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METHOD FOR DETERMINING FLUID CONTENT AND SALINITY OF MOIST SOLIDS

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This invention relates to a method for determining the fluid content of moist solids, and it is more particularly concerned with ascertaining the oil and water content of oil bearing sands.

In the drilling of oil wells, a great deal of information is gained by a careful study and analysis of sands brought to the surface during the drilling process. One important characteristic of the sand, and that with which this invention is principally concerned, is the degree to which available pore space in the sand is filled by the water present therein, and, upon occasion, by any oil in the sand.

The available pore space in a given specimen of a sand, such as may be found in a test core, is determined by conventional methods. However, no satisfactory, yet quick method for determining the respective quantities of water and oil present in a given sample of sand has heretofore been known.

It is accordingly an object of this invention to provide a method and apparatus for determining the fluid content of moist solids.

A further object is to provide an improved method and apparatus for rapidly determining the water content of a moist sample of sand from a well, for optionally determining the respective quantities of both water and oil present therein, and for substantially simultaneously determining the salinity of the determined water content.

I have discovered that in accomplishing the foregoing objects, the water content of moist soils may be rapidly determined with accuracy by a method which is based upon the determination of the chloride salts present in solution in the moist soil, or sand, sample under test. For convenience of description, herein and in the appended claims, the terms "salinity" or "saline concentration," will be used to express the chloride concentration of the sample, and the term "saline solution" will be used to designate an aqueous solution having a chloride solute.

In practicing this method the following steps are carried out.

(*a*) A given sample of the sand or other material to be tested is selected, and the salinity of the sample, or more properly of the aqueous solution present in the sample, is determined. The method which I use is described in a succeeding paragraph.

(*b*) A known volume of water or other chloride solvent is then added to the sample, and is thoroughly mixed therewith. This solvent is normally chloride-free, but in the event the solvent does have a chloride content, the amount thereof is taken into account in making the necessary calculations. The amount of this solvent addition should be such as to materially reduce the chloride concentration, or salinity, of the sample. In the case of well sand analysis, it may sometimes be desirable to dilute the material with a solution of a salt other than a chloride in order not to disturb the existing adsorption equilibrium. It may also sometimes be desirable to dilute the material with a solution of a detergent material, or other material capable of lowering the interfacial tension between oil and water, in order to facilitate mixing of the chloride solvent with the sample.

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(*c*) The saline concentration of the now diluted solution present in the sample is again determined.

Once the salinity, both before and after dilution, of the sample has been determined, the amount of water present in the untreated sample before its dilution may readily be determined by the equation

$$V_1=\frac{C_2V_2}{C_1-C_2}$$

where

V_1=Amount of water originally present in the untreated sample.

V_2=Amount of chloride solvent added in step *b* above.

C_1=Saline concentration of original solution present in the untreated sample.

C_2=Saline concentration of the solution present in the sample after addition of the chloride-free solvent.

In employing the above formula, the units expressing volumes and concentrations need not be related. For example, volumes may be expressed in metric units and concentrations in English units.

I determine the factor of salinity by measuring the potential which results when a chloride-sensitive electrode, preferably a silver electrode having a thin coating of silver chloride fused thereon, and the free end of the salt bridge of a standard reference half-cell (as a calomel half-cell) are placed in contact with the moist sand or other sample under test. Once this potential is known for a particular sand, its salinity may be determined by reference to the curve obtained from data resulting from calibrating the chloride-sensitive electrode and the reference half-cell against known solutions of varying chloride concentration.

Although the above description mentions the use of a silver-silver chloride electrode and a calomel half-cell, it is contemplated that other reference half-cells as well as other chloride-sensitive electrodes could be used instead. As regards the chloride sensitive electrode, care should be taken to employ a metal chloride which is sparingly soluble and which is capable of forming an adherent coating on the underlying metal.

The above description has dealt with the de-

termination of only the water content and the salinity of the sample. When it is desired to learn the weight of oil present in the sample as well, the procedure is somewhat modified. In this case the following sequence of steps should be used:

(1) A given sample of the moist sand or other material is selected and weighed.

(2) The salinity of the solution present in the sample is determined as in step *a* described above.

(3) The solution is then diluted with a chloride solvent as in step *b* described above.

(4) The salinity of the diluted solution present in the sample is determined as in step *c* described above.

(5) The sample is then thoroughly washed with a solvent to remove any oil present therein.

(6) The resulting washed sample is then thoroughly dried to remove all water and other solvent present therein.

(7) The dried sample is then weighed.

The total weight of all liquids, both oil and water, present in the untreated sample, is determined by subtracting the weight of the dried sample (obtained in step 7) from the weight of the untreated sample (obtained in step 1). The water content of the sample is calculated from the data obtained in steps 2, 3 and 4 above, by application of the formula

$$V_1=\frac{C_2V_2}{C_1-C_2}$$

as before. The weight of oil present in the untreated sample is obtained by subtracting this calculated water content from the amount found to represent the weight of all liquids present in the sample. In making this calculation, it may be assumed that no liquids other than water and oil are present in the sands which are normally obtained from wells.

The approximate densities of the liquids present in a given sample of sand are normally known. Accordingly, knowing the weight of each liquid, the volume thereof may be readily computed. If the porosity of the sample be known, the contents of oil and water may then be expressed as fluid saturation in terms of percentage of pore space occupied in the sample.

