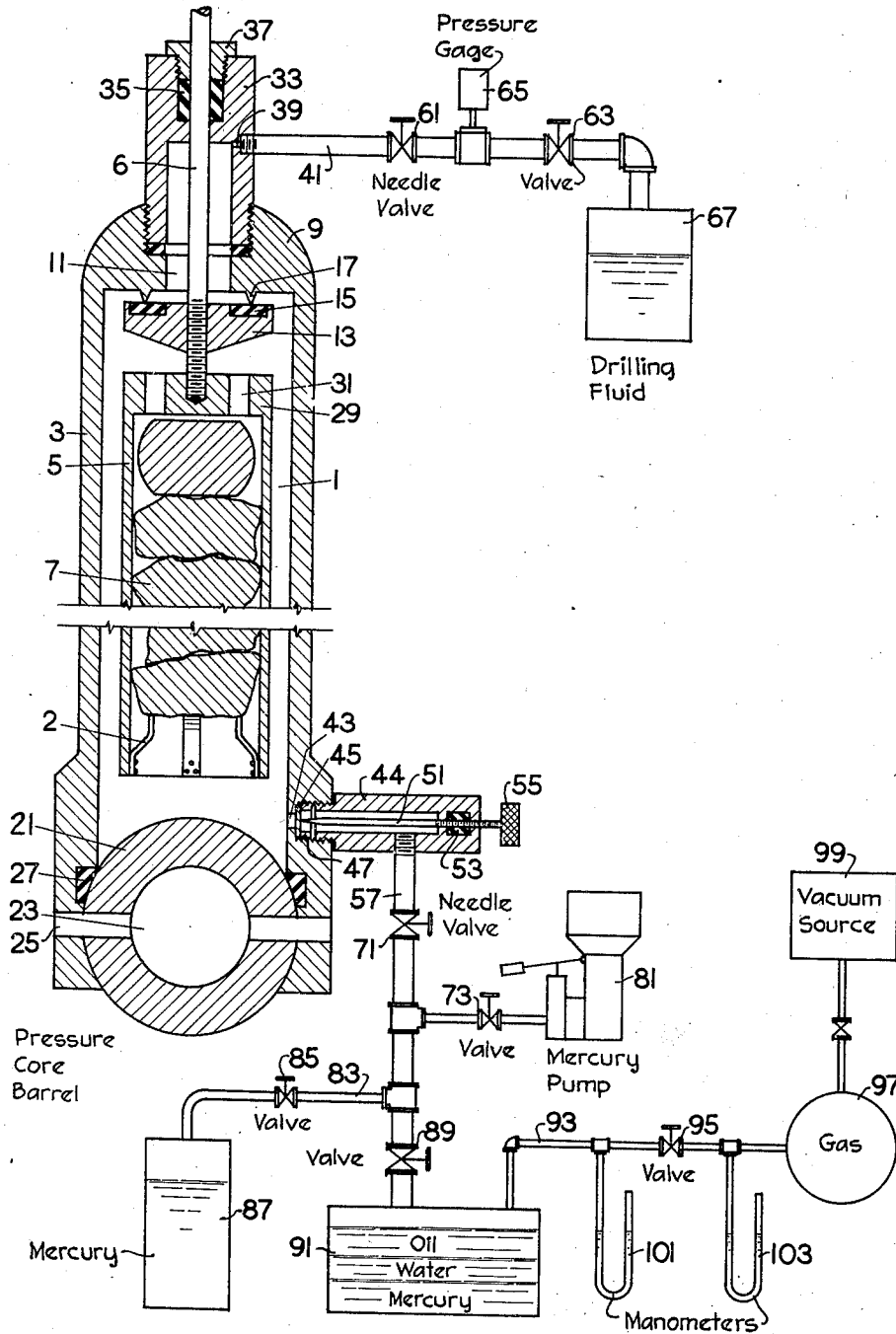


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METHOD OF DETERMINING THE FLUID CONTENTS
OF UNDERGROUND FORMATION SAMPLES
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METHOD OF DETERMINING THE FLUID
CONTENTS OF UNDERGROUND FORMA-
TION SAMPLES

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This invention pertains to the art of production of hydrocarbon materials from underground formations, and relates more specifically to a method for accurately determining the exact fluid contents of formation samples or cores obtained in drilling wells.

It is at present standard procedure to core wells throughout most of their depth during the drilling process for purposes of geological exploration and correlation of data pertaining to underground structures in a particular area. Besides examining and analyzing recovered cores from a geological point of view, it is of vital importance in petroleum production work to measure with accuracy the natural fluid contents of the cores, such as gas, oil and water, since the information thus obtained is essential for ascertaining the nature and characteristics of a given reservoir, and in estimating the mineral oil reserves held therein.

