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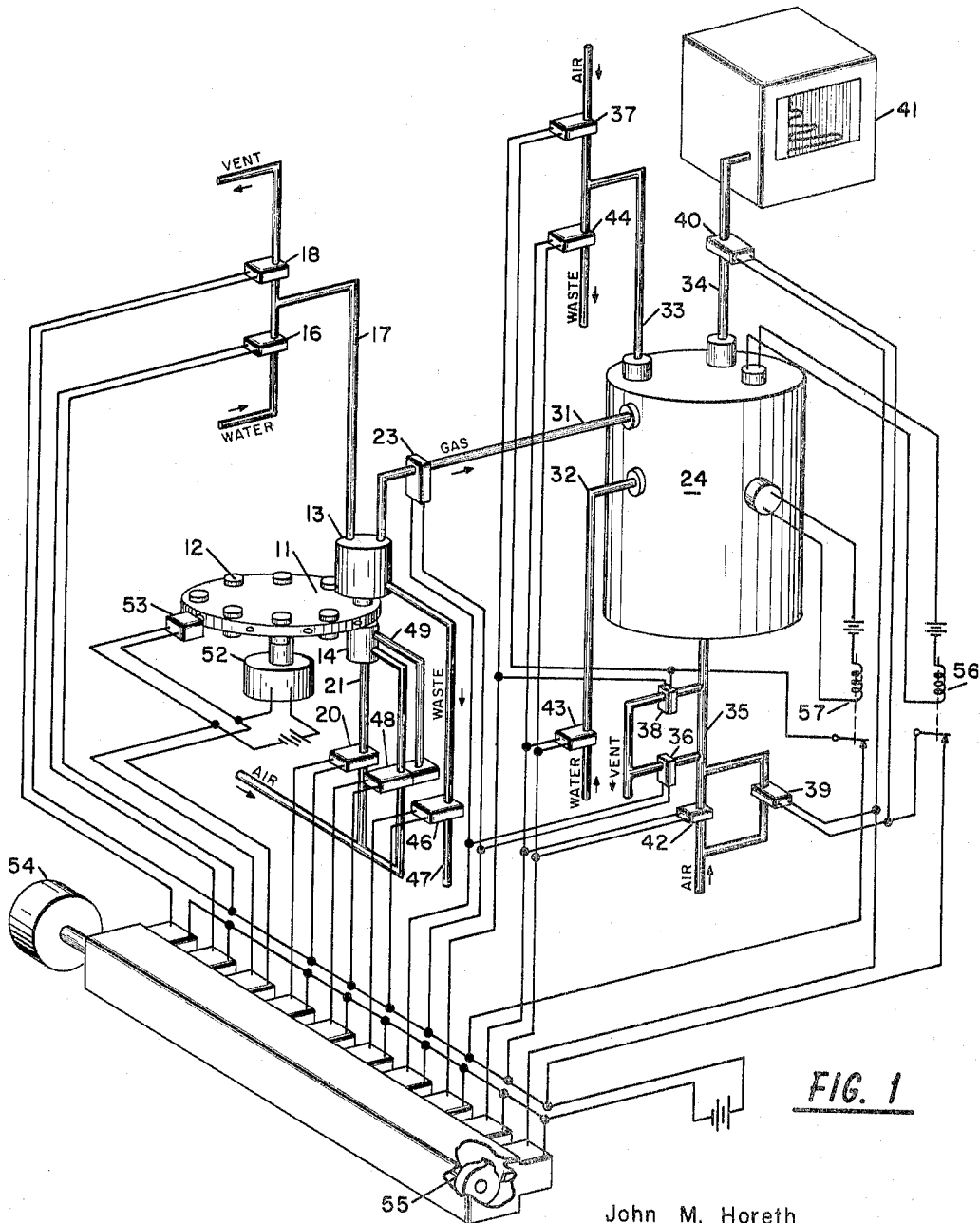
J. M. HORETH ETAL

3,240,068

MUD-GAS SAMPLING SYSTEM

Filed July 12, 1963

3 Sheets-Sheet 1



John M. Horeth
Richard H. Langenheim
William D. Howard
INVENTORS.

