Dec. 27, 1960 J. H. TRACHT ET AL 2,966,055

VARIABLE VOLUME CELL

Filed July 30, 1956 3 Sheets-Sheet 1

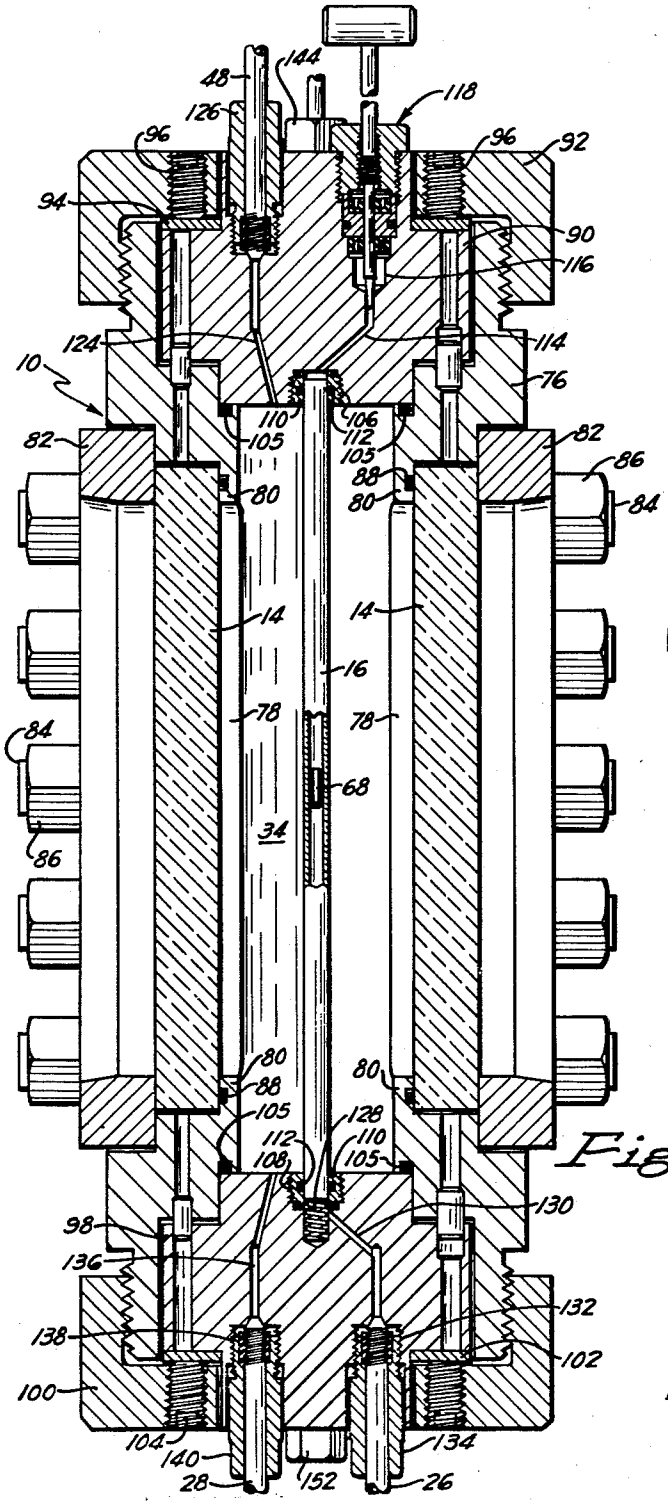


Fig. 2

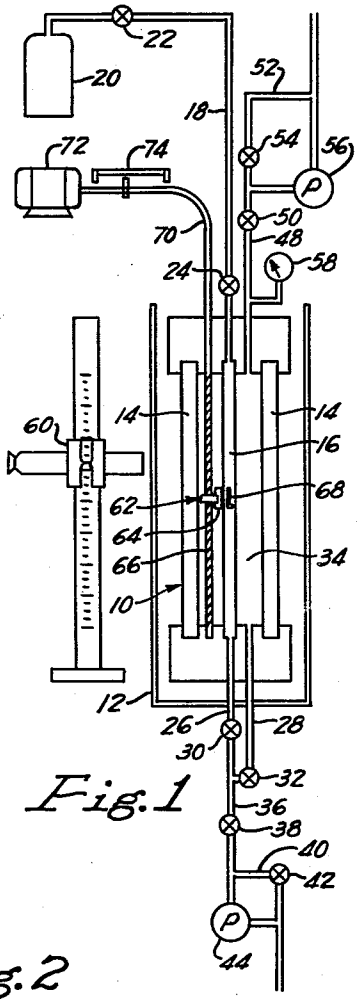