Accurate measurements of the true amount of the natural fluid contents of a core are, however, difficult to obtain.

First, cores are normally drilled out at relatively very high underground pressures. When a core is raised to the surface, for example in a core barrel, the pressure drop from bottom hole to atmospheric pressure causes the fluids present in the core to expand and to flow out of the core. Furthermore, certain hydrocarbons, which may be present in the core in liquid phase at bottom hole pressure, pass to a gaseous phase at atmospheric pressure and are thus lost. When so-called pressure core-barrels are used, since some drilling fluid is unavoidably entrapped therein and brought to the surface together with the core, fluids which escape from the core when the pressure is released become admixed with this well fluid and cannot be separated therefrom for purposes of accurate measurement of the fluid contents of the core proper.

Second, cores which are obtained by rotary drilling with drilling fluid circulation are subject to flushing or penetration by said drilling fluid, whereby varying quantities of gas, oil or formation water originally present in a core are flushed off and replaced therein by the contaminating drilling fluid. The measurement of the actual fluid content of cores is therefore often meaningless, since it is not possible to determine what portion of the measured core fluids is present there by reason of natural causes, and what portion by reason of contamination.

It is therefore an object of this invention to provide a method whereby the exact amount of

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the fluids naturally present in a core can be accurately determined.

It is also an object of this invention to provide a method whereby the flushing and contamination of the natural fluid contents of cores by the drilling fluid is eliminated or minimized, and the degree of this contamination, if any, is accurately measured to permit proper corrections in measurements made. It is also an object of this invention to provide a method whereby intermixing, upon pressure release, of the natural fluid contents of a core with the drilling fluid recovered therewith is eliminated, and the accuracy of the measurements is thereby greatly increased.

These and other objects of this invention will be understood from the following description taken with regard to the attached drawing which represents, in diagrammatic form, a preferred arrangement of apparatus used in practicing the method of the present invention, which is carried out in the following manner.

First, a core is drilled out by ordinary rotary drilling means, using a suitable core head or drill.

While drilling the core out, a special drilling fluid is used to prevent the flushing and contamination of the core. This contamination is due to the fact that some fluid is forced into the core by the hydraulic pressure of the drilling fluid circulating in the borehole. The degree of contamination of a core by this fluid depends upon the plastering properties of said fluid, that is, its ability of immediately forming, on the walls of the borehole or of the core, a thin and substantially fluid-imperious sheath which prevents any appreciable penetration of the core by the liquid phase of the drilling fluid. Since oil-base muds have plastering properties normally greatly superior to water-base drilling fluids, which properties are furthermore not subject to deterioration by admixture with formation brines, it is preferred to use an oil-base drilling fluid when drilling out cores according to the present invention. For example, an oil-base drilling fluid such as described in U. S. Letters Patent Nos. 2,222,949, 2,223,027, 2,297,660, 2,350,154, etc., and preferably an oil-base drilling fluid giving a zero fluid loss in a filter test run at 100 p. s. i. pressure at 70° F. for 60 minutes, can be advantageously used.

Since, however, even oil-base drilling fluids with the best possible plastering properties will allow a certain amount, however small, of core contamination, a tracer material is added to said drilling fluid in a predetermined concentration in order that the degree of such contamination may be quantitatively determined and proper correc-

tions made in the ultimate measurements of the natural fluid contents of the cores.

As tracer materials, it is possible to use, according to the present invention, those substances whose physical or chemical properties will allow an accurate quantitative determination of the degree of contamination by physical or chemical examination or analysis methods.

For example, radioactive materials may be added to the oil-base drilling fluids in the form of oil-soluble soaps of radium, such as radium oleates, palmitates, stearates, etc., and the presence of the radium together with the contaminating fluid in the core can then be detected and quantitatively measured by means of suitable apparatus involving the use of Geiger-Mueller counters or ionization chambers. It has been found that by using quantities of radium as small as 0.159 milligram of radium per thousand barrels of the drilling fluid, contaminations of the order of 10 percent of the fluid content of the core can be detected, while use of larger but still entirely practical amounts of radium, such as 1 milligram per thousand barrels, permits carrying such contamination determinations to a much greater degree of quantitative accuracy.