BY *Gary C. Honeycutt*
ATTORNEY

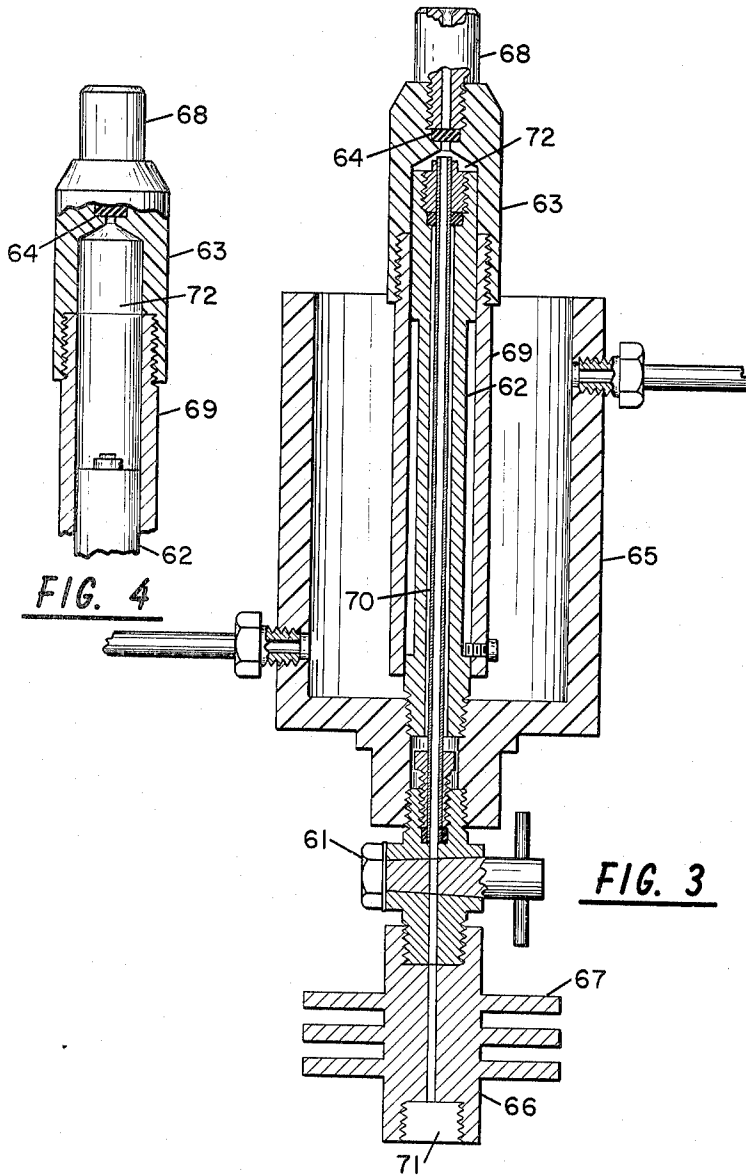
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MUD-GAS SAMPLING SYSTEM

John M. Horeth, Richard H. Langenheim, and William D. Howard, Tulsa, Okla., assignors, by mesne assignments, to Esso Production Research Company, Houston, Tex., a corporation of Delaware

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9 Claims. (Cl. 73—421.5)

This invention relates to the drilling of boreholes in the earth, and more particularly, to the logging of a rotary drilling operation wherein a liquid is circulated downhole for various purposes, including primarily the lifting of cuttings to the surface. Specifically, apparatus is provided for automatically processing and analyzing successive samples of drilling mud to obtain a log of the hydrocarbon gases contained therein.

The apparatus of this invention is a new approach to quantitative mud-gas logging. In particular, the apparatus is designed to automatically analyze small drilling mud samples for their gas contents in terms of methane, ethane, propane, isobutane, normal butane and heavier hydrocarbons, such as heptanes and hexanes, and to record the analysis of these hydrocarbons by means of a recording gas chromatograph.

