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[Continued on next page]

(54) **Title:** METHOD AND APPARATUS TO PREPARE DRILL CUTTINGS FOR PETROPHYSICAL ANALYSIS BY INFRARED SPECTROSCOPY AND GAS SORPTION

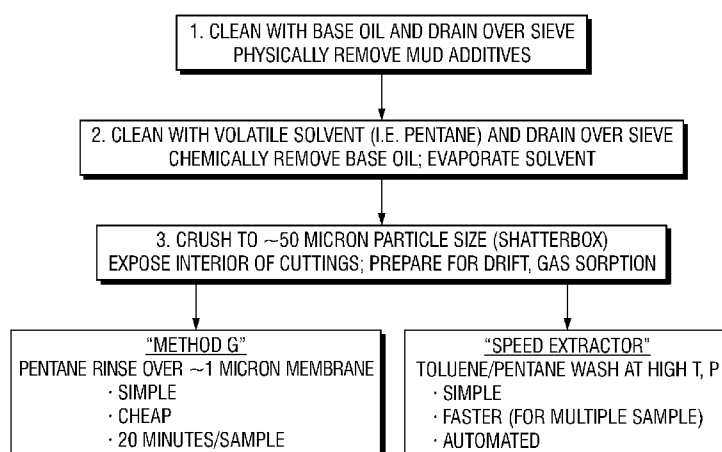


FIG. 2

(57) **Abstract:** Embodiments relate to a method for recovering hydrocarbons from a formation including collecting a formation sample, forming the sample into particles, exposing the sample to a cleaning fluid, and analyzing the sample. Embodiments also relate to a method for recovering hydrocarbons from a formation including the steps of collecting a formation sample, first exposing the sample to a cleaning fluid, forming the sample into particles, exposing the sample to a second cleaning fluid and analyzing the sample.



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GM, KE, LR, LS, MW, MZ, NA, RW, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, RU, TJ, TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

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**METHOD AND APPARATUS TO PREPARE DRILL CUTTINGS FOR
PETROPHYSICAL ANALYSIS BY INFRARED SPECTROSCOPY AND GAS
SORPTION**

Field

[0001] This application relates to methods and apparatus to characterize subterranean formations. Specifically, embodiments described herein use methods to collect, prepare, and analyze solid formation samples.

Background

[0002] Some embodiments may require geomechanical properties of a formation for a variety of reasons without the use of a logging while drilling tool or wireline tool. There may be a need to complement tool failure. A wellbore may be drilled without core data or log information. A drilling regime may include multiple lateral wells from one initial wellbore and the costs for core and/or log data may be unreasonably burdensome. Some embodiments may use a drill string with no tools for logging. Some embodiments may be performed on site in near real time without time for data actualization, that is, the drill string may remain in the wellbore as people timely use the information available to them without remote mathematical analysis and without operating time lag. Some embodiments may manipulate the data in time to guide the completion time. Also, some of the techniques to address these issues, such as laboratory measurements and some logs, require post-analysis, and interpretation of the data that cannot be done within the drilling timeframe.

[0003] Further, while some vertical pilot wells are logged and evaluated in an unconventional play, stimulated horizontal wells are rarely logged or cored. The cost of acquiring the information and/or the associated rig time needed during acquisition (which means that the rig cannot be used for drilling or stimulation elsewhere) are two main reasons for this trend. The solution must be low cost and efficient in terms of delivery times (real or near real-time). It must not introduce any inefficiency into the development program (such as extended rig time for data acquisition) and must be based on a simple workflow that can be carried at the wellsite by non-experts.

[0004] Also, the hydraulic fracturing stimulation of unconventional organic shale reservoirs is performed today in mostly horizontal wells where heterogeneities of

petrophysical and mechanical properties along the well are known to be very significant. Staging requires the identification of sections of the well with both good reservoir quality and good completion quality. Completion quality estimates rely on changes in elastic, rock strength, and stress properties along the well reflect variations (heterogeneity) of mechanical properties along the well.