Example

As an example of the manner in which the invention described above finds application, the following illustration is given. A sample of sand was selected from a well in the Elk Hills district. The sample weighed 17.26 grams. By use of the silver-silver chloride electrode and a calomel half-cell, the salinity of the solution present in the sample was observed to correspond to a chloride concentration of about 2200 grains of sodium chloride per gallon. A quantity of about 3.0 grams of distilled water was added to the sample and thoroughly intermixed therewith. The salinity of the solution was then measured and was found to have decreased to about 560 grains of sodium chloride per gallon as a result of the addition of the distilled water. The sample was then washed several times with a quantity of acetone and toluene to thoroughly remove all traces of oil therefrom. It was then carefully dried under an infra-red lamp. The dried sample was found to weigh 15.73 grams. Applying the formula

$$V_1=\frac{C_2V_2}{C_1-C_2}$$

wherein $V_2=3.00$ cc., $C_1=2200$, $C_2=560$, it is found that $V_1=1.02$ cc., or, assuming a specific gravity of 1.03, 1.05 grams. By difference the oil present in the sample weighed 0.48 gram, which for oils found in this area, represented a volume of about 0.54 cc.

Inasmuch as the sample of sand under test was known to have a grain density of about 2.54 grams per cc. and a porosity of about 35.0 per cent, this represented an available pore space in the sample of about 2.05 cc. Since the sample was found to contain about 1.02 cc. water and 0.54 cc. oil, this represented a fluid saturation with respect to water of about 49.8 per cent, and with respect to oil of about 26.3 per cent, representing a total fluid saturation of about 76.1 per cent.

In this particular case the interstitial water content of the sand was fairly high and the oil content fairly low. Furthermore, the salinity of the interstitial water was high, approximating that of the water found in the oil reservoir. The high salinity of the sample indicates that little or no contamination of the well core by the fresh water used in drilling had obtained, and that the fluid saturations found may be accepted as representative of those in that portion of the reservoir from which the core sample was taken. On the basis of these results it can be inferred that a well tapping the portion of the reservoir in question would be productive of water with only a small amount of oil.

While this invention is described in terms of particular materials and methods of using the same, it is obvious that many modifications and variations in both materials and methods may be made without departing from the spirit and scope of my invention, and only such limitations should be imposed as are indicated in the appended claims.

The invention described herein may be manufactured and used by or for the Government of the United States of America for governmental purposes without the payment of any royalties thereon or therefor.

I claim:

1. The method of simultaneously determining the quantity of water present in and the salinity of the aqueous saline solution in a sample of wet oil sand comprising measuring the potential generated upon the insertion into said sample of both a chloride sensitive electrode and the free end of the salt bridge of a standard reference half cell; providing a measured known quantity of water to be used as a diluent; diluting said sample with said measured quantity of water; and then measuring the potential generated upon insertion of said electrode and said free end into the diluted sample, the salinity of the solutions of said undiluted and diluted sample being functions of said potential measurements, and the quantity of water present in said undiluted sample being given by the formula

$$V_1=\frac{C_2V_2}{C_1-C_2}$$

where V_1 is the quantity of water present in the undiluted sample, V_2 is the amount of solvent added to the sample to dilute the solution present therein, C_1 is the saline concentration of solution present in the sample before its dilution, and C_2 is the saline concentration of the solution present in the sample after its dilution.

2. The method of simultaneously determining the quantity of water present in and the salinity

of the aqueous saline solution in a sample of wet oil sand comprising measuring the potential generated upon the insertion into said sample of both a silver electrode having fused on the surface thereof a coating of silver chloride and the free end of the salt bridge of a standard reference half cell; providing a measured known quantity of water to be used as a diluent; diluting said sample with a known quantity of water; and then measuring the potential generated upon insertion of said electrode and said free end into the diluted sample, the salinity of the solutions of said undiluted and diluted sample being functions of said potential measurements, and the quantity of water present in said undiluted sample being given by the formula

$$V_1=\frac{C_2V_2}{C_1-C_2}$$

where V_1 is the quantity of water present in the undiluted sample, V_2 is the amount of solvent added to the sample to dilute the solution present therein, C_1 is the saline concentration of solution present in the sample before its dilution, and C_2 is the saline concentration of the solution present in the sample after its dilution.

3. The method of simultaneously determining the quantity of water in, the quantity of oil in, and the salinity of the aqueous saline solution in a sample of wet oil sand comprising weighing said sample; measuring the potential generated upon the insertion into said sample of both a chloride sensitive electrode and the free end of the salt bridge of a standard reference half cell; providing a measured known quantity of water to be used as a diluent; diluting said sample with a known quantity of water; measuring the potential generated upon insertion of said electrode

and said free end into the diluted sample; washing said diluted sample with an oil solvent to remove any oil present in the sample; drying said washed sample; and weighing said dried sample, the salinity of the solutions of said undiluted and diluted sample being functions of said potential measurements, the quantity of water present in said undiluted sample being given by the formula

$$V_1=\frac{C_2V_2}{C_1-C_2}$$

where V_1 is the quantity of water present in the undiluted sample, V_2 is the amount of solvent added to the sample to dilute the solution present therein, C_1 is the saline concentration of solution present in the sample before its dilution, and C_2 is the saline concentration of the solution present in the sample after its dilution, and the amount of oil being present in the sample being the difference between the weight of said undiluted sample and the sum of the weight of said dried sample and the weight of the water present in the undiluted sample.

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