Various techniques and devices have been employed heretofore in the sampling and analysis of drilling muds. It is known in the art to volatilize hydrocarbons from mud samples and to subject the hydrocarbons to analysis by gas chromatography. A recent disclosure of such practice is found in U.S. Patent No. 3,050,449.

A serious difficulty which we have encountered in the chromatographic analysis of hydrocarbon gases obtained from drilling mud in accordance with prior sampling techniques is baseline drift. The chromatograms obtained have been unreliable due to a persistent failure of the baseline to remain constant and stable throughout a repetitive analysis program. In an attempt to avoid this problem, it has been necessary to delay the analysis of each successive sample for a period of ten to twenty minutes. In actual field operations, such periods of delay are frequently intolerable.

This invention is based in part upon the discovery that such baseline drift is caused by a contamination of the chromatographic column with small amounts of water and heavy hydrocarbons inadvertently introduced therein, along with the gaseous sample. Accordingly, it is a primary object of the invention to provide a sampling apparatus which is capable of supplying a mud-gas sample free of contamination. This and other objects are revealed by the following description of the invention.

The complete system includes a turntable or similar rack for supporting a number of sample containers, a retorting chamber in combination with a device for transferring the mud samples from the turntable containers into the retorting chamber, a collecting unit for receiving the gas liberated by the retorting step, and a recording gas chromatograph. The entire combination is automated by a system of cam-actuated solenoid valves.

A schematic representation of the complete apparatus is shown in FIGURE 1.

FIGURE 2 is a vertical cross-section, showing in detail certain essential features of the automatic system.

FIGURE 3 is a vertical cross-section of an alternate, manually operated gas collecting unit which may be readily adapted for use in the automatic system.

FIGURE 4 is a fragmentary vertical cross-section showing the collecting unit of FIGURE 3, partially filled with gas and with condensed water and oil.

Referring to FIGURE 1, turntable 11 carries a number of sample containers 12, each of which is loaded with approximately 10 cc. of drilling mud. The rotating table is

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operated in such a manner that it will position each of the mud sample containers underneath the gas extraction unit 13 in a regular sequence, with a suitable time interval being allowed between samples. Once a sample container is properly positioned underneath the extraction unit 13, a ram 14 presses the mud sample container against the bottom of the gas extraction unit 13 in such a manner that an air-tight seal is formed by means of O-ring 15 (FIGURE 2). Once the sample container is so positioned, water is injected into the gas extraction unit 13 through valve 16 and line 17. The gas extraction unit is running at a high temperature, for example 350° F., and will therefore heat the water, readily building up approximately 100 p.s.i. pressure inside the vessel. After a suitable time, the steam and air which are now contained in the vessel 13 are vented through valve 18 to the atmosphere.

The reason for injecting water into vessel 13 and allowing it to heat is to eliminate substantially all the air and also to form a partial vacuum in vessel 13 after the steam has been allowed to escape through valve 18. After vessel 13 has been purged, piston 19 of sample container 12 is activated by means of compressed air through valve 20 and passage 21. The compressed air forces piston 19 upward, thus causing the drilling mud sample in container 12 to be introduced into the gas extraction vessel 13, after popping the relief valve 22. After the mud has been introduced into gas extraction vessel 13, it is allowed to heat for at least about two minutes at a temperature of 350° F. The optimum heating time will vary, depending upon the particular type and composition of mud being analyzed. The heating of the mud at this temperature causes the mud to boil and therefore causes the hydrocarbon gases which are contained in the drilling mud sample to be released from the mud into the void space above the mud sample inside the vessel 13.

After a suitable period of heating the drilling mud in vessel 13, valve 23, which had been closed prior to this time, is opened. The opening of valve 23 will allow the hydrocarbon gases and steam vapor from chamber 13 to escape through the passage leading through valve 23 and into gas collecting unit 24.