[0005] Historically, limited information has been collected by FTIR or visual inspection of formation solids under a microscope, especially cuttings with residual drilling mud solvents. More involved analysis have not been selected because of the time and cost for equipment and low likelihood of return of useful information. For example, Figure 1 shows how an FTIR analysis may be distorted by the presence drilling mud fluids. Also, core samples may undergo more sophisticated analysis, and core samples do not have the same exposure to drilling fluids that cuttings samples undergo.

[0006] It is possible to use the drilling cuttings mixed with epoxy in order to perform nano-indentation tests to estimate elastic properties, although this technique may be unpractical for field rig site conditions the procedure to build cuttings epoxy cubes is time consuming and also because nano-indentation tests need to operate in laboratory conditions where mechanical deformation and vibrations are reduced to a minimum.

SUMMARY

[0007] Embodiments relate to a method for recovering hydrocarbons from a formation including collecting a formation sample, forming the sample into particles, exposing the sample to a cleaning fluid, and analyzing the sample. Embodiments also relate to a method for recovering hydrocarbons from a formation including the steps of collecting a formation sample, first exposing the sample to a cleaning fluid, forming the sample into particles, exposing the sample to a second cleaning fluid and analyzing the sample.

FIGURES

[0008] The patent or application file contains at least one drawing executed in color. Copies of this patent or patent application publication with color drawing will be provided by the Office upon request and payment of the necessary fee.

[0009] Figure 1 is an FTIR plot illustrating how the presence of additives may distort results.

[0010] Figure 2 is a flowchart for a procedure for one embodiment of the invention.

DESCRIPTION

[0011] At the outset, it should be noted that in the development of any such actual embodiment, numerous implementation—specific decisions must be made to achieve the developer's specific goals, such as compliance with system related and business related constraints, which will vary from one implementation to another. Moreover, it will be appreciated that such a development effort might be complex and time consuming but would nevertheless be a routine undertaking for those of ordinary skill in the art having the benefit of this disclosure. In addition, the composition used/disclosed herein can also comprise some components other than those cited. In the summary of the invention and this detailed description, each numerical value should be read once as modified by the term "about" (unless already expressly so modified), and then read again as not so modified unless otherwise indicated in context. Also, in the summary of the invention and this detailed description, it should be understood that a concentration range listed or described as being useful, suitable, or the like, is intended that any and every concentration within the range, including the end points, is to be considered as having been stated. For example, "a range of from 1 to 10" is to be read as indicating each and every possible number along the continuum between about 1 and about 10. Thus, even if specific data points within the range, or even no data points within the range, are explicitly identified or refer to only a few specific, it is to be understood that inventors appreciate and understand that any and all data points within the range are to be considered to have been specified, and that inventors possessed knowledge of the entire range and all points within the range.

[0012] The statements made herein merely provide information related to the present disclosure and may not constitute prior art, and may describe some embodiments illustrating the invention.

Definitions

[0013] The reservoir quality (hereafter RQ) is defined by a number of petrophysical and hydrocarbon properties (e.g., porosity, permeability, total organic content versus total inorganic content and maturation, hydrocarbon content and type, gas sorption mechanisms) defining reservoir potential.

[0014] The completion quality (CQ) depends on the poromechanical properties of the field and reservoir, which means the conditions that are favorable to the creation, propagation and containment of hydraulic fractures, as well as the placement of proppant and retention of fracture conductivity. It depends mainly on the intrinsic geomechanics properties, i.e., *in situ* stress field, pore pressure, material properties (elastic, yield or quasi-brittle failure, hardness, rock-fluid sensitivity), their anisotropic nature and their spatial heterogeneities, as well as the presence of discontinuities (such as natural fractures or geological layering) and the orientation of the well. SPE 144326 provides more information for the definitions of RQ and CQ and is incorporated by reference herein.

