condenser 16 is rigidly secured to plate 13 by threaded holding rods 22.

Fixedly attached to the bottom plate 13 in any suitable manner, as by screw threads, welding, or the like, is a fluid inlet conduit 23 in communication with the space within the lower end of the boiler 11. A fluid discharge conduit 24 also extends through the lower plate member 13 and into the boiler 11 to a predetermined height whereby the upper open end of said discharge conduit 24 acts as an overflow standpipe and serves to determine the level of liquid within the boiler. Preferably, the discharge conduit 24 is mounted in an adjustable manner whereby the height of the open upper end thereof may be changed as desired.

While the intake and discharge conduits 23 and 24, respectively, may be mounted in any manner desired, the outside diameter of the discharge conduit 24 is preferably substantially smaller than the inside diameter of the intake conduit whereby said conduits 23 and 24 may be arranged concentrically with an annular space 25 being formed therebetween. By such an arrangement a suitable heat exchange unit is provided whereby the heat given off by the effluent fluid passing through conduit 24 is utilized to pre-heat the incoming fluid passing upward in the annular space 25. The lower end of the heat exchanger may be closed by welding the inlet and outlet conduits 23 and 24 at a point 26, or by otherwise closing the annular space 25 by a suitable pressure seal diagrammatically shown at 27.

Positioned within the lower end of chamber 28 within the boiler 11 is a suitable heater for heating a fluid sample within the boiler. While suitable steam, gas, electric, or other types of heaters may be employed as heating means, either inside or outside of the present boiler, preferably a small electric heater is used such, for example, as a 50-watt cartridge-type heater 29. The heater 29 may be threadedly mounted in the bottom plate 13 of the boiler 11, and sealed in fluidtight engagement by a gasket 30 and nut 31. The heater is preferably positioned so that the major portion thereof is within the boiler 11. Electrical leads 32 connect the heater with a suitable power source.

To prevent the escape of vapors from the boiler 11 out the discharge line 24 thereof, a suitable trap is provided in the discharge line 24. The trap may be of any desired design and may simply comprise a metallic plug 33 having a tortuous channel 34, 35 and 36 therethrough. The liquid is trapped in channel 34 and prevents the passage of vapors down the discharge conduit 24.

The plate 12 covering the top of the boiler 11 has a central opening 37 through which a temperature sensing device, for example, a thermocouple unit or a mercury-bulb thermometer 38, may be inserted for determining the boiling point temperature of said boiler 11. The temperature-sensing device 38 may be supported within the opening 37 in any suitable manner with its lower end 39 at a predetermined height above the upper end of the discharge conduit 24. The thermometer support means 40 may comprise a disk or stopper of rubber, cork, glass, ceramic, metal or any other insulating or non-insulating material. As shown in Figure 2, the support means may comprise a metal collar 41 press-fitted on a thermocouple unit 42, said collar 41 being externally threaded so as to fit in screw-threaded engagement in the top of the boiler. Vent means 43 may be provided in the support means 40 or the top boiler plate 12 for preventing any pressure build-up within the boiler 11 and for releasing any non-condensable vapors which may accumulate within said boiler, said non-condensable vapors being present in negligible amounts and being released in accordance with ASTM procedure.

The choice of a support means 40 made of a thermally insulating or a non-insulating material depends upon the boiling point temperature of the liquid to be measured by the apparatus, i. e., whether the apparatus is arranged to determine the initial boiling point of the liquid or whether it is arranged to determine the temperature after a certain volume (say, 10 or 20 per cent) has been distilled off. In determining a 10 or 20 per cent boiling point temperature, a support means 40 made of heat-insulating material is preferably employed so that there is no heat conducting path between the condenser 16 and the thermometer 38 whereby the condenser 16 would tend to cool the stem of the thermometer 38. This cooling effect of the condenser 16 on the thermometer 38 which causes a definite lag in the thermometer is desirable, however, when the present apparatus is employed to determine continuously the initial boiling point of a liquid. In determining the initial boiling point of a liquid by an ASTM distillation apparatus, the boiling point is observed on a rising mercury column of the thermometer. Therefore, in order to compensate for the lag in the rise of the mercury column in a thermometer in running a single boiling point determination, it is desirable to provide the thermometer of the present apparatus with a permanent depression of temperature when making a continuous determination of the initial boiling point. It has been found that the condenser 16 of the present invention can furnish suitable depression to the temperature sensing means when a metal support means 41 (Fig. 2) is employed for transmitting the cooling effect of the condenser to the thermocouple 42.