The gas collecting unit, FIGURE 2, includes a water reservoir 25 containing a movable piston 26. The water reservoir is contained within cooling jacket 27 which is maintained at a constant temperature of approximately 150° F. This temperature is cool enough to permit a rapid condensation of the steam and oil vapors, and yet hot enough to avoid any significant solubility of the gaseous hydrocarbons in the condensed liquids. The gas collecting unit also contains float 28 and electrode pairs 29 and 30, inside water reservoir 25, as well as passages 31, 32, 33 and 34. It should be noted that the electrodes are electrically insulated from cooling jacket 27 and water reservoir 25.

The operation of the gas collecting unit is as follows. At the start of a cycle, movable piston 26 is in position 26a, and float ball 28 is in position 28a, resting against electrode pair 30. At the start of a gas collecting cycle, valve 23 is opened, allowing the gases to be forced into water reservoir 25. This causes piston 26 to move downward from position 26a. It should be noted that reservoir 25 is filled above the piston and is empty below. Air contained in the reservoir below the piston is allowed to escape through passage 35 and valve 36. Since the volume of gases collected from a given mud sample is small, float 28 will come to rest somewhere between its initial starting point 28a and its lowermost position, against electrode pair 29.

While the gases are being forced into water reservoir 25, the water in the cooling jacket is being maintained

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at a constant temperature of approximately 150° F., as has been previously noted. At the end of the gas collecting cycle, valve 23 is closed, valve 37 is opened, and air is passed through valve 37 into passageway 33, which in turn allows the air to go into water reservoir 25. Venting of air contained in the reservoir below piston 26 is continued through line 35 and valve 38. Enough air is introduced into the water reservoir to cause float 28 to rest against the electrode pair 29. The contact of float 28 on electrode pair 29 causes valve 37 to be closed electrically. The purpose of introducing air into reservoir 25 is to assure a constant air-gas volume after the gases have been obtained from the gas extraction unit 13. Constant air-gas volume is necessary to provide quantitative chromatographic analyses having a consistent calibration from one mud sample to another.

After the air-gas mixture has been reduced to a constant volume inside the water reservoir, valves 39 and 40 are operated simultaneously. Valve 40 allows the air-gas mixture to pass from water reservoir 25 into chromatograph 41 through passage 34. To assist in the transfer of gases from the reservoir to the chromatograph, air is introduced through valves 39 and passageway 35, which forces piston 26 upward. The movement of piston 26 in an upward direction also carries float 28 upward. The gas-air mixture is thereby transferred into the chromatograph, until float ball 28 makes contact with electrode pair 30, at which time valves 39 and 40 are closed again. This will stop the transfer of the air-gas mixture from water reservoir 25 into the chromatograph. The gases which have been transferred into the chromatograph are now analyzed for individual hydrocarbon components.

Gas chromatography is a standard analysis procedure and need not be described herein. A suitable gas chromatograph for use in accordance with the present invention is the "Chronofrac" Model VP-I of Fisher Scientific Co., as fully described in Bulletin No. 619.

Since there may be some air-gas mixture remaining in water reservoir 25, it is necessary to purge the chamber, in preparation for the next analysis cycle. The purging of the chamber 25 is accomplished as follows. Valves 42, 43 and 44, FIGURE 1, are opened simultaneously, allowing air to pass through valve 42 into passageway 35, forcing piston 26 against stop 45. While the piston is moving up, water is introduced through valve 43 into passageway 32 above the piston. The introduction of water into the reservoir causes the flushing of any air-gas mixture and oil which may remain in water reservoir 25. The water is allowed to escape through passageway 33 and valve 44. After sufficient water has been passed through the upper portion of the water reservoir, valves 42, 43 and 44 are closed. After the completion of this step, the gas collecting unit is ready for another analysis cycle.

