

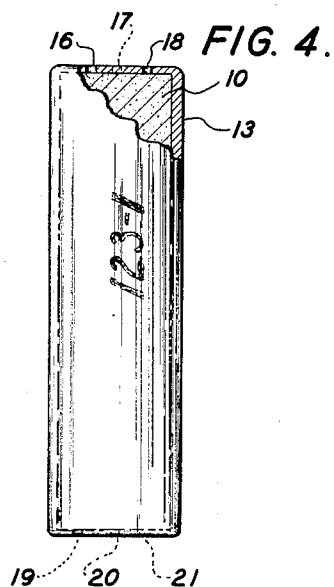
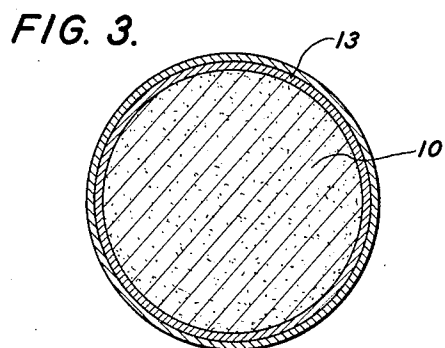
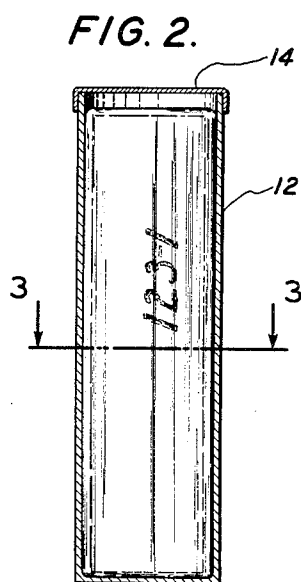
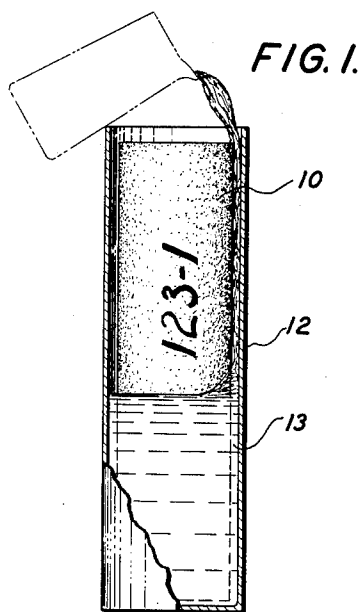
Dec. 15, 1953

F. H. BAILLY

2,662,401

SAMPLING AND HANDLING OF MINERAL SPECIMENS

Filed April 25, 1949



INVENTOR.
FLORENT H. BAILLY

BY

James D. Christie

ATTORNEY

UNITED STATES PATENT OFFICE

2,662,401

SAMPLING AND HANDLING OF MINERAL SPECIMENS

Florent H. Bailly, Pasadena, Calif.

Application April 25, 1949, Serial No. 89,381

8 Claims. (Cl. 73-153)

1

2

This invention relates to sampling and testing methods and more particularly to such methods as employed in mineralogical analysis. A typical field for the application of the present invention is in the sampling and testing of core samples or chip samples from oil and gas and other types of wells. Although not so limited, the invention is described with respect to this use.

In drilling oil, gas and other types of wells, it is conventional practice to take samples of the strata through which the drill bit is passing; these samples, depending upon the type of drill equipment, are known as "core" samples or "chip" samples. By analyzing the same with respect to permeability, porosity, saturation, interstitial water, etc. a great deal can be learned regarding the nature of the particular strata or horizon from which the core was taken. These several tests are generally included in the generic term "core analysis" which term will be used throughout the present specification. The practice of core sampling not only allows the characteristics of a particular well bore to be valued, but, when combined with the same procedures in adjacent and distant wells provides a valuable aid in mapping the sub-surface geology of the region.

The constant aim in obtaining core samples for subsequent core analysis is to employ methods and techniques in the field that will ensure to the greatest possible extent that the core as it reaches the laboratory for analysis will be possessed of its original properties. It is evident that any intervening loss or increase in moisture content or loss in oil content as well as any physical changes due to deterioration or inordinant temperature changes will result in erroneous interpretation of the characteristics of the sub-surface strata from which the core was taken. The present invention is directed in part to improved techniques for core sampling to increase the reliability of data obtainable therefrom.