[0015] Further, as a well is being drilled, the rock that is undergoing the drilling is cut or otherwise fragmented into small pieces, called “cuttings” that are removed from the bulk of the formation via drilling fluid. The process is similar to drilling a hole in a piece of wood which results in the wood being cut into shavings and/or sawdust. Cuttings are representative of the reservoir rock – although they have been altered by the drilling process, they still may provide an understanding of the reservoir rock properties. This is often referred to as “mud logging” or “cuttings evaluation.” For effective logging or evaluation as described below, the cuttings are prepared by removing residual drilling fluids.

[0016] The term “unconventional” is used refer to a formation where the source and reservoir are the same, and stimulation is required to create production.

[0017] The “source” aspect implies that the formation contains appreciable amounts of organic matter, which through maturation or biological processes has generated hydrocarbons (gas or oil, as in Barnett and Eagle Ford, respectively).

[0018] The “reservoir” aspect signifies that the hydrocarbons have not been able to escape and are trapped in the same space where they were generated. Such formations have extremely low permeabilities, in the order of nanodarcies, which explains why stimulation in the form of hydraulic fracturing is needed.

[0019] Bitumen and kerogen are the non-mobile, organic parts of shales. Bitumen is defined as the fraction that is soluble in a solvent (typically a polar solvent such as chloroform or a polarizable solvent such as benzene). Kerogen is defined as the fraction that is insoluble.

[0020] Rock cores are reservoir rocks collected with a special tool that produces large samples with little exposure to drilling fluids.

[0021] Embodiments described herein relate to the field of geomechanics and its application to the oil and gas industry. Geomechanics is an integrated domain linking *in situ* physical measurements of rock mechanical properties via wellbore logging or wellbore drilling, *in situ* hydraulic measurements of *in situ* pore pressure and stress field, surface laboratory measurements on cores to engineering practices for drilling, fracturing and reservoir purposes via the construction of integrated earth models, and modeling tools and workflows.

[0022] Characterization of the mineral (inorganic) and nonmineral (organic) content of formation samples is the objective including weight fractions of inorganic and organic content, total organic content (TOC), and/or mineralogy. Additional information may be obtained via United States Patent Application Serial No. 13/447,109 (Attorney Docket No. IS11.1074-US-NP), entitled RESERVOIR AND COMPLETION QUALITY ASSESSMENT IN UNCONVENTIONAL (SHALE GAS) WELLS WITHOUT LOGS OR CORE by Ridvan Akkurt, Romain Charles Andre Prioul and Andrew E. Pomerantz; and United States Patent Application Serial No. 13/446,975 (Attorney Docket No. IS11.1036-US-NP), entitled METHOD AND APPARATUS FOR SIMULTANEOUS ESTIMATION OF QUANTITATIVE MINERALOGY, KEROGEN CONTENT AND MATURITY IN GAS SHALE AND OIL-BEARING SHALE by Andrew E. Pomerantz; both filed on April 13, 2012 both applications are incorporated by reference herein. One quantity that is generally desired for unconventional is the kerogen content (also referred to as the TOC). TOC can be measured by FTIR. TOC can also be measured by techniques known in the art such as Rock

Eval, acidization followed by combustion analysis (often referred to as LECO) or indirect analysis, Fischer Assay, and many others. However, none of those measurements can be performed successfully without prior cleaning. The reason is that mud additives and base oil (diesel or synthetic) contain organic carbon, and all methods that detect organic carbon will by definition respond to these contaminants. These contaminants therefore must be removed to make a TOC measurement that is representative of the subsurface regardless of which measurement technique is applied. There are similar examples for other quantities (mineralogy, microstructure, porosity, etc), and in each case cleaning is required independent of which analytical technique is employed.

Cuttings Preparation

[0023] Drilling rock cuttings flow from the drill bit to the surface of the well through the circulated drilling mud. The cuttings may be analyzed to estimate many quantities relevant to RQ and CQ, including the maturity, organic matter content (TOC), mineralogy, surface area, pore volume, and porosity. Cuttings samples preparation often historically involves collecting material from a shale shaker, additional sorting via a small hand held sieve, rinsing the material with the drilling fluid base oil, and then exposing the material to hexane. The hexane and other volatile organic material are baked out of the sample in an oven at 80 °C. Soap and water may also be used to remove residual base oil.