When the present apparatus is utilized to determine continuously a boiling point temperature of a fluid sample in the bottom of the boiler 11 other than the initial boiling point, a suitable condensate or reflux trap 44 (Fig. 1) is mounted within the boiler 11 on the inner wall thereof at a point above the liquid level in the boiler. The trap 44 has the form of an internal annular flange or collar provided with a trough or groove 45 in the upper face thereof containing a quantity of liquid. An axial opening 46 through the center of the trap 44 permits the insertion of a thermometer bulb 39 therethrough and provides conduit means for returning the overflow liquid from the trap to the bottom of the boiler 11.

The trap 44 is designed so that its trough 45 contains a predetermined amount of liquid. The purpose of the trap 44, when used in an apparatus for determining the 10 per cent boiling point temperature, is to withhold substantially 10% of the fluid sample in the upper portion of the boiler while the other 90% of the sample is in the lower portion of the boiler 11. Since it has been found that up to 2% or more of a liquid sample may be flowing down the inside wall of the boiler 11 at all times, the trap 44 is designed with a capacity to hold about 8% of the liquid sample under test. The liquid capacity of the trough 45 may be readily varied by adding small pieces of material thereto, for example glass beads, or removing said material therefrom, whereby the apparatus may be readily changed to determine whatever boiling point temperature is desired, e. g., 5%, 10%, 15%, 20%, etc. boiling point. At the same time, if it is desired to use only a 100 ml. sample of liquid in making different boiling point determinations in accordance with ASTM procedure, the height of the discharge conduit draw-off 24 within the boiler must be raised or lowered to allow only 95%, 90%, 85% etc. of the sample under test (say, 100 ml.) to remain in the bottom of the boiler 11. The reflux trap 44 may be secured in the boiler 11 in any desired manner but is preferably press-fitted therein whereby it may be readily removed. It is realized that the reflux trap 44 may be of any

form desired. For example, as an extreme, a coil spring of suitable length and size inserted coaxially in said boiler tube against the inner wall thereof may be used to hold 10% of the liquid sample in the upper portion of the boiler.

In the embodiment of the present apparatus illustrated in Figure 2, the boiler 46 comprises a thin metal tube closed at the top and bottom by plates 47 and 48 which are welded thereto. A condenser 49, having inlet and outlet ports 50 and 51, respectively, is also welded around the upper portion of said boiler 46. Inlet and discharge conduits 52 and 53, respectively, are rigidly secured to the lower plate 48 of the boiler 46 for continuously introducing to and discharging from said boiler a liquid sample. In this embodiment all the heat is furnished to the liquid sample by a cartridge-type electric heater 54 threadedly secured to the bottom plate 48 of the boiler. If it is desired to use this embodiment in the form of an initial-boiling point apparatus, the reflux trap 44 would be removed from the boiler 46. The thermocouple 42 of the present embodiment is connected by leads 55 and 56 to a recorder or recorder-controller 57 which may be adapted to control the output current passing through leads 58 and 59 to control an electrically-operated valve 60 in a flow line 61 whereby the fluid flow to or from a distillation column (not shown) may be automatically regulated as any change is detected in the boiling point of the liquid being determined by the present apparatus. Likewise, the recorder-controller 57 may be connected to control the amount of heat being supplied to a distillation column, or any other piece of equipment employed. The recorder-controller 57 could obtain power from any suitable power source, not shown, through leads 62 and 63.

The heater 29 provides a continuous boilup of about 5% (e. g., 5 ml. per min. for a 100 ml. sample) which is totally refluxed by the condenser 16. The refluxed liquid on flowing down the inner wall of the boiler 11 is caught in the trap 44, and once the trap is filled, the incoming liquid mixes with the liquid already there with any excess liquid overflowing the trap and returning to the bottom of the boiler.

The temperature of the 10% boiling point may be observed on the graduated stem of the thermometer 30, or on the recorder-controller 57 connected to the thermocouple 42 in the embodiment shown in Figure 2. In tests of several different liquid having 10% boiling points varying from 250° F. to 423° F., all temperatures determined by the present apparatus come within 2° F. of the results obtained on an ASTM distillation apparatus.