It is likewise possible to add to the oil-base drilling fluid suitable tracer materials which will make it possible to determine the presence of a contaminating fluid in a core by means of chemical analysis. Thus, tracer compounds comprising elements such as nitrogen, phosphorus, sulfur, chlorine, etc., for example amines such as aniline, tricesyl phosphate, mercaptans, etc., may be added in known concentrations to the drilling fluid, and cores subsequently analyzed for a quantitative determination of the amount of the contaminating fluid present therein.

It has, however, been found that certain organic compounds of chlorine, such as carbon tetrachloride, and particularly a compound technically known as "Aroclor" are especially suitable for the purposes of this invention. Aroclor, which is a chlorinated bi-phenyl resin, has a large content (about 70 percent) of chlorine, an element which allows of very sensitive yet relatively simple quantitative tests. Aroclor is furthermore soluble in oil and insoluble in water, which is of especial importance when drilling cores with an oil-base drilling fluid in a water-bearing layer. Aroclor can be added to the drilling fluid in known quantities to give desired concentrations, for example from 200 to 600 lbs. per 1000 barrels of the drilling fluid.

As a core is drilled out, according to the present invention, by the core head out of the formation, it is received in a pressure core barrel, that is, a core barrel adapted to be automatically closed at the bottom of the well, whereby the core may be raised to the surface together with said core barrel without releasing the pressure at which the core was obtained. A pressure core barrel such as described in U. S. Letters Patent No. 2,238,609 to Sewell may be suitably adapted for the purposes of this invention by means of a few simple modifications, as described hereinbelow.

This core barrel, disengaged from the core head or drill, raised to the surface, and connected to the system used in practicing the present invention, is shown in schematic form at 1 in the drawing.

The pressure barrel comprises an outer barrel or cylindrical housing 3, and an inner barrel or cylinder 5, suspended and axially movable within the barrel 3 by means of a rod 6.

The upper end of the barrel 3 is closed at the top by means of a head 9 having a central axial orifice 11, through which the rod 6 extends into the pressure barrel. The rod 6 carries, within the cylinder 3 and above the cylinder 5, a circular member 13, provided with a gasket or sealing ring 15, made of a suitable resilient packing material. The head 9 is formed with an annular knife-edge seat 17 registering with the sealing ring 15. When the inner cylinder 5 is raised by means of the rod 6 to the upper limit of its stroke, as for example when the pressure core barrel 1 is being lifted from the core head, the knife edge 17 engages the sealing ring 15 and forms therewith a pressure-tight closure hermetically sealing the upper end of the pressure core barrel.

The lower end of the pressure core barrel is provided with a valve such as a spherical or cylindrical valve 21 having a circular axial passage 23 of the same internal diameter as the inner core barrel 5. The valve 21 is affixed to the outer barrel 3 by means of a shaft or spindle 25, about which it is rotatable, so that during the core-drilling operations it occupies a position displaced by 90 degrees from that shown in the drawing, thus permitting the core to enter the inner core barrel 5 through the axial passage 23 which is in register therewith. When the core or core fragments 7 have entered the inner core barrel, wherein they are held by means of core-breaker springs or fingers 2, and while the core barrel is still at the bottom of the well the valve 21 is rotated about the shaft or spindle 25 by means operable from the surface, which means form no part of the present invention and are therefore not shown in the drawing, said means being however fully described in said Patent No. 2,238,609 to Sewell.

When the valve 21 is thus rotated to the position shown in the drawing, the lower end of the pressure core barrel is hermetically closed, packing 27 providing a pressure-tight seal, so that the core within the inner barrel and the well fluid entrapped therewith can be lifted to the surface, for example, together with the drill bit and string, and by means involving a pull on the rod 6, without releasing the bottom hole pressure obtaining within the pressure core barrel.

The inner barrel 5 is closed at the top by a head 29, provided with passages or orifices 31, said head being attached, for example, by means of screw-threads to the rod 6.

The central passage 11 through the upper head 9 of the outer barrel is provided with internal screw-threads adapted to engage the external screw-threads of a special head 33, which may be attached to the pressure core barrel when desirable, for example when said barrel has been removed from the core head.

The special head 33 has an upper axial passage adapted to fit around the rod 6, and provided with a pressure-tight packing 35, held in place by a screw 37. The space within the head 33 is in communication with the outside by means of a port or lateral passage 39 having an enlarged screw-threaded outer portion, adapted for engagement with a screw-threaded element such as a pipe or conduit 41.

The lower portion of the outer barrel 3 is also provided with a lateral port or passage 43, which is normally closed during core-drilling operations by means of a frangible disk 45. The passage 43 is enlarged and screw-threaded in its outer portion, and is adapted to receive a threaded retainer ring 47, which holds the disk 45 in place.