[0024] Embodiments of the invention may use a procedure designed to prepare shale cuttings drilled with oil-based drilling fluid for analysis by FTIR and gas sorption as well as other measurements. With these specifications, the goals of the cleaning procedure are as follows:

1. Remove the base oil from the cuttings. Initially, it is desirable to remove the cuttings from the mud, as is necessary for subsequent analysis of the cuttings. Cuttings can be removed from the mud using a shale shaker, which is a vibrating mesh with an opening around 150 microns. Cuttings are collected from the top of the shaker while mud falls through the shaker. The typical base oil is mostly diesel fuel and contains large amounts of organic carbon and aliphatic hydrocarbon; other base oils such as synthetic oil are used occasionally and also contain large amounts of organic carbon and aliphatic hydrocarbon. Some embodiments may use pentane, hexane, heptane, acetone, toluene, benzene, xylene, chloroform, dichloromethane, and a combination thereof Detection of kerogen by FTIR

involves detecting the amount of aliphatic hydrocarbon, so residual drilling fluid will be interpreted as kerogen. Detection of TOC by other techniques such as acidization, Rock Eval, and Fischer Assay involve detection organic carbon, so residual drilling fluids will be interpreted organic matter. Residual drilling fluid remaining in pores will also prevent the gas in the gas sorption measurements from accessing those pores, reducing the measured surface area and pore volume.

2. Remove the mud additives from the cuttings. In particular, some drilling additives also contain large amounts of organic carbon and aliphatic hydrocarbon and thus will be interpreted as organic matter in the FTIR, acidization, Rock Eval, Fischer Assay and other measurements. Other mud additives will be interpreted as minerals that may be indigenous to shale, which will harm measurements of mineralogy such as FTIR, XRD, XRF, EDX, WDX, etc.

3. Do not alter the composition of the cuttings. The goal is to measure the properties of the cuttings in a manner that is representative of their state in the reservoir, so the cleaning process should not alter those properties. Most importantly, the cleaning procedure should not alter the mineralogy, kerogen content, maturity, surface area, pore volume or porosity. An optional goal is to remove as little bitumen as possible. One of the goals of the mud logging is to estimate the kerogen content of the shale, so the preparation process should not destroy the kerogen. This is mostly straightforward because kerogen is insoluble in any solvent. Estimating the bitumen content is somewhat desirable; however, the bitumen content is typically an order of magnitude smaller than the kerogen content and estimation of the bitumen content is of secondary importance. Additionally, bitumen can be dissolved by drilling mud, so in some cases the bitumen may be mostly removed by the time the cuttings reach the surface.

4. Reduce the cuttings particle size to around 10 micron. This particle size is required for many analyses, including FTIR and gas sorption. Briefly, large particles result in specular rather than diffuse reflection, complicating the interpretation of reflection FTIR measurements. With respect to gas sorption, the low permeability of shale necessitates small particle size to speed up the measurement.

[0025] A detailed procedure for preparing shale cuttings drilled with oil-based drilling fluid for analysis by FTIR and gas sorption is below. The procedure could be carried out at

the well site or in the laboratory. Figure 2 is a flow chart of one embodiment of the procedure.

1. Collect cuttings from the shale shaker on a sieve. The sieve must have a pore size about the same as that of the shale shaker.
2. Rinse the cuttings with the base oil of the drilling fluid. The base oil of some embodiments may include diesel, mineral oil, paraffin oil, and synthetic oils such as ester and olefin oils. This step removes residual mud additives mixed with and loosely attached to the cuttings. Large quantities of base oil are typically readily available on the rig floor for this purpose. The rinsing should last for a couple of minutes. For example, one might rinse the cuttings over the sieve until the rinsate appears free of particulate contamination. Box 1 of Figure 2 lists cleaning the cuttings with a base oil and draining the cuttings over a sieve to physically remove the mud additives.