Fig. 1

INVENTORS
JOSEPH H. TRACHT
JOHN K. RODGERS
BY
Arthur Crooke
THEIR ATTORNEY

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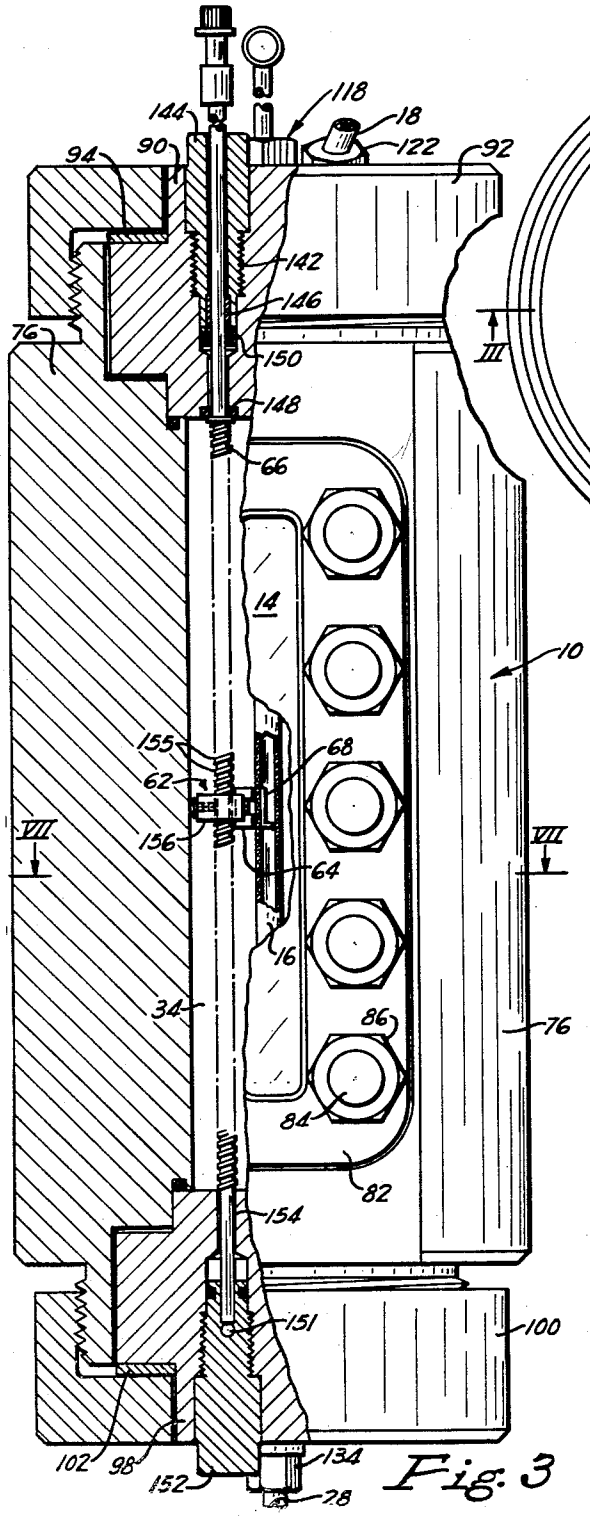


