June 1, 1943. H. H. THOMPSON 2,320,681

METHOD OF ANALYZING EARTH FORMATIONS

Filed May 21, 1940 2 Sheets-Sheet 1

Fig. II.

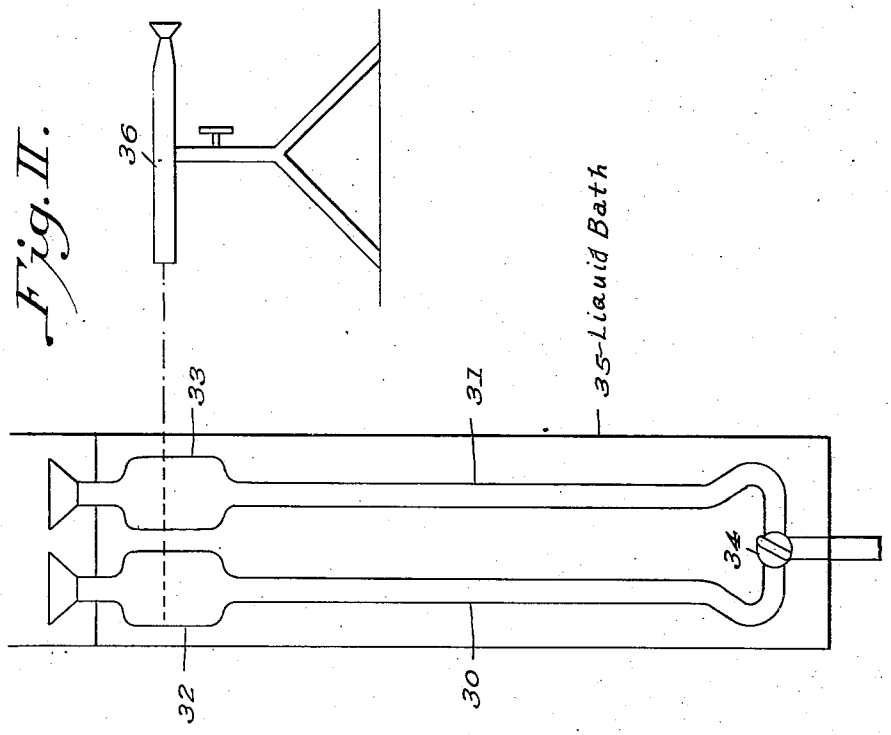
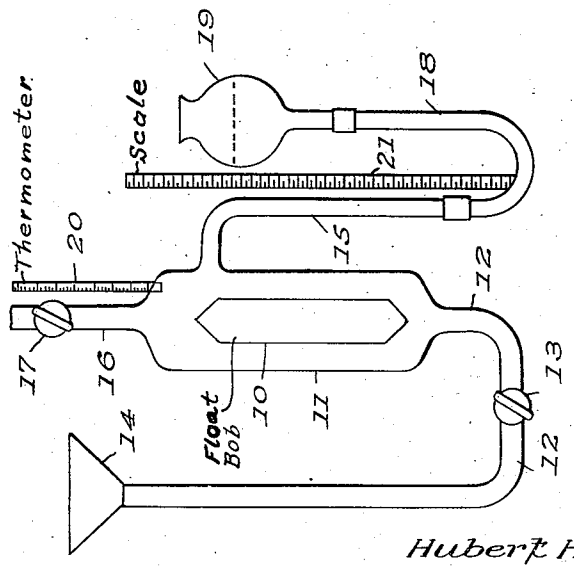


Fig. I.