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A special fitting 44 is adapted to be connected in screw-threaded pressure-tight engagement with port 43.

The fitting 44 carries therein an axial stem or needle 51, which passes screw-threadedly through the closed outer end of the fitting, provided with pressure-tight packing 53. A knob 55 is attached to the outer end of said stem. By rotating the knob 55, the pointed end of the stem 51 may be caused to perforate the frangible disk 45, whereupon the fluids held under pressure within the core barrel can pass to the sealed hollow space within the fitting 44, and from there to a pipe 57 in communication therewith.

The upper port 39 is in communication, through pipe 41, provided with valves 61 and 63 and a pressure gage 65, with a reservoir 67.

The lower port 43 is in communication, through pipe 57, provided with valves 71 and 73, with a mercury pressure pump 81. A pipe 83 branches off the pipe 57 and leads, through a valve 85, to a first reservoir 87, while pipe 57 leads through a valve 89 to a second closed reservoir 91.

A pipe 93, provided with a valve 95, is connected to the top of reservoir 91 and leads to a gas reservoir 97, which is adapted to be exhausted by a source of vacuum 99, such as a vacuum pump. Manometers 101 and 103 are installed in the line 93 on both sides of valve 95 to determine the amount of gas which enters the evacuated gas reservoir 97.

As stated hereinabove, the pressure core barrel, mounted as shown in the drawing, contains besides the core 7 a considerable amount of the drilling fluid entrapped within both the outer and the inner barrels. It will be realized that if an attempt were made to remove this fluid, for example by opening the main valve 21 and letting this fluid drain out, the ensuing release of the pressure would cause some of the natural fluid content of the core proper to be forced out of said core. Assuming that the core had been drilled out in an oil-bearing layer, the oil flowing out of the core would become commingled with the oil-base mud and could not be separated therefrom for the purpose of determining the natural fluid content of the core. Likewise, some of the lighter hydrocarbons, which were present in the core in liquid phase under bottom hole pressures, would pass to the vapor phase at atmospheric pressure and would thus be lost.

The procedure according to the present invention is therefore as follows:

All valves shown in the drawing remaining closed, the knob 55 is rotated to advance the needle stem 51, thereby perforating the disk 45. The stem 51 is then drawn back, valves 71 and 73 are opened, and the mercury pump 81 is started, forcing mercury into the pressure barrel. Simultaneously, the rod 6 is moved downwards, disengaging the seal ring 15 from the knife-edge seat 17, and permitting the entrapped well fluid surrounding the core 7 to flow into the special head 33 as the mercury begins to fill the pressure core barrel from the bottom. Valves 61 and 63 are opened to permit the flow of said well fluid to the reservoir 67. The opening of the needle valve 61 is adjusted with regard to the pumping rate of the mercury so that a substantially constant pressure is maintained in the system. This pressure should preferably be slightly in excess of the initial pressure in the core barrel to prevent the flow of fluids from the core during this operation, but not substantially in excess thereof to prevent any penetration of the core by the mercury,

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which may take place under conditions of excessively high displacing pressure, especially if free gas is present in the core. As the presence of mercury in the pore space of the core will reduce its permeability and will cause difficulties in the core analysis procedure, this condition should be avoided by a proper adjustment of the valve 61 in accordance with the indications of the gage 65.

When the rising level of the mercury has completely displaced the well fluid from the pressure core barrel and special head 33, and mercury starts to flow into the reservoir 67, the operation of the mercury pump is discontinued, valve 73 closed, and valve 85 opened, permitting the mercury to drain from the core barrel into the reservoir 87. When a quantity of mercury nearly equal to that forced into the core barrel has been received in the reservoir 87, valve 85 is closed and valve 89 opened. The natural formation fluids present in the core, such as gas, oil and/or connate water, which are released therefrom due to the drop of pressure, are received into the reservoir 91, as well as any gases issuing from the core. Reservoir 91, being a closed reservoir, serves as a trap for the liquids, whereas the gases accumulating in the upper portion thereof are drawn through pipe 93 and valve 95, which is opened at this time, to the gas reservoir 97, which has been evacuated by means of the vacuum pump 99.

Care is taken that all the fluids present in the core are removed therefrom, which may be effected by applying, in well known manner, heat and/or vacuum to the core, and then, if desired, crushing and extracting said core with suitable solvents, such for example as toluene. The total amounts of the natural fluid contents of the core, such as water, oil and gases, can in this manner be accurately determined.