Present methods of core sampling are many and varied. In general these methods consist of enclosing the sample in a moisture resistant wrapper or immersing the same in a heavy fluid medium, say motor oil and sealing the thus encased core in an air tight container. Such methods do not entirely prevent sample alteration due to atmospheric changes in transportation. Particularly is this so where the samples are shipped long distances by air express or where the atmospheric conditions at the laboratory vary markedly from those prevailing at the drilling site. Changes in temperature and pressure induce

changes in fluid content of the sample through evaporation and recondensation on the walls of the container or wrapper.

The physical structure of the core may be altered if the method of obtaining it requires that it be frozen. Such a method is presently being practiced. Where the sample is immersed in a heavy oil medium, it is impossible thereafter to determine the hydrocarbon-water ratio thereof.

In accordance with the method of the present invention the core sample, chip sample or other mineralogical sample is cast into a chemically and physically inert material which prevents any alteration in the sample during the period of transit from the site to the laboratory. To the sample itself as well as to the fluids contained therein the importance of physical inertness of the casting material is emphasized by the foregoing discussion of changes occurring due to fluctuations in atmospheric temperatures and pressure. When the core sample is encased in the physically inert materials such changes cannot affect it. The properties and characteristics of an ideal casting material in accordance with the invention are as follows:

1. Complete chemical inactivity with respect to sands, shales, clay, minerals, salts, brines, lime, crude oil, hydrocarbon and other gases, etc. normally encountered in drilling or mineralogical exploration work.

2. Physical resistance with respect to changes in atmospheric conditions. This requires that the material possess a certain amount of resiliency to relieve stresses caused by volume changes of fluids and gases within the sample and that the material be non-absorbent and non-adsorbent to sample gases.

3. The material must be capable of shaping itself to the contours of the sample under ambient conditions of pressure existing in the field.

4. The material must adapt itself to handling both in the field and in the laboratory.

A material possessing most closely the ideal characteristics outlined above was found to be a thermo setting plastic comprising an alkyd polyester-styrene copolymer. Other materials will fulfill these conditions to a degree but none have been found which are superior to the co-polymer mentioned. However, the invention is not limited to the use of this particular plastic since others may be found in the future which will satisfy the demands of the method of the invention to the same or greater degree than above mentioned material.

The method of the invention, so far as it per-

tains to obtaining samples from the field is described in conjunction with the accompanying drawing in which:

Fig. 1 is an elevation partly in section showing the method of casting a core sample in the plastic;

Fig. 2 is a sectional elevation showing the encased sample ready for shipment;

Fig. 3 is a section taken on a line 3—3 of Fig. 2; and

Fig. 4 is a sectional elevation of the sample ready for laboratory test.

In obtaining core samples at a well, a core barrel is included in the drilling string so that a cylindrical sample of, say ten feet in length and an inch or more in diameter is obtained from the well when the drill pipe is removed therefrom. At the surface the contents of the core barrel are emptied into horizontal core trays. A suitable sample, or samples, is selected and excess drilling mud is removed therefrom. To this point the method of the invention is in accordance with conventional practice. According to preferred practice of the invention, the specific sample to go through all the laboratory tests is selected and cut to the desired shape at the drill rig. One method of cutting the sample is with a double diamond saw by means of which a cube of rock of predetermined size and regular dimensions can be obtained.

Variations in the above procedures might include lowering the temperature of the core sample so as to decrease the rate of evaporation of the oil or water content. The sample may be cut by any means and need not be a cube although this form is preferred since it permits subsequent measurement of permeability in three directions.

Referring to Fig. 1 the cut sample 13 is identified by suitable marking as 123—1, shown in the drawing, and is inserted in a cylindrical, open top tube 12 containing a layer 13 of liquid plastic. Additional plastic is poured over and around the sample 10 until it is completely encased therein. Thereafter a cover 14 is placed on the tube which is then ready for shipment to the laboratory.

Tube 12 may be made of substantially any material which will withstand the temperature of the casting plastic during polymerization thereof. A thermo-plastic transparent plastic is preferred since it permits observation of the core at all times and since it is light and thus holds shipping weight to a minimum. An acetate-butyrate plastic is an example of a satisfactory material for this purpose.

When the sample arrives at the laboratory, tube 12 is removed therefrom as shown in Fig. 4. A series of novel testing methods, made possible by the plastic jacket 13 around the core, are hereinafter described.