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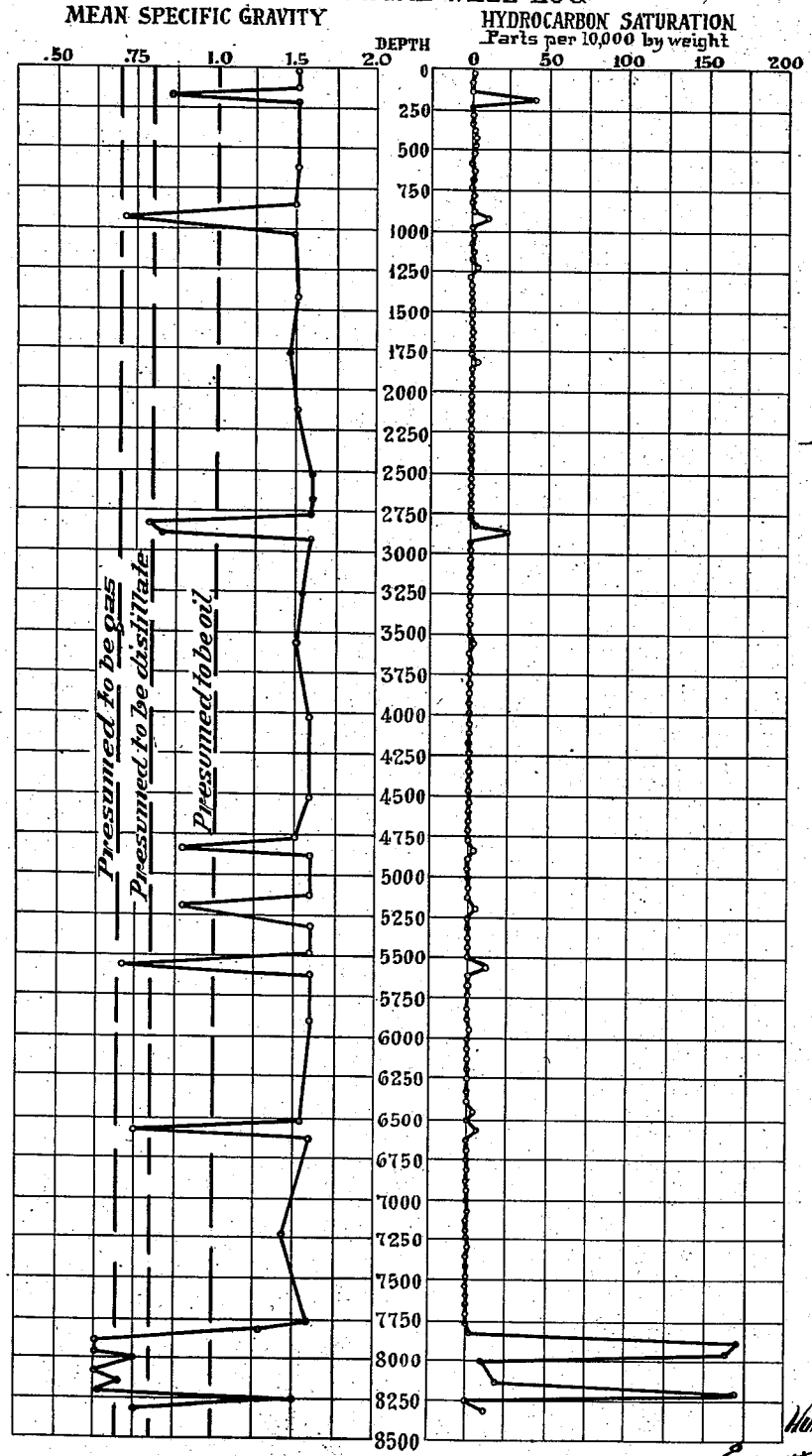


Fig. III.

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Patented June 1, 1943

2,320,681

UNITED STATES PATENT OFFICE

2,320,681

METHOD OF ANALYZING EARTH FORMATIONS

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Application May 21, 1940, Serial No. 336,411

3 Claims. (Cl. 73—51)

This invention relates to geochemical well logging, geochemical prospecting and the analysis of earth formations. It relates more particularly to the determination of the hydrocarbon and water content of drill cuttings from subsurface formations. It also relates, as will hereinafter appear, to the determination of hydrocarbon constituents in soil samples.

It has been found that the presence of various hydrocarbons in drill cuttings from subsurface formations is related to the presence of oil and gas deposits within the particular stratum and in somewhat deeper strata. These constituents which serve as indicia of the petroleum deposits include gaseous, liquid and solid materials. The present invention relates principally to the determination of the liquid, gaseous, and/or solid constituents, notably the former. More particularly, this invention is directed to a specific gravity method of determining the hydrocarbon and water content of cuttings or cores from subsurface formations. Such determinations can be used in preparing a log of a well, for example in order to obtain an indication of the presence and location of oil and gas deposits.