Fig. 3

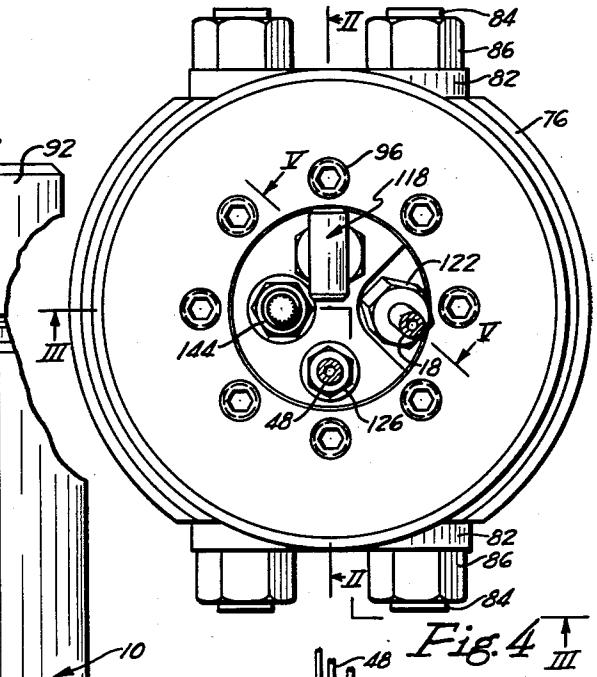


Fig. 4

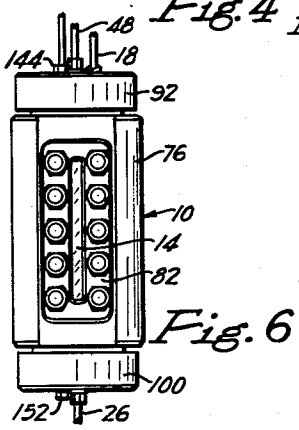


Fig. 6

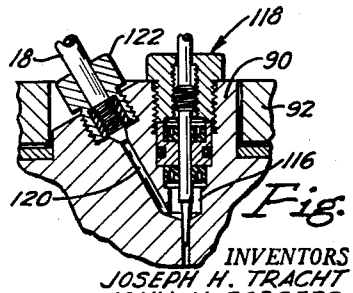


Fig. 5

INVENTORS
JOSEPH H. TRACHT
JOHN K. RODGERS

BY

THEIR ATTORNEY

Dec. 27, 1960 J. H. TRACHT ET AL 2,966,055

VARIABLE VOLUME CELL

Filed July 30, 1956 3 Sheets-Sheet 3

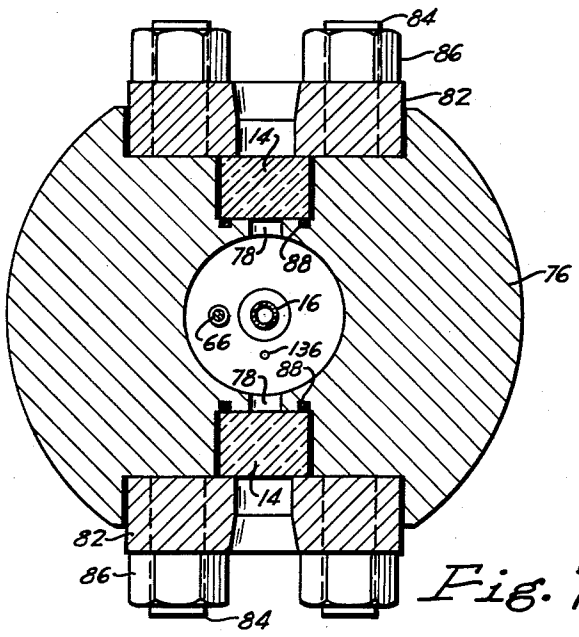


Fig. 7

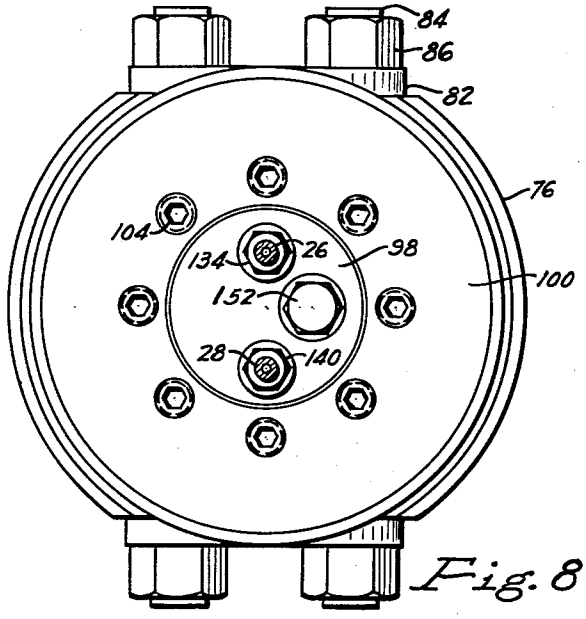


Fig. 8

INVENTORS
JOSEPH H. TRACHT
JOHN K. RODGERS
BY
THEIR ATTORNEY

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2,966,055

VARIABLE VOLUME CELL

Joseph H. Tracht, Pittsburgh, and John K. Rodgers, O'Hara Township, Allegheny County, Pa., assignors to Gulf Research & Development Company, Pittsburgh, Pa., a corporation of Delaware

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7 Claims. (Cl. 73—17)

This invention relates to a variable volume cell and more particularly to a cell which permits visible observation of phase equilibria, measurement of the volumes of the several phases, and means of sampling them.

Most condensate reservoirs or high pressure reservoirs, containing both normally gaseous and normally liquid hydrocarbons in a single gaseous phase, require careful study and extensive planning if they are to be produced in the most economical manner. As a condensate reservoir is produced and its pressure declines, liquids will gradually condense and accumulate in the reservoir, reaching a maximum at some intermediate pressure, often around 1200 p.s.i., and then will behave in a normal manner for gaseous systems, i.e., they will begin to vaporize on further reduction of pressure. This phenomenon is called retrograde-condensation. Unfortunately all of the liquids that condense in the reservoir upon reduction of the pressure are not revaporized and cannot be produced. These liquids wet the formation and are lost. The amount of condensation upon production from the formation can be reduced by maintaining a high pressure on the formation. This is ordinarily accomplished by cycling dry, non-condensable hydrocarbon gases through the formation. Because of the expense of cycling dry gases it is desirable to know how much of an increase in production from the formation can be obtained before planning cycling operations.