Several considerations are of importance in selecting the conditions under which the core sample is cast in the plastic jacket. Ordinarily no steps need be taken to protect the sample from ambient conditions of temperature and pressure although this may be done if deemed desirable. Of more importance are such factors as gell time of the plastic coating and the temperature rise in the plastic while gelling. The particular type of plastic found to be most suited to the instant application, i. e. the copolymer of an alkyd polyester and styrene, is obtainable commercially as a mixture of the monomers. Upon the addition of a suitable catalyst, say a hydrocarbon peroxide or other per-

oxide, and an accelerator, say cobalt naphthenate, copolymerization sets in and the plastic will gell. The gell time, i. e. the time it takes the plastic to set, is of particular important where-as the relatively longer time required for the plastic to reach its stable state is of little or no importance.

The gell time of the plastic is a function of the amount of catalyst and accelerator added as well as the ambient temperature and may range from as little as four minutes to as high as several hours. It is important to the practice of the present method that the gell time be established at a minimum consistent with other related factors of importance.

Although the catalyst and accelerator are added in the field a certain amount of time is required to perform the operations necessary to cast the sample. The gell time should be sufficiently long to permit of these operations. Additionally, if the gell time is too short stresses and strains will occur which may produce cracking of the casting. One further factor to be considered is the temperature rise in the plastic attendant upon the exothermic polymerization reaction. To preserve the sample in the best possible condition the temperature rise should be held to less than 20° F. This constitutes another limitation on the practicable minimum gell time since the temperature rise is inversely proportional to gell time. All of these factors dictate the type of plastics suitable for the practice of the invention as well as the amounts of catalyst and accelerator which should be used.

The effects of catalyst and accelerator on a commercially available mixture of alkyd polyester and styrene monomers is illustrated in the following tables. The catalyst employed in every case was a commercially available hydrocarbon peroxide and the accelerator was cobalt naphthanate.

TABLE I

Effect of accelerator concentration on setting time

Vol. of Monomer	Percent Catalyst by Volume	Percent Accelerator by Volume	Setting Time in Minutes	Temperature Rise in Degrees F.
50 parts.....	4	1/2	39	between 5-10.
Do.....	4	1	27	Do.
Do.....	4	2	9	Do.
Do.....	4	4	4	40.

From these data it is evident that 4% accelerator by volume is too large an amount in the presence of 4% by volume of catalyst since the temperature rise of 40° F. greatly exceeds the desirable maximum of 20° F. and the setting or gell time of 4 minutes is too short to permit of proper use of the catalyst.

TABLE II

Effect of catalyst on setting time

Vol. of Monomer	Percent Catalyst by Volume	Percent Accelerator by Volume	Setting Time (in minutes)
50 parts.....	3	1	23
Do.....	2	1	29
Do.....	1	1	35
Do.....	1/2	1	62

From these data it is evident that at least 1% by volume of catalyst should be employed where 1% of accelerator is used. No definite limits

can be established for catalyst and accelerator concentrations since their effects are to a certain extent overlapping. In fact the reaction may, if desired, be carried out in the absence of an accelerator. Additionally, the gell time is further effected by prevailing temperatures and particularly by the temperature of the core. Moreover, the particular plastic, as well as the type of catalyst and accelerator used in each application will result in variations.

However, certain criteria can be established whereby anyone skilled in the art can select the proper amount of catalyst or the proper amounts of catalyst and accelerator to satisfy these criteria. Thus the temperature rise in the body of plastic consequent upon exothermic reaction therein should be kept as low as possible and preferably below 20° F. At the same time the setting time should be held at a minimum consistent with the foregoing temperature limitation, required period of use (in workable state) and a resultant structure free of cracks or fissures. In general, setting times of from approximately 8 to 50 minutes have been found satisfactory in practice.

To establish the advantage of the sampling method of the invention over prior practices, a series of core samples were taken in horizontally contiguous pairs. One sample of each pair was handled according to conventional practice by wrapping it in a moisture proof foil and sealing the wrapped sample in an air tight container. The other sample of each pair was cast in plastic in the manner herein described. The samples of each pair were then shipped together to the laboratory and their moisture contents were determined. The results of this test are shown in Table III.

TABLE III

Sample No.	Water Content grams/100 grams of core		Incremental Water in Plastic Cast, Percent
	Foil Wrapped	Plastic Cast	
1.....	4.861	5.152	6.6
2.....	7.994	8.130	1.7
3.....	7.481	7.618	1.8
4.....	2.953	3.034	2.7
5.....	3.240	3.465	6.9
6.....	2.436	2.928	20.0
7.....	4.907	5.097	3.9
8.....	6.359	6.482	1.5
9.....	6.275	6.836	8.9

From these results it is evident that the method of the invention improves the accuracy of the data obtainable from core samples. The conventional method is subject to errors of from 1.5 to 20% which are avoided by application of my method.