I claim as my invention:

1. An apparatus for continuously determining the boiling point of a predetermined fraction of a liquid mixture having a varying boiling point, said apparatus comprising a distillation chamber, inlet conduit means opening to said chamber for introducing a continuous stream of liquid mixture thereinto, outlet conduit means opening to said chamber at a predetermined height for discharging liquid therefrom and thereby maintaining the liquid level in said chamber at a predetermined level, means for heating the lower portion of said chamber to vaporize the lighter components of said liquid mixture, a condenser for cooling the upper portion of said chamber to condense the vapors of said lighter components, trap means secured to the inner wall of said chamber above the liquid level therein for collecting said condensed vapors, the capacity volume of said trap means bearing a predetermined ratio to the volume of the liquid standing in said chamber, and temperature-sensing means carried within said chamber below the level of said trap means for determining continuously the temperature of the vapor therein.

2. An apparatus for continuously determining the boiling point of a predetermined fraction of a liquid having components of different boiling points, said apparatus comprising an upright cylindrical boiler, fluid inlet port means opening to the lower portion of said boiler for introducing into said boiler a continuous stream of liquid, standpipe liquid discharge means in said boiler for maintaining the liquid therein at a predetermined level, heater means carried within the lower portion of said boiler for vaporizing the lighter components of said liquid, a condenser surrounding the upper portion of said boiler for condensing the vapors of said lighter components on the inner wall of said boiler, trap means carried on the inner wall of said boiler above said liquid sample therein for collecting a predetermined portion of said condensed vapors, temperature-sensing means mounted within said boiler, said means extending through said trap means axially thereof to a point below the level of said trap means for continuously determining the temperature of said vapors, and means at the top of said boiler for venting said boiler to the atmosphere.

3. An apparatus for continuously determining the boiling point of a predetermined fraction of a liquid mixture having varying boiling points, said apparatus comprising an upright cylindrical distillation chamber, inlet conduit means opening to said chamber for introducing a continuous stream of liquid thereinto, adjustable height discharge conduit means opening to said chamber at a predetermined height for discharging liquid therefrom and thereby maintaining the liquid level in said chamber at a predetermined level, means for heating the lower portion of said chamber to vaporize the lighter components of said sample, a condenser for cooling the upper portion of said chamber to condense the vapors of said lighter components, annular liquid trap means secured to the inner wall of said chamber above the liquid sample therein for collecting said condensed vapors, the capacity volume of said trap means bearing a predetermined ratio to the volume of the liquid standing in said chamber, thermocouple means carried within said chamber for determining continuously the temperature of the vapor therein below the level of said trap means, and recorder-controller means electrically connected to said thermocouple means for receiving the output signal therefrom.

4. An apparatus for continuously determining the boiling point of a predetermined fraction of a liquid having components of different boiling points, said apparatus comprising an upright cylindrical transparent boiler, fluid inlet port means opening to the lower portion of said boiler for introducing into said boiler a continuous stream of liquid, standpipe liquid discharge means in said boiler for maintaining the liquid therein at a predetermined level, heater means carried within the lower portion of said boiler for vaporizing the lighter components of said liquid, a condenser surrounding the upper portion of said boiler for condensing the vapors of said lighter components on the inner wall of said boiler, an annular trough carried on the inner

wall of said boiler above said liquid sample therein for collecting a predetermined portion of said condensed vapors, temperature-sensing means mounted within said boiler, said means extending downward through said annular trough axially thereof to a point below the level of said trough for continuously determining the temperature of said vapors, and means at the top of said boiler for venting said boiler to the atmosphere.

5. An apparatus for continuously determining a boiling point of a predetermined fraction of a liquid having components of different boiling points, said apparatus comprising an upright cylindrical boiler, fluid inlet port means opening to the lower portion of said boiler for introducing into said boiler a continuous stream of liquid, heater means carried within the lower portion of said boiler for vaporizing the lighter components of said liquid, a condenser surrounding the upper portion of said boiler for condensing the vapors of said lighter components on the inner wall of said boiler, liquid trap means carried on the inner wall of said boiler above said liquid sample therein for collecting a predetermined portion of said condensed vapors, temperature-sensing means mounted within said boiler, said means extending downwardly through said liquid trap means to a point below the level of said trap means for continuously determining the temperature of said vapors, a liquid discharge standpipe in said boiler for maintaining the liquid therein at a predetermined level, and vapor trap means in said standpipe for preventing the escape of vapors therethrough.

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