In order to obtain data which will allow the prediction of the performance of a condensate reservoir, samples of reservoir fluids are introduced into a bomb maintained substantially at the temperature of the reservoir. The sample is then expanded by reduction of the pressure at constant temperature and the amount of condensation occurring is determined. Characteristics of the condensate are also obtained by a constant mass expansion in a cell of variable volume. It is additionally desirable to obtain the retrograde dew point of the hydrocarbon mixture, which is defined as that pressure at which the first drop of liquid condenses from the gaseous phase on the isothermal reduction of pressure.

In the variable volume cells heretofore available, accurate visual measurement of the volumes of the phases in equilibrium has been difficult if not impossible. In those cells in which heavy glass sight glasses were provided directly in the cell the sight glasses generally covered such a small area that only a small portion of the change in liquid volume could be observed. In some instances the shape of the cell or the location of the sight glass is such that accurate measurement of a slight change in volume of the liquid is not possible.

The extremely high pressures at which the cells are used which may range up to 10,000 p.s.i. or more, make it desirable that the cell allow accurate measurement of small samples. Then, if there should be a failure of the apparatus the release of energy that occurs is limited. It is further desirable that the apparatus allow equilibria to be obtained in the cell at known temperatures.

This invention resides in apparatus for the visual measurement of phase equilibria on constant temperature and constant mass expansion of samples of mixtures of normally gaseous and normally liquid substances, and for displacement of accurately measured samples, in which an elongated vertical transparent sample tube is supported within a jacket adapted to withstand high pressures. Sight glasses in the jacket permit observation of the sample tube over substantially its full height. The pressure within the sample tube and within the jacket is equalized by sealing fluids in a connecting line joining the lower end of the sample tube and the space surrounding it within the jacket. A stirring mechanism within the jacket stirs both the hydraulic fluid surrounding the tube and the sample within the tube and thereby facilitates reaching equilibrium conditions within the sample cell.

In the drawings:

Figure 1 is a diagrammatic arrangement of apparatus constructed according to this invention for the measurement of phase equilibria.

Figure 2 is a vertical sectional view along the section line II—II in Figure 4 through the jacket and sample tube.

Figure 3 is a front view, partly in section along section line III—III in Figure 4, showing the arrangement of the stirring apparatus.

Figure 4 is a top view illustrating the connections to the top of the jacket.

Figure 5 is a detailed sectional view along the section line V—V in Figure 4 of the valve at the upper end of the sample tube for control of the release of pressure and removal of the sample.

Figure 6 is a front elevation of the jacket showing the sight glasses permitting observation of the sample within the cell 2.

Figure 7 is a horizontal sectional view, taken along the section line VII—VII in Figure 3.

Figure 8 is a plan view of the bottom of the jacket.

Referring to the drawings, in Figure 1 is shown a diagrammatic sketch of the variable volume cell of this invention with connected apparatus used for measurement of phase equilibria. The variable volume cell, indicated generally by reference numeral 10, is supported in a constant temperature bath 12. Conventional means, not shown, can be used to control the temperature of the bath 12. It is important that the liquid in the constant temperature bath be transparent. The variable volume cell 10 comprises an elongated vertically arranged tubular member closed at each end and having sight glasses 14 extending longitudinally thereof to permit observation within the cell for substantially its full length. A transparent sample tube 16 extends vertically into the upper and lower closure blocks of the variable volume cell. The upper end of the sample tube 16 is connected through high pressure sample tubing 18 to a sample container 20. Suitable valves 22 and 24 are provided between the sample container 20 and the sample tube 16 to permit purging the sample tubing 18 and disconnecting the sample container 20 without disturbing the sample within the sample tube 16.

The lower end of the sample tube 16 is connected to a pressure equalizing conduit consisting of tubing sections 26 and 28 provided with valves 30 and 32. The tubing section 28 of the pressure equalizing conduit opens into the space 34 in the cell 10 surrounding the sample tube 16. A sealing fluid supply line 36 provided with a valve 38, a by-pass line 40, a by-pass valve 42, and a pump 44 allows control of the pressure on a sealing fluid, preferably mercury, delivered through tubing section 26 into the lower end of the sample tube 16 to form a lower boundary of the sample.

A passage extends from the space 34 through the upper end of variable volume cell 10 and is connected with a

hydraulic fluid supply line 48. A valve 50, by-pass 52, by-pass valve 54 and pump 56 allows control of the pressure on the hydraulic fluid. During operation of the apparatus a hydraulic fluid such as a light transparent oil occupies space 34. A pressure gauge 58 is connected to the hydraulic fluid supply line 48 for measurement of the pressure on the hydraulic fluid, and, because of the connection through the pressure equalizing line, on the sample in tube 16.