In making a geochemical well log, samples of the cuttings are taken at frequent vertically spaced intervals along a well bore. Samples thus taken are analyzed in one way or another for various hydrocarbon or quasi-hydrocarbon constituents or groups of such constituents and the results for the various samples are compared and plotted in order to obtain a log of the well which indicates the presence and location of oil and gas deposits.

It is an object of my invention to provide a new and improved method of geochemical well logging and geochemical prospecting. Another object of the invention is to provide a particularly rapid method of analysis of core cuttings and surface soils for hydrocarbon constituents. A further object is to measure directly the water saturation and the oil saturation of a core sample from a subsurface formation.

A still further object is to provide a method of this type by which one can readily determine the proportionate quantities of hydrocarbons in a given formation having a relatively low density or specific gravity and those having a relatively high density or specific gravity. It is also an object of my invention to provide a method of well logging wherein the total quantity of hydrocarbons present in drill cuttings taken during a drilling operation can be determined. Other and more detailed objects, advantages and uses of my invention will become apparent as the description thereof proceeds.

Samples of the drill cuttings or cores taken at selected spaced vertical points as previously described are preferably first weighed and crushed to a grain size approaching that of course sand, and then extracted with a suitable solvent. This extraction can be carried out in various ways, for instance by leaching or by use of a Soxhlet apparatus. It is also contemplated that the cuttings as taken from the well can be heated and immersed in a cold solvent which will penetrate the pores and extract the hydrocarbon matter. After the extraction, the solvent extract freed of solid material is placed in a suitable apparatus for determining the relative density of the pure solvent as a reference liquid and the liquid containing dissolved material.

It will be understood, of course, that any solvent or solvent mixture having certain solvent properties for hydrocarbon constituents of earth formations such as drill cuttings can be used, so long as the solvent or solvent mixture chosen has a specific gravity substantially different from that of the hydrocarbon constituents present. Just how different the specific gravities must be is determined by the sensitivity of the apparatus used and by the accuracy desired in the particular analysis. Examples of organic solvents which can be used for the extraction are benzene, cyclo hexane, normal pentane, light naphtha, carbon disulfide, carbon tetrachloride, and the like. If desired, two extractions of separate portions of the sample may be made, one with a relatively low specific gravity solvent and the other with a solvent of relatively high specific gravity. Suitable solvents for the two stage extraction are normal pentane having a specific gravity of about 0.6300 and carbon tetrachloride with a specific gravity of about 1.5835.

Suitable apparatus for the density or specific gravity method of determining hydrocarbons is shown in the drawings, wherein Figures I and II represent partly by diagram, two devices which may be used in following my method of analysis. It will be understood, however, that any method and apparatus which is sufficiently accurate to give comparative data can be used to determine the density or specific gravity of the solvent and solutions. For example, a hydrometer, a pycnometer, a Westphal balance, or a Westphal balance type plummet used on sensitive analytical balances, can be used with considerable accuracy.

Figure III represents a geochemical well log showing the saturation of the formation and the

mean specific gravity of the soluble materials contained therein as a function of the depth at which the samples were taken.

Referring to Figure I, the bob 10 is a relatively incompressible float which has been annealed and carefully ground down so that it will just float in the selected solvent liquid at a standard temperature, for example 75° F. The sensitivity of the bob will depend on its size and can be checked at intervals with the pure solvent, but a volume of about 10 c. c. is satisfactory. The glass chamber or reservoir 11 encloses the bob 10. At its lower end is a U-shaped line 12 having a valve 13 and terminating in a funnel or thistle 14 at a point above the fluid level in the chamber 11. A tube 15 communicates with the reservoir near its top and extends from the wall of the chamber 11. Flexible conduit 18 communicates with line 15 and its free end terminates in thistle 19. The upper end of chamber 11 is provided with line 16 having valve 17. A thermometer 20 may be provided and from its reading a correction made from the known coefficient of thermal expansion, instead of attempting accurate temperature control of the device, although the latter means can be used satisfactorily. Scale 21 can be calibrated to measure the change in height of the liquid in terms of change in pressure to restore equilibrium. On the other hand, it is also possible and usually preferable to indicate the density or specific gravity of the dissolved material directly. Because of the low coefficient of compression, the reading of applied pressure gives a sensitive indication of the amount of compression and hence of the density of the extract before compression to restore equilibrium. The expansion of the reservoir or leaks through the valves are immaterial since the compression depends only on the pressure. Of course, in applying the pressure, valves 13 and 17 are closed.