For this step and step 3 below, the sieve may be a hand held device that is not automated. For some embodiments, the sieve may be an automated shaking instrument, such as a rock tumbler. Further, a surfactant may be selected for both of these steps. The surfactant may include ethylene glycol monobutyl ether or a similar surfactant.

3. On the same sieve, rinse the cuttings with a solvent such as pentane. The purpose of this step is to remove enough of the base oil from the surface of the cuttings that the cuttings can be crushed (see the following step; wet cuttings form a mud during crushing and do not crush well). The rinsing should last for a couple of minutes. Pentane is ideal for this step for two reasons. First, pentane is volatile, meaning that it will evaporate quickly after being used to clean the cuttings. Hence, without requiring an additional step, pentane evaporates, resulting in a sample that is sufficiently dry for crushing. Second, pentane dissolves diesel, but it does not dissolve kerogen (no solvents do) and also does not dissolve much bitumen. Bitumen is a complex mixture of compounds with a wide range of solubilities, and selection of a solvent that dissolves diesel but does not dissolve any fraction of bitumen is impossible. However, bitumen is dominated by resins and asphaltenes, neither of which are dissolved by pentane, meaning that pentane dissolves only a small amount of bitumen. Other common laboratory solvents that will suffice for this application include hexane, heptane, acetone, toluene, benzene, xylene, chloroform, dichloromethane, etc. Figure 2 lists box 2 to clean the cuttings

with a volatile solvent such as pentane and drain the cuttings over a sieve. This is to chemically remove the base oil and evaporate the solvent.

4. Crush the cuttings to a particle size of around 10 micron. This is the optimum size for gas sorption (small enough to allow measurement in reasonable time but not so small as create additional surface area) and for FTIR (small enough to minimize specular reflection in reflection mode and to minimize scattering in transmission mode). The crushing should be accomplished with an automated instrument such as a ShatterboxTM (commercially available from SPEX SamplePrep of Metuchen, NJ). The crushing could be accomplished with a mortar and pestle, but the reproducibility of this processing is not comparable to automated techniques. This crushing could also be accomplished with a mixer mill such as Mixer Mill MM 400 which is commercially available from Retsch of Newton, PA. Figure 2 lists box 3 which describes crushing the particles to approximately 50 microns or less to expose the interior of the cuttings and prepare for DRIFT or gas sorption analysis. Some embodiments may use any method to reduce the size of the sample such as crushing, grinding, shaking or a combination thereof

5. Rinse the cuttings again with solvent. The initial rinsing steps are unlikely to remove most of the drilling fluid that invaded the cuttings while the cuttings were in the borehole: the drilling fluid invaded the cuttings for 1 - 3 hours (the average time required for the cuttings to reach the surface) and did so at elevated temperature (resulting in low viscosity) and elevated pressure (resulting in enhanced saturation), while the initial rinses last only a few minutes and occur at ambient temperature and pressure. It is likely that the effect of temperature and pressure support more thorough invasion of drilling fluid than solvent achieves under ambient conditions. After crushing, the characteristic length of the particles is reduced by orders of magnitude, which promotes more efficient cleaning. So the purpose of this step is to clean the cuttings more efficiently than can be performed with the steps 1 - 3 above. Below are two possible techniques for accomplishing this step:

a. Cleaning over a vacuum filter (A vacuum filter is a standard piece of equipment in a chemistry laboratory. It involves a fritted piece of glassware, with a filter membrane resting on it.). Because the cuttings have been crushed to 10 micron, a filter membrane with a smaller pore size required (sieves are not an option here because sieves with openings below 10 micron are not available). An example filter membrane that is readily available is a 0.45 micron polycarbonate filter membrane.