During operation of the cell the level of the liquid in the transparent sample tube 16 will change when the pressure on the sample is changed. Accurate measurement of the liquid level is made by a cathetometer 60 positioned to allow observation of the liquid level in sample tube 16 through the sight glasses 14.

In order to speed reaching equilibrium conditions within the variable volume cell, a stirrer indicated generally by 62 agitates the hydraulic fluid in the space 34 as well as the sample in the sample tube 16. The stirrer consists of a permanent type magnet 64 adapted to move vertically in the space 34 along a shaft 66. As the magnet 64 moves, it pulls an agitator plate 68 of a magnetic material within the sample tube 16 along with it. The shaft 66 is driven through a flexible drive cable 70 by an electric motor 72. Electric motor 72 is provided with a conventional limit switch indicated diagrammatically by 74 which periodically reverses the direction of rotation of the motor to cause the magnet 64 to move alternately up and down within the space 34.

Referring to Figures 2 and 7 in which the cell 10 is illustrated in detail, a heavy walled cylindrical jacket 76 has slits 78 to allow observation into the cell surrounded by shoulders 80 which support the sight glasses 14. The sight glasses, which are of heavy glass adapted to withstand the high pressure, are held in place by cover plates 82 secured by bolts 84 and nuts 86. Suitable sealing means such as O-rings 88 are provided to prevent leakage between the sight glasses 14 and jacket 76.

The upper end of the cell 10 is closed by an upper closure block 90 secured in place by an upper cap 92 which is screwed on the jacket 76. A wear plate 94 of hardened steel is engaged by the lower ends of adjusting screws 96 for final tightening of the top closure block 90 in place. The lower end of the cell 10 is closed in a manner similar to the upper end by a lower closure block 98, a lower cap 100, a wear plate 102 and adjusting screws 104. Leakage between the jacket 76 and the upper and lower closure blocks is prevented by O-rings 105.

The inner ends of the upper and lower closure blocks are recessed at 106 and 108 respectively to receive the ends of the sample tube 16. Bushings 110 provided with sealing rings 112 support the ends of the sample tube 16 in the recesses 106 and 108. A duct 114 extends from the upper end of the recess 106 angularly upward through the upper closure block 90 into a socket 116 in which a needle valve, indicated generally by 118, is mounted. The needle valve is provided with a suitable bearing and packing arrangement to prevent leakage along the valve stem out of the cell when the sample in sample tube 16 is under either pressure or vacuum. A passage 120, best illustrated in Figure 5 of the drawings, extends from the socket 116 above the seat engaged by the needle valve 118 upwardly through the upper closure block. The sample tubing 18 illustrated in Figure 1 is secured to the closure block 90 for communication with passage 120 by a suitable high pressure fitting such as the Aminco fitting 122 illustrated in Figure 5.

A passage 124 extends upwardly through the upper closure block 90 from a position outside of the sample tube 16 to allow flow into the space 34 within the jacket 76. Passage 124 opens at its upper end into the lower end of a socket which receives a high pressure fitting 126 on the end of the hydraulic fluid tubing 48, illustrated in Figure 1.

Referring to Figure 2, the lower end of sample tube 16 is supported above the bottom of recess 108 by a helical spring 128. A duct 130 extends angularly downward from the recess 108 below the lower end of the sample tube 16 and opens into a socket 132 in the lower surface of the lower closure block 98. Tubing section 26 of the pressure equalizing conduit illustrated in Figure 1 is secured in recess 132 by high pressure fitting 134. A duct 136 extends downwardly through the lower closure member 98 from a position outside of the sample tube 16 and opens into a socket 138 in the lower surface of the lower closure member 98. The tubing section 28 of the pressure equalizing line illustrated in Figure 1 of the drawings is secured in socket 138 by means of a high pressure fitting 140. Tubing sections 26 and 28 of the pressure equalizing line are connected together by high pressure tubing to form a pressure equalizing line connecting the space 34 with the space within sample tube 16 and are provided with valves in the manner illustrated in Figure 1 of the drawings. A reservoir for the sealing liquid frequently is desirable in tubing section 26.

The stirrer 62 is best illustrated in Figure 3 of the drawing. Referring to that figure, the upper closure block 90 is drilled and threaded at 142 to receive a bushing 144 having a bronze bearing 146 supported in the lower end of the bushing 144 to rotatably support the stirrer shaft 66. Leakage of hydraulic fluid along the shaft 66 is prevented by a sealing cup 150 at the lower end of the bushing 144. The lower end of the shaft 66 is supported on a ball bearing 151 resting in a socket in the upper end of a plug 152 screwed into a recess in the lower closure block 98. The shaft 66 extends downwardly onto the bearing 151 through an opening 154 in the lower closure block 98.