The glass chamber or reservoir 11 and tube 15 are filled with the fluid under test and normally the float bob 10 sinks to the bottom of 11. Flexible conduit 18 and thistle 19 contain a heavy immiscible fluid such as mercury. With valves 13 and 17 closed, pressure on the fluid in 11 is changed by raising or lowering the column of mercury in 18—19 until the bob 10 remains suspended in the compressed fluid. The heights of mercury and hence the degrees of compression of the fluids necessary to increase the specific gravity enough to float the bob 10 is an indication of the comparative densities of the solvents and extracts.

Another apparatus suitable for use in my gravimetric method of determining hydrocarbons in an extract liquid is shown in Figure II of the drawings. This device comprises a pair of balanced columns 30 and 31 having common stop cock 34 at their lower end and terminating at their free ends with reservoirs 32 and 33. The entire assembly can be immersed in a constant temperature bath; for example, a water bath in cylinder 35 at about room temperature. A cathetometer 36 can be provided for noting and comparing the relative heights of the liquid in the balanced columns.

In this device the specific gravity of the solvents containing the hydrocarbons extracted from the bit cuttings is determined by placing in one arm of the balanced columns of the manometer, with the stop cock closed, a volume of the original pure solvent as a reference fluid and in the other arm, to the same level, the solvent bearing the hydrocarbons. The stop cock is then positioned so as to form a continuous tube and permit the flow of the two fluids to a new level. Likewise, the reference fluid may be a fluid having the same specific gravity as the pure solvent, or some multiple or fraction thereof, and preferably be immiscible therewith.

The proportionate quantities of hydrocarbons having a relatively low specific gravity to those having a relatively high specific gravity can be determined for example, as follows. One portion of the same formation or soil sample may be extracted with a light solvent such as pentane and another with a heavy solvent such as carbon tetrachloride. The mean specific gravity of the hydrocarbons extracted by the two solvents can then be determined by dividing the observed difference in column height when using the one solvent by the corresponding measurement with the other. By referring this ratio to an experimentally calibrated curve, the mean specific gravity is shown.

In one of the simplest applications of my invention, the density of the extracted hydrocarbons is determined for each of a number of samples taken at vertically spaced points. These values can then be plotted or compared for the various samples in order to obtain an indication of the presence and location of hydrocarbon deposits.

The values of the mean specific gravity and the saturation in terms of per cent hydrocarbons extracted from the drill cuttings can be plotted against depth to give a complete log of the well. The specific gravity or relative density ranges which indicate the classification of hydrocarbons the formations are expected to produce have been determined by actual production data obtained in the practice of my invention.

One specific application of my invention to geochemical well logging is shown in the accompanying Figure III which is a log made in the course of drilling a well in Liberty County, Texas. This log shows the saturation of the formations and the mean specific gravity of the soluble materials contained therein, as a function of the depth of the points where samples were taken.

In preparing this log, samples of bit cuttings were taken from the shale shaker screen at intervals of one sample each 50 feet of hole drilled to a depth of 3000 feet. Thereafter samples were taken at intervals of 60 feet. The log was plotted by considering the sample as representing the entire interval and plotting the saturation and means specific gravity values obtained.

These samples were taken from the screen as a heavy paste and immediately sealed in one-half pint jars for transfer to the laboratory for analysis. The samples were prepared for analysis by air drying in the open air for a period of about 48 hours.

The quantity of hydrocarbons in these various samples was determined by leaching weighed quantities of them with standardized amounts of carbon tetrachloride and measuring the difference between the specific gravity of the original solvent and that of the solution. This difference was measured using the apparatus of Figure II, measuring the difference in the height of the columns in the two arms of the manometer to 0.001 mm.

To determine the mean specific gravity of the hydrocarbons, the change in specific gravity of normal pentane as a leaching agent was determined for those samples showing large amounts of light substances extracted by carbon

tetrachloride. This change in specific gravity of the normal pentane solvent was determined in the manner above described in the case of carbon tetrachloride. The ratio of the difference in column heights when using carbon tetrachloride as a solvent to the difference in column heights using pentane as the solvent was referred to an experimentally developed calibration curve to give the mean specific gravity of the hydrocarbons dissolved by the two solvents.