Below the frit is a volume evacuated by a pump. The cuttings are placed on top of the filter membrane at atmospheric pressure, solvent is added and the vacuum on the other side of the frit forces the solvent to flow through the cuttings. This process efficiently removes residual drilling fluid from the cuttings because of the small particle size. Pentane is the optimum choice of solvent for the same reasons as above. Alternative solvents listed above may be selected for this step. Figure 2 lists a Method G box which uses a pentane rinse over a 1 micron membrane which is simple, cheap, and takes approximately 20 minutes per sample.

b. Cleaning at elevated temperature and pressure. Cleaning at elevated temperature and pressure can be achieved in an instrument such as the SPEED EXTRACTORTM manufactured by Buchi of Newcastle, DE lowers the viscosity, allowing the solvent to invade the particles quickly: high temperature also increases the solvating power, allowing the diesel to be dissolved more easily: high pressure forces the solvent into the cuttings more quickly: high pressure also allows the temperature to be increased beyond the atmospheric-pressure boiling point without vaporizing, allowing further increase in temperature. Combined with the small particle size, this technique cleans the cuttings quickly and effectively. However, this technique is more likely to remove bitumen. If retaining bitumen is not a goal, this step could be performed with powerful solvents such as toluene that will remove drilling fluid even more effectively. Example operating conditions include using toluene as a solvent, at 150 C temperature and 50 bar pressure for approximately 30 minutes. This technique can be handled in an automated way, requiring only a few minutes of operator time. Taking advantage of the automation, a quick final rinse with a volatile solvent such as pentane can be applied after the toluene rinse to accelerate evaporation. Another advantage of this technique is that these conditions can dissolve mud additives that are not dissolved in room temperature solvent (save for very long exposure times) thereby removing mud additives beyond those loosely attached to the cuttings. This technique can also be performed on multiple samples at once. Figure 2 lists a SpeedExtractor box which uses a toluene and/or pentane wash at temperatures or pressures higher than the sample temperature which is simple, faster for multiple samples, and automated.

[0026] Some embodiments may benefit from exposing the sample to a second cleaning fluid and using vacuum filtration and/or solvent extraction. In some embodiments, the extraction occurs at higher temperature and/or higher pressure than the sample temperature and pressure. Some embodiments may have a final rinse with a volatile solvent.

[0027] After completing these steps, the cuttings are sufficiently clean, have the correct particle size and have retained their kerogen and bitumen. They are now ready for analysis of maturity, organic content, mineralogy, surface area, pore volume, porosity, etc by instruments such as FTIR and gas sorption among many others. Additional tests may include infrared spectroscopy, TOC analysis by acidization, Rock Eval, Fischer Assay, XRD, XRF, WDX, EDX, gas sorption, pyconometry, and porosimetry.