Riding on threads 155 on the shaft 66 within the space 34 is a follower 156 which moves up or down on the shaft depending upon the direction of rotation of the shaft. The permanent magnet 64 is mounted on the follower 156. The force exerted by magnet 64 pulls the agitator plate 68 along with the magnet 64 as it moves up and down in the space 34.

The equalization of pressure on the inside and outside of the sample tube 16 eliminates the restrictions placed on the design of the sample tube when the tube must withstand high pressures. The sample tubes are readily interchangeable, thereby permitting the use of tubes with either large or small bores, depending on the nature of the sample. If a large sample is desired, for example when the sample is only available at a low pressure, sample tubes with bulbs at their lower end can be used and the sample compressed into the smaller diameter upper section. However, one advantage of this apparatus is the use of small, accurately dimensioned sample tubes made possible by the very accurate visual measurement of liquid volumes that can be obtained. The small samples reduce the danger in handling materials under very high pressures by limiting the amount of energy that can be released.

For convenience, the use of this invention will be described for the determination of phase equilibria of a mixture of normally liquid and normally gaseous hydrocarbons from a condensate reservoir. In the use of the variable volume cell of this invention the space 34 within the jacket is filled with the hydraulic fluid, and the sample tube 16 and tubing 18 are filled with a sealing liquid. Mercury is a preferred sealing liquid. The constant temperature bath is adjusted to the desired temperature, usually the temperature of the condensate reservoir. The pressure on the hydraulic fluid in space 34 is adjusted to approximately the pressure on the sample. A sample of the mixture of hydrocarbons is introduced from the sample container 20 into the sample tube 16 by displacement of the mercury. After the sample has been introduced into the sample tube 16 the valve 118 is closed and the pressure on the sample is adjusted to the

desired level by control of the valves **38** and **42** in the sealing liquid supply line and the valves **50** and **54** in the hydraulic liquid supply line. The valves **30** and **32** are open to allow flow through tubing sections **26** and **28** of the pressure equalizing conduit to maintain substantially equal pressures on the inside and outside of the sample tube **16**. Pressure gauge **58** then indicates the pressure on the sample. Slight differences in level of the mercury on the inside of tube **16** and the outside cause only negligible differences in pressure compared to the very high pressure at which the apparatus is used. The mercury level should be lower in space **34** than in tube **16** to permit the tube to be seen.

Once the sample has been introduced into the sample tube **16** the pressure on the sample can be varied to obtain phase equilibria data. For example, the pressure on the sample can be reduced by lowering the level of the sealing liquid in the sample tube **16**, with a corresponding reduction of the pressure in the space **34**. The retrograde dew point of the sample is determined from the pressure gauge **58** when the first droplets of liquid are formed upon reduction of the pressure. The compressibility of the hydrocarbons in the sample is determined by a series of measurements of the volume at different pressures. The cathetometer **60** allows accurate measurement of the volume of the sample and of the liquid by observation of the level of the sealing liquid and the liquid portion of the hydrocarbon sample in the sample tube **16**. The valves **30** and **32** can be adjusted to allow raising the level of the sealing liquid in the sample tube without substantial change of pressure in the space **34** thereby allowing easy and rapid displacement of very accurately measured samples of gases or liquids from the sample tube.

After a series of determinations of the characteristics of the sample by constant mass expansions has been made, production from the reservoir can be duplicated by the slow discharge of a portion of the sample through valve **118** into a suitable receiver. The amount of retrograde condensation for any given drop in pressure can be observed directly by means of the cathetometer.

The apparatus of this invention can be used to determine the bubble point of normal crude oils, to determine the critical constants of fluid systems, to bring fluid systems into equilibrium so that their different phases can be separated and removed for analysis, as well as to determine the dew point, compressibility, and amount of retrograde condensation of the fluids. The internal stirring magnet allows the sample quickly to be brought to equilibrium at the desired temperature. The movement of the magnet in the body of hydraulic fluid produces a uniform temperature throughout the hydraulic fluid and speeds heat transfer to that liquid. By positioning the magnet close to the sample tube permanent magnets can be used and thereby avoid extraneous heat effects introduced by electric magnets. The permanent magnet also eliminates the necessity of electric leads into the high pressure cell.