Hydrocarbons having a mean specific gravity from 0.800 to 1.000 were classed as crude oil; those having a mean specific gravity between 0.700 and 0.800 were classed as distillate, while those having a specific gravity of 0.700 or lower were classed as gas. Other classifications can, of course, be used depending on the correlation between the experimental data and the production data in any given area.

The results of a log of this sort can be improved by taking samples during very short time intervals, in order to make them represent exact depth. Also rapid methods of drying the samples with a minimum loss of hydrocarbon content can be used, in order to minimize the time elapsed between the taking of the sample and the availability of the data.

In Figure III the right-hand curve shows the saturation which is the experimentally determined difference between the heights of the columns of the two arms of the manometer of Figure II when using carbon tetrachloride as the solvent. The observed difference in column height is a function of the density of the extract solution and also a function of the weight percentage of the hydrocarbons dissolved therein. The hydrocarbon saturation is expressed as parts per 10,000 by weight.

As previously indicated, the left-hand curve of Figure III is the mean specific gravity of the dissolved hydrocarbons determined as above described.

The log shown in Figure III indicates a small show of light crude or distillate at 2800 feet to 2900 feet and somewhat more important shows of gas at 7830 feet continuing to 8220 feet with a slight show of distillate at about 8100 feet. Thus, the right-hand curve indicates in general the amount of material present, while the left-hand curve indicates its nature.

Earth formations recovered by a core drill can be analyzed very advantageously by the methods above described. Cores and drill cuttings have long been taken from oil-bearing formations or formations which may be oil-bearing and it is desirable to determine the amount of oil present and also something about its probable chemical and physical nature. Previously available methods for determining hydrocarbon and water content have been time-consuming and uncertain, while the method above described can be applied very rapidly and satisfactorily to all earth formations, including cores, drill cuttings, and soil samples.

In the preceding discussion of my invention, it has been described with more or less particular reference to the use of my method in geochemical well logging by the analysis of samples of drill cuttings. It is also applicable, however, to prospecting techniques other than that described and to the analysis of earth formations other than drill cuttings. Thus for example, in making a geochemical survey, samples of the surface soil are taken at points along a survey line or at points spaced over a survey area. The samples thus taken can be analyzed in accordance with my method and the results compared, in order to obtain some indication of the presence and location of deep seated oil and gas deposits which in general correspond to anomalies in the hydrocarbon composition of the surface soils.

My invention has been described with particular reference to the measurement of specific gravities but obviously density defined in any type of unit is satisfactory and, for the purposes of the appended claims specific gravity determination is merely one form of density determination. Also since all that is required in geochemical well logging or geochemical surveying is comparative data, the density units need not always be known, so long as relative densities for various earth formation samples taken from vertically or horizontally spaced points are determined.

While I have described my invention in connection with certain preferred embodiments thereof, it is to be understood that these are by way of example rather than by way of limitation and I do not mean to be restricted thereto but only to the scope of the appended claims in which I have defined my invention.

I claim:

1. In a method of prospecting, the steps comprising taking samples of earth formations, extracting a portion of each of said samples with an organic solvent of relatively high specific gravity, extracting a second portion of each of said samples with an organic solvent of relatively low specific gravity, measuring the density of each of the extracts thus formed, and determining the mean specific gravity of the hydrocarbons extracted from each sample, to secure comparative data indicative of the presence and location of petroleum deposits.

2. In a method of well logging wherein a plurality of samples of earth formations are taken at a plurality of vertically spaced points, the improvement comprising treating at least a portion of each of said samples with a liquid of known density, recovering at least a portion of each of said liquids, separately determining the pressure change necessary to render the apparent density of the recovered liquids equivalent to the initial known density of the treating liquid.

3. In the method of analyzing earth formations for hydrocarbon constituents wherein samples are taken from said formations at spaced points and treated with a solvent for said hydrocarbons, the improvement comprising ascertaining the density of an organic solvent under selected pressure conditions, treating each of a plurality of earth samples with a portion of the organic solvent, determining the density of each of the extracts thus formed, and measuring the change in pressure necessary to render the apparent density of the extract equivalent to that of the organic solvent alone.

HUBERT H. THOMPSON.