Another aspect of the present invention resides in improved laboratory techniques made possible by the application of the above described sampling method. Improvements in laboratory handling are directed primarily to core analysis although similar practices may be applicable in other mineralogical testing procedures. As indicated above the core samples can be so-called chip samples which are frequently obtained by cable tool drilling rather than rotary drilling and are brought to the surface in a bailer rather than in a core barrel.

The presently practiced procedure for obtaining core samples is described above. With respect to handling these samples upon arrival at the laboratory, they are customarily un-

wrapped and removed from the can, cut into sub-sample size and weighed in an extraction thimble. Water is removed quantitatively in an ASTM water determination apparatus and hydrocarbons are removed by Soxhlet extraction for a period of several hours, say 12 to 18 hours. Water and hydrocarbon contents are calculated from weight differences of the sample before and after such treatment. The thus treated sample is then run through a porosimeter to determine porosity. If the sample happens to be friable as is the case of unconsolidated sands, it is necessary at this stage to coat it with a cellulose acetate or similar film to hold it together. If the sample is extremely friable the film of cellulose acetate is insufficient to hold it together through subsequent permeability tests. Further, in the case of small or irregular samples, they must be set in optical pitch to adapt them to the permeability apparatus. The foregoing constitutes the usual complement of core analysis tests although other tests are often required if the sample at this stage is still in condition to undergo such additional tests. In any case it is necessary throughout the analysis to exercise judgment as to whether subsequent tests in the series will destroy the sample unless some special procedure is used to protect it.

I have described in considerable detail above, my new method of obtaining core samples in the field. By practicing this method the test procedures at the laboratory become very simple and are standard for any type of sample, i. e. unconsolidated and friable sands as well as more rugged samples. As previously described, the samples are cut at the site to the correct size for testing and are received in the laboratory completely encased in a jacket of transparent plastic. At the laboratory several holes are drilled in two opposite faces of the jacket sample to allow entry and exit of fluid. A sample prepared for laboratory testing is shown in Fig. 4. The sample comprises core sample 10 cast in plastic jacket 13, container 12 having been removed preparatory to the testing operations. A series of holes 16, 17, 18 are drilled through the plastic jacket at one end and a like number of holes 19, 20, 21 are drilled through the jacket at the opposite end of the sample. Thus prepared, the sample may be subjected to repeated laboratory testing without fear of damage and without requiring any additional protective measures.

By reason of the plastic jacket, I am enabled to employ improved test procedures not heretofore possible, and which greatly reduce the time required for obtaining a given series of test values. In accordance with the improved procedure, the sample, prepared in the manner illustrated in Fig. 4, is weighed once and placed in a pressure washer. A suitable solvent, as for example hexane or toluene, is forced through the sample under pressure for a few minutes. This treatment removes the water and oil quantitatively, the water being recovered in a separate receiver for quantitative measurement and the solvent containing the oil being recovered separately for quantitative measurement of oil content. This pressure washing procedure which may be carried out in 15 minutes or less replaces the presently used ASTM water determination method and Soxhlet extraction which together take from 12 to 20 hours. Further, pressure washing is made possible only because the sample is jacketed and hence is not subject

to disintegration and solvent flow through the sample is confined between the inlet and outlet holes.

Subsequently, porosity and permeability measurements are made with exactly the same technique on all samples whether they be hard, soft, large, small or irregular because the external plastic jacket is of uniform size and shape. No special precautions need be taken for friable or other unusual samples. Throughout this procedure the identity of the sample cannot possibly be lost as the number thereof is marked on the sample prior to casting and is observable through the transparent plastic until such time as the sample itself is destroyed. Further, even after undergoing the porosity and permeability tests the sample remains in excellent condition for any or all of the subsequent special tests which are frequently required as for example, interstitial water, and formation resistivity.

The methods of the invention possess a great number of advantages over the presently practiced methods. A few of these advantages are listed:

1. There is no loss of oil or water from the original sample during shipment and storage by evaporation and condensation since the plastic jacket fits exactly the outer contour of the sample. The sample retains its original properties to a greater degree than heretofore possible since it is exposed to the air only once at the well and thereafter remains encased in plastic throughout its entire progress through the laboratory.

2. The jacketed sample is much lighter and less bulky to ship than the canned or bottled samples. This is of particular benefit when the samples are to be shipped by air express for considerable distances. The sample is suitable for all laboratory operations with a minimum of effort regardless of the fact that its physical properties may make it very difficult if not impossible to handle by present techniques. The testing time is materially decreased from an average of about 20 hours by conventional methods to about one hour by the method of the invention. This becomes important when the completion of a well depends on results from the core laboratory.