We claim:

1. Apparatus for observing conditions of phase equilibria comprising an elongated vertical jacket adapted to withstand high pressure, means closing the upper and lower ends of the jacket, sight glasses in the jacket extending for substantially the full height of the jacket, a vertical transparent sample tube in the jacket in alignment with the sight glasses whereby liquid in the sample tube may be observed, said sample tube having its upper and lower ends mounted in the means closing the upper and lower ends of the jacket, respectively, a conduit connecting the lower end of the sample tube with the space in the jacket surrounding the sample tube, a sealing liquid within the conduit serving as a bottom closure for the sample tube, a transparent hydraulic fluid in the jacket surrounding the sample tube, an agitator plate of magnetic material free to move in the sample tube, a shaft extending vertically through the jacket adjacent the sample tube, a permanent magnet mounted on the shaft adapted to move vertically within the jacket to move the agitator plate in the sample tube and thereby agitate the sample and the hydraulic fluid, means for rotating the shaft, and means for reversing the direction of rotation of the shaft whereby the permanent magnet moves alternately up and down on the shaft.

2. Apparatus for observing conditions of phase equilibia comprising an elongated vertical jacket adapted to withstand high pressures, means closing the upper and lower ends of the jacket, sight glasses in the jacket extending for substantially the full height of the jacket, a transparent sample tube in the jacket in alignment with the sight glasses whereby liquid in the sample tube may be observed, said sample tube having its upper and lower ends mounted in the means closing the upper and lower ends of the jacket, respectively, a conduit connecting the lower end of the sample tube with the space in the jacket surrounding the sample tube, a sealing liquid within the conduit serving as a bottom closure for the sample tube, a transparent hydraulic fluid in the jacket surrounding the sample tube, an agitator plate of magnetic material free to move in the sample tube, a rotatable shaft extending vertically through the jacket adjacent the sample tubing, said shaft having threads on its outer surface, a permanent magnet mounted upon the shaft movable on the shaft by the threads thereon as the shaft rotates to move the agitator plate in the sample tube, a motor connected for rotating the shaft, and means for periodically reversing the direction of rotation of the motor to reverse the direction of movement of the magnet and thereby agitate the sample and the hydraulic fluid.

3. Apparatus for observing conditions of phase equilibria comprising an elongated vertical jacket adapted to withstand high pressures, means closing the upper and lower ends of the jacket, sight glasses in the jacket extending for substantially the full height of the jacket, a transparent sample tube in the jacket in alignment with the sight glasses whereby liquid in the sample tube may be observed, said sample tube having its upper and lower ends mounted in the means closing the upper and lower ends of the jacket, respectively, a conduit connecting the lower end of the sample tube with the space in the jacket surrounding the sample tube, a sealing liquid within the conduit serving as a bottom closure for the sample tube, a transparent hydraulic fluid in the jacket surrounding the sample tube, an agitator plate of magnetic material free to move in the sample tube, a rotatable shaft extending vertically through the jacket adjacent the sample tubing, said shaft having threads on its outer surface, a permanent magnet mounted upon the shaft movable on the shaft by the threads thereon as the shaft rotates to move the agitator plate in the sample tube, a motor connected for rotating the shaft, and means for periodically reversing the direction of rotation of the motor and thereby reversing the direction of movement of the magnet on the shaft.

4. Apparatus for observing conditions of phase equilibria comprising an elongated tubular vertical jacket, means closing the upper and lower ends of the jacket, diametrically opposed vertical slits through the jacket extending for substantially the full height of the jacket, a sight glass secured over each of the slits, a transparent sample tube extending vertically into the means closing the upper and lower ends of the jacket in alignment with the slits whereby the sample tube may be observed from outside of the jacket, a conduit connecting the lower end of the sample tube with the space in the jacket surrounding the sample tube, a sealing liquid within the conduit serving as a bottom closure for the sample tube, means for adjusting the pressure on the sealing liquid, a transparent hydraulic fluid in the jacket surrounding the sample tube, means for adjusting the pressure on the hydraulic fluid, an agitator plate of magnetic material free to move in the

sample tube, a rotatable shaft extending vertically through the jacket adjacent the sample tube, said shaft having threads extending along its outer surface from its upper to its lower end, a permanent magnet mounted upon the shaft adapted to move along the shaft as the shaft rotates to move the agitator plate in the sample tube, means for rotating the shaft, and means to reverse periodically the direction of rotation and thereby the direction movement of the magnet to agitate the hydraulic fluid and contents within the sample tube.