Returning to FIGURES 1 and 2, the gas extraction unit must now be purged of the mud sample which it has just now analyzed. This is accomplished as follows. Approximately 10 cc. of water is introduced into chamber 13 through valve 16, FIGURE 1, and passageway 17. The water introduced into the chamber 13 is heated for approximately 10 seconds. Thereafter, valve 46 is opened, which allows the water and mud contained in chamber 13 to be exhausted through passageway 47 to be suitably disposed of. Since the chamber is operating at 350° and approximately 100 p.s.i., opening of the valve 46 allows adequate purging of chamber 13 of all contaminants which may be contained therein. After the purging of chamber 13, FIGURES 1 and 2, the three-way valve 48 is reversed, allowing air to enter the valve through passageway 49. Air from valve 48 enters through passage 49 into the chamber 50 and pushes piston 51 down. This allows sample container 12 to move in the downward direction, away from gas extraction unit 13, and to rest on turntable 11. A motor 52 will operate turntable 11 to advance the turntable to the next sample container

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position. The turntable and motor 52 are stopped by microswitch 53 to ensure proper positioning of the new sample container 12 underneath the gas extraction unit 13. The overall system, as shown in FIGURE 1, is now ready for the next gas analysis cycle.

The programming of the various valve and switch operations in the above described sequence, is accomplished by timing motor 54 and a set of cams 55 attached to motor 54. The cams in turn will operate a series of microswitches or air valves. In FIGURE 1 the cams are shown to operate microswitches only.

Relays 56 and 57 are controlled by the float ball 28, FIGURE 2. Relay 56 controls valves 39 and 40, while relay 57 controls valves 37 and 38. Gas extraction unit 13 is heated by means of electric cartridge-type heaters 58. Also, the temperature of the gas extraction unit 13 is regulated by means of a thermostat. It should be noted, of course, that heating of this unit can be accomplished by other means, without departing from the scope of the invention. The constant temperature of the water bath, FIGURE 2, is maintained by an auxiliary heater, not shown. It should also be mentioned that while a 150° F. water bath is used in the above embodiment, other temperatures may be used, without departing from the scope of the invention. Also, the 350° F. operating temperature of gas extraction unit 13 may readily be changed to, say for example 400° F. Preliminary experiments indicate that the higher operating temperatures are desirable for effective gas release, particularly with respect to the iso and normal butanes in oil emulsion mud samples. Suitable operating temperatures range from about 212° up to cracking temperatures, which is around 700° F.

FIGURE 3 shows an alternate gas collecting unit which is manually operated in the embodiment shown, and may be readily automated for use in the system of FIGURE 1, as a substitute for collecting unit 24. The essential elements of the alternate unit include valve 61, tube 62, sleeve 63, self-sealing plug 64 and cooling jacket 65. Auxiliary cooling block 66, having fins 67, may be omitted; however, not without some loss of efficiency. Element 68 threadedly engages sleeve 63, and may be removed to replace plug 64 as needed. Construction detail in the drawing reveals the incidental presence of additional elements 69 and 70, the first of which may properly be regarded as a mere extension of sleeve 63. The latter may conveniently be considered an integral part of tube 62, insofar as the essence of the invention is concerned.

In operation, the mixture of steam, oil vapor and hydrocarbon gases expelled from the drilling mud sample is introduced through port 71, passed upward through tube 62, wherein the steam and oil condenses, and is collected in chamber 72. Sleeve 63 is thereby lifted, as shown in FIGURE 4, to accommodate the continued introduction of fluids into chamber 72. The clearance between sleeve 63 and the upper portion of tube 62 is adequate to permit the necessary sliding action, and yet close enough to ensure a fluid seal. The collected gas phase is then withdrawn from chamber 72 by inserting a hypodermic needle through plug 64. The withdrawn sample is then injected into a suitable chromatograph for analysis.

Numerous modifications and other embodiments of the disclosed apparatus will readily occur to those skilled in the art, without departing from the scope of the invention. For example, although the system shown in the drawings is conveniently designed for handling 10 cc. mud samples, it may readily be modified to process smaller or larger samples. Moreover, pneumatic ram 14 may be replaced by a hydraulic or mechanical ram. Other means for liquid level control may be used in reservoir 25 of the gas collecting unit. For example, optical control may be provided by means of a light source and photocell combination. The use of gamma ray absorp-

tion is also a standard technique in liquid level control, and is suitable for the purposes of the invention.