As stated above, even oil-base drilling fluids with the highest possible plastering properties will allow a certain amount of contamination, although the ultimate error in the determination of the natural fluid contents of the core will be the smaller, the smaller the magnitude of said contamination. To determine the extent of said contamination, the total natural fluids recovered from the core can be analyzed for the amount of "Aroclor" chlorine present therein. The concentration of the "Aroclor" in the drilling fluid being known, the volume of the contaminating drilling fluid forced into the core may be easily determined from the amount of chlorine found present in the core fluid contents, and proper corrections may be introduced into the final calculations.

I claim as my invention:

1. In a method for measuring the natural formation fluid contents of a core drilled out while circulating an oil-base drilling fluid in a borehole, the steps of confining the core and a portion of the surrounding drilling fluid in situ at bottom hole pressure in a sealed container, raising said container to the surface, injecting mercury into said container to displace the drilling fluid therefrom while maintaining the pressure within said container substantially constant at bottom hole pressure until the drilling fluid has been completely displaced therefrom, releasing said pressure, removing said mercury from said container and the fluids present in the core from said core, whereby the amount of the natural fluid in the core may be measured.

2. In a method for measuring the natural formation fluid contents of a core drilled out while circulating an oil-base drilling fluid in a bore-

hole, the steps of adding a tracer material to the drilling fluid in known concentration, confining the core and a portion of the surrounding drilling fluid in situ at bottom hole pressure in a sealed container, raising said container to the surface, injecting mercury into said container to displace the drilling fluid therefrom while maintaining the pressure within said container substantially constant at bottom hole pressure until the drilling fluid has been completely displaced therefrom, releasing said pressure, removing said mercury from said container and the fluids present in the core from said core, determining the amount of the fluids present in said core, and analyzing said fluids for the concentration of said tracer material in said fluids, whereby the respective amounts of the drilling fluid contaminating said core and of the natural formation fluid in said core may be determined.

3. In a method for measuring the natural formation fluid contents of a core drilled out while circulating an oil-base drilling fluid in a bore-hole, the steps of adding in known concentration to the drilling fluid a tracer material comprising an oil-soluble, water-insoluble organic compound of chlorine, confining the core and a portion of the surrounding drilling fluid in situ at bottom hole pressure in a sealed container, raising said container to the surface, injecting mercury into said container to displace the drilling fluid therefrom while maintaining the pressure within said container substantially constant at bottom hole pressure until the drilling fluid has been completely displaced therefrom, releasing said pressure, removing said mercury from said container and the fluids present in the core from said core, determining the amount of the fluids present in said core, and analyzing said fluids for the concentration of said tracer material in said fluids, whereby the respective amounts of the drilling fluid contaminating said core and of the natural formation fluid in said core may be determined.

4. In a method for measuring the natural formation fluid contents of a core drilled out while circulating an oil-base drilling fluid in a bore-hole, the steps of adding in known concentration to the drilling fluid a tracer material comprising a chlorinated bi-phenyl resin, confining the core and a portion of the surrounding drilling fluid in situ at bottom hole pressure in a sealed container, raising said container to the surface, injecting mercury into said container to displace the drilling fluid therefrom while maintaining the pressure within said container substantially constant at bottom hole pressure until the drilling fluid has been completely displaced

therefrom, releasing said pressure, removing said mercury from said container and the fluids present in the core from said core, determining the amount of the fluids present in said core, and determining the amount of the tracer material contained in said fluids, whereby the respective amounts of the contaminating drilling fluid and of the natural formation fluid in the core may be determined.

5. In a method for measuring the natural formation fluid contents of a core drilled out while circulating an oil-base drilling fluid in a bore-hole, the steps of adding in known concentration to the drilling fluid a tracer material comprising an oil-soluble, water-insoluble organic compound of chlorine, having a large content of chlorine, drilling out a core with the employment of said drilling fluid, raising the core to the surface, removing the fluids present in the core from said core, and analyzing said fluids for the content of the tracer material present therein whereby the respective amounts of the drilling fluid contaminating said core and of the natural formation fluid in said core may be determined.

6. In a method for measuring the natural formation fluid contents of a core drilled out while circulating an oil-base drilling fluid in a bore-hole, the steps of adding in known concentration to the drilling fluid a tracer material comprising a chlorinated bi-phenyl resin, drilling out a core with the employment of said drilling fluid, raising the core to the surface, removing the fluids present in the core from said core, and analyzing said fluids for the content of the tracer material present therein whereby the respective amounts of the drilling fluid contaminating said core and of the natural formation fluid in said core may be determined.

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