5. A variable volume cell for observing conditions of phase equilibria comprising a vertical tubular jacket constructed to withstand high pressures, means closing the upper and lower ends of the jacket, each of said means having a passage therethrough, a sight glasses secured to the jacket covering longitudinal slits in the jacket, a transparent sample tube extending longitudinally within the jacket with its ends mounted in the passages in the means closing the upper and lower ends of the jacket, said sample tube being in alignment with the sight glasses, said sample tube having an external diameter smaller than the internal diameter of the jacket to leave an annular space within the jacket surrounding the sample tube, a transparent hydraulic liquid in the annular space, a valve in the passage in the means closing the upper end of the jacket, a conduit from the passage in the means closing the lower end of the jacket to the annular space adapted to equalize pressure in the sample tube and annular space, a sealing liquid in the conduit, a sealing liquid supply line opening into the conduit, means for supplying sealing liquid under high pressure into the sealing liquid supply line, valves in the conduit allowing selective introduction of sealing liquid into the sample tube and annular space, a hydraulic liquid line opening into the annular space, means for delivering hydraulic liquid at a controlled pressure into the hydraulic liquid line, a rotatable shaft extending through the annular space from the means closing the upper end to the means closing the lower end of the jacket, a traveler on the shaft adapted to move upwardly on the shaft as the shaft rotates in one direction and downwardly as the shaft rotates in the other direction, a permanent magnet mounted on the follower, and an agitator plate of magnetic material free to move in the sample tube in response to movement by the magnet.

6. A variable volume cell for observing conditions of phase equilibria of fluids comprising a vertical tubular jacket constructed to withstand high pressure, means closing the upper and lower ends of the jacket, each of said means closing the ends of the jacket having a first and a second passage therethrough, a sight glass secured to the jacket covering a longitudinal slit in the jacket, a transparent sample tube extending longitudinally within the jacket in position to be seen through the sight glass, said sample tube having its upper end mounted in the first passage in the means closing the upper end of the jacket and its lower end mounted in the first passage in the means closing the lower end of the jacket, the sample tube having an external diameter less than the internal dimensions of the jacket to leave an annular space within the jacket surrounding the sample tube, the second passage in each of the means closing the upper and lower ends of the jacket communicating with the annular space surrounding the sample tube, a valve in the first passage in the means closing the upper end of the jacket, a conduit connecting the first and second passages in the means closing the lower end of the jacket adapted to equalize pressure in the sample tube and the annular space, a sealing liquid in the conduit and the lower end of the sample tube for confining the fluid in the sample tube between the valve in the first passage in the means closing the upper end of the jacket and said sealing liquid, said sealing liquid being immiscible with the fluid in the sample tube, a sealing liquid supply line opening into the conduit, means for supplying sealing liquid under high pressure into the sealing liquid supply line for displacement into the sample tube to reduce the volume above the sealing liquid in the sample tube, a valve in the conduit on each side of the intersection of the conduit with the sealing liquid supply line for the selective introduction of sealing liquid into the sample tube and annular space, a transparent hydraulic liquid in the annular space within the jacket, a hydraulic liquid supply line connecting with the second passage in the means closing the upper end of the jacket for delivery of the hydraulic liquid into the annular space within the jacket, and means for displacing hydraulic liquid through the hydraulic liquid supply line at a controlled pressure approximately equal to the pressure in the sample tube.

7. A cell for observing conditions of phase equilibria of fluid comprising a constant temperature bath, a vertical tubular jacket within the constant temperature bath, said vertical tubular jacket being constructed to withstand high pressure, means closing the upper and lower ends of the jacket, each of said means closing the ends of the jacket having a first and a second passage therethrough, a sight glass secured to the jacket covering a longitudinal slit in the jacket, a transparent sample tube extending longitudinally within the jacket in position to be seen through the sight glass, said sample tube having its upper end mounted in the first passage in the means closing the upper end of the jacket and its lower end mounted in the first passage in the means closing the lower end of the jacket, the sample tube having an external diameter less than the internal dimensions of the jacket to leave an annular space within the jacket surrounding the sample tube, the second passage in each of the means closing the upper and lower ends of the jacket communicating with the annular space surrounding the sample tube, a valve in the first passage in the means closing the upper end of the jacket, a conduit connecting the first and second passages in the means closing the lower end of the jacket adapted to equalize pressure in the sample tube and the annular space, a sealing liquid in the conduit and the lower end of the sample tube for confining the fluid in the sample tube between the valve in the first passage in the means closing the upper end of the jacket and said sealing liquid, said sealing liquid being immiscible with the fluid in the sample tube, a sealing liquid supply line opening into the conduit, means for supplying sealing liquid under high pressure into the sealing liquid supply line for displacement into the sample tube to reduce the volume above the sealing liquid in the sample tube, a valve in the conduit on each side of the intersection of the conduit with the sealing liquid supply line for the selective introduction of sealing liquid into the sample tube and annular space, a transparent hydraulic liquid in the annular space within the jacket, a hydraulic liquid supply line connecting with the second passage in the means closing the upper end of the jacket for delivery of the hydraulic liquid into the annular space within the jacket, and means for displacing hydraulic liquid through the hydraulic liquid supply line at a controlled pressure approximately equal to the pressure in the sample tube.

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