What is claimed is:

1. A gas sample collecting unit comprising a substantially vertically disposed, cylindrical, enclosed chamber having upper and lower ports therein, and containing a piston which is free to move between upper and lower stops intermediate said ports; first and second electrode pairs mounted within said chamber intermediate the upper stop and the upper port; and an electrically conductive float the limiting positions of which are defined by said electrode pairs.

2. A gas sample collecting unit comprising a substantially vertically disposed, cylindrical, enclosed chamber having upper and lower ports therein, and containing a piston which is free to move between upper and lower stops intermediate said ports; first and second electrode pairs mounted within said chamber intermediate the upper stop and the upper port; an electrically conductive float the limiting positions of which are defined by said electrode pairs; and means for cooling said chamber.

3. Gas sampling apparatus comprising a first elongated tubular element having means associated therewith for controlling fluid flow therethrough; a second elongated tubular element surrounding a portion of said first tubular element and extending beyond one end thereof, a substantial length of the bore of said second tubular element slidably engaging a part of the outer surface of said first tubular element to form a fluid seal therewith, that portion of said second tubular element which extends beyond one end of the first tubular element comprising an elastomeric seal designed to permit withdrawal of a collected gas sample; and means for cooling said tubular elements intermediate said flow control means and said one end of the first tubular element.

4. Gas sampling apparatus comprising an elongated tubular element having means associated therewith for controlling fluid flow therethrough; a sealing sleeve surrounding a portion of said tubular element and extending beyond one end thereof, that portion of the sealing sleeve which extends beyond one end of said tubular element comprising a self-sealing elastomeric plug designed to permit withdrawal of a collected gas sample; and means for cooling said tubular element and said sleeve intermediate said flow control means and said one end of the tubular element.

5. Apparatus for collecting a hydrocarbon gas sample from drilling mud which comprises a retort having a first inlet port and a first outlet port, a gas collecting unit having a second inlet port and a second outlet port, a conduit connecting said first outlet port with said second inlet port and having flow control means therein; said gas collecting unit further comprising a sealed chamber equipped with means for regulating its gaseous volume to a fixed limit in response to the introduction of gases through said second inlet port; said regulating means comprising means for diluting the collected gas with an

extraneous gas, first and second vertically spaced electrode pairs mounted within said chamber, an electrically conductive float within said chamber, the limiting positions of which are defined by said electrode pairs, and means actuated by said electrically conductive float for interrupting the dilution at said fixed limit of gaseous volume.

6. A system for automatically processing a series of mud samples in preparation for chromatographic analysis of their gas content which comprises in combination a plurality of sample containers for the temporary storage of preselected mud samples, a retorting chamber and a gas collecting unit; each of said containers and said chamber being mutually adapted to form a temporary, sealed connection upon being urged together with a predetermined alignment; means for successively placing each of said containers in alignment with said chamber; means for forming a temporary, sealed connection between said chamber and each of said containers while each of the latter is aligned therewith; means for transferring a mud sample from each said container to said chamber during the time of said temporary seal; means for heating said retorting chamber whereby the gas content of each successive mud sample is expelled therefrom; means for transferring the expelled gas to said gas collecting unit; means for adjusting each successive gas sample to a constant volume and pressure; and means for ejecting each successive adjusted sample for analysis.

7. Apparatus as defined by claim 6 further comprising means for injecting water into said retorting chamber prior to each introduction of a mud sample therein, in combination with means for venting the resulting mixture of steam and air from said retorting chamber in preparation for the introduction of each successive mud sample into said chamber.

8. Apparatus as defined by claim 6 wherein each of said sample containers comprises a free piston, the stroke of which within the container acts to expell the mud sample.

9. Apparatus as defined by claim 8 wherein said means for transferring each successive mud sample from its respective container into said retorting chamber comprises means for pressing each said container against said retorting chamber to form said temporary seal and means for exerting a fluid pressure against one side of the free piston located within each said container, whereby said piston is forced along a path which expells each said mud sample from said container, and into said retort chamber.

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LOUIS R. PRINCE, *Primary Examiner*.

RICHARD QUEISSER, *Examiner*.