3. Any test which is suspected of inaccuracy can be duplicated any number of times without risk of the sample deteriorating or falling apart.

4. Standardization of the sampling method and testing procedures allows the same quality of results to be obtained in the laboratory for all types of samples with personnel of lower training level.

Many other advantages of the invention could be set forth, the foregoing being sufficient to show the benefits to be gained by practice of the invention. In essence, the invention contemplates a sampling method whereby rock samples are cast in plastic as quickly as possible after they are recovered and are retained, as thus encased, throughout the rest of their useful existence. All required laboratory tests are made on the sample while still encased in the plastic by the simple expedient of drilling a number of holes through opposite faces of the plastic into the rock sample. Any type of test may then be carried on without departing from the scope of the invention. Similarly, any type of plastic which meets to a satisfactory degree the conditions herein set forth may be employed to encase the samples. The particular plastic referred to in the foregoing description represents the presently preferred plastic, although superior plastics may be dis-

covered at a later date. The invention is not limited to the type of plastic employed.

These and other modifications of the invention may occur to those skilled in the art without departing from the scope thereof.

I claim:

1. A method for treating a mineral sample which comprises completely sealing the sample shortly after its recovery by placing the sample in a liquid plastic bath and solidifying the bath around the sample to form an impervious plastic case, making holes through the plastic case on opposite sides of the sample, and forcing a fluid through the holes and the sample enclosed in the case.

2. A method for treating a mineral sample which comprises completely sealing the sample shortly after its recovery by placing the sample in a liquid plastic bath and solidifying the bath around the sample to form an impervious plastic case, making holes through the plastic case on opposite sides of the sample, and forcing a solvent through the holes and the sample enclosed in the case to remove constituents of the sample which are soluble in the solvent.

3. A method for treating a mineral sample which comprises freezing the sample shortly after its recovery, and, while still frozen, completely sealing the sample by placing the sample in a liquid plastic bath and solidifying the bath around the sample to form an impervious plastic case, making holes through the plastic case on opposite sides of the sample, and forcing a fluid through the holes and the sample enclosed in the case.

4. A method for treating a mineral sample which comprises sizing the sample to desired dimensions, completely sealing the sample in a liquid plastic bath and solidifying the bath around the sample to form an impervious plastic case, making holes through the plastic case on opposite sides of the sample, and forcing a fluid through the holes and the sample enclosed in the case.

5. A method for treating a mineral sample which comprises sizing the sample to the shape of a right cylinder of desired dimensions, completely sealing the sample in a liquid plastic bath and solidifying the bath around the sample to form an impervious plastic case, making holes through the plastic case on opposite sides of the sample, and forcing a fluid through the holes and the sample enclosed in the case.

6. A method for determining the porosity of a mineral sample which comprises completely sealing the sample shortly after its recovery by placing the sample in a liquid plastic bath and solidifying the bath around the sample to form an impervious plastic case, making holes through the plastic case on opposite sides of the sample, forcing a fluid through the holes and the sample, and determining the porosity of the sample from the rate of fluid flow.

7. A method for treating a mineral sample which comprises marking the sample with an identifying legend, completely sealing the marked sample shortly after its recovery by placing the sample in a bath of a transparent liquid plastic and solidifying the bath around the sample to form a transparent impervious plastic case, making holes through the plastic case on opposite sides of the sample, and forcing a fluid through the holes and the sample enclosed in the case.

8. A method for testing a core sample containing hydrocarbons which comprises sizing the sample to desired dimensions, completely sealing the sample in a liquid plastic bath and solidify-

9

ing the bath around the sample to form an impervious plastic case, making holes through the plastic case on opposite sides of the sample, removing hydrocarbons from the sample by forcing a fluid through the sample by way of said holes, and measuring the removed hydrocarbons.

FLORENT H. BAILLY.

References Cited in the file of this patent

UNITED STATES PATENTS

Number	Name	Date
1,588,164	Brunner	June 8, 1926
2,192,525	Rosaire et al.	Mar. 5, 1940

10

Number

Name

Date

2,293,488	Bays	Aug. 18, 1942
2,296,852	Horner	Sept. 29, 1942
2,300,495	Gerhart	Nov. 3, 1942
2,449,627	Sweeney	Sept. 21, 1948
2,539,355	Reichertz	Jan. 23, 1951
2,617,296	Wisenbaker	Nov. 11, 1952

OTHER REFERENCES

10 Bulletin R. I. 4004 of Bureau of Mines, Dec. 1946, page 3.