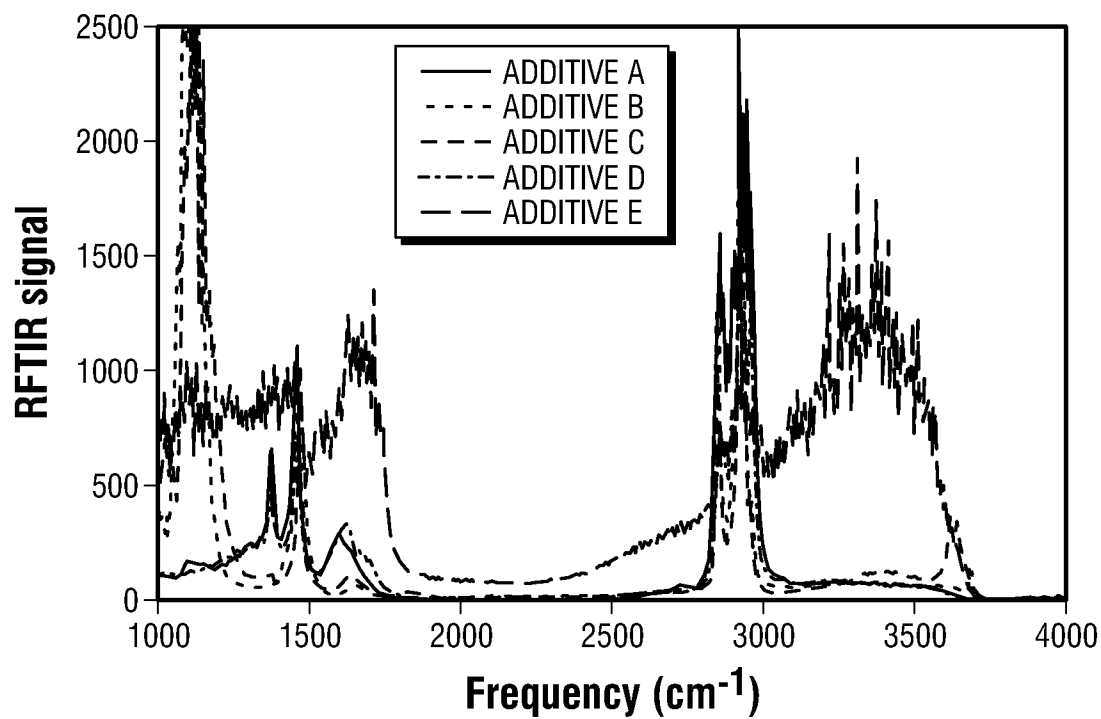
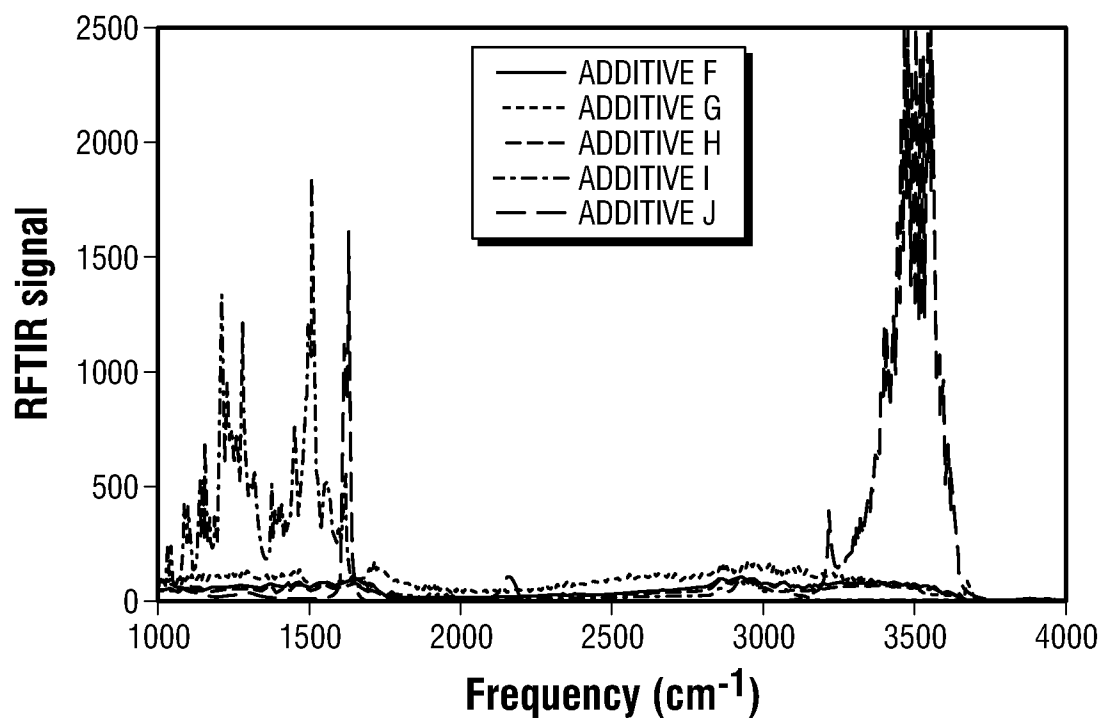
[0028] Time and location are important considerations for embodiments of this procedure. The analyzing occurs in less than an hour and/or in less than 24 hours in some embodiments. The analyzing occurs before recovering hydrocarbons begins in some embodiments or after producing hydrocarbons begins in some embodiments. The analyzing may occur during reservoir characterization during production. Some embodiments may use equipment within 500 meters of a wellbore. In some embodiments, analyzing occurs while drilling the formation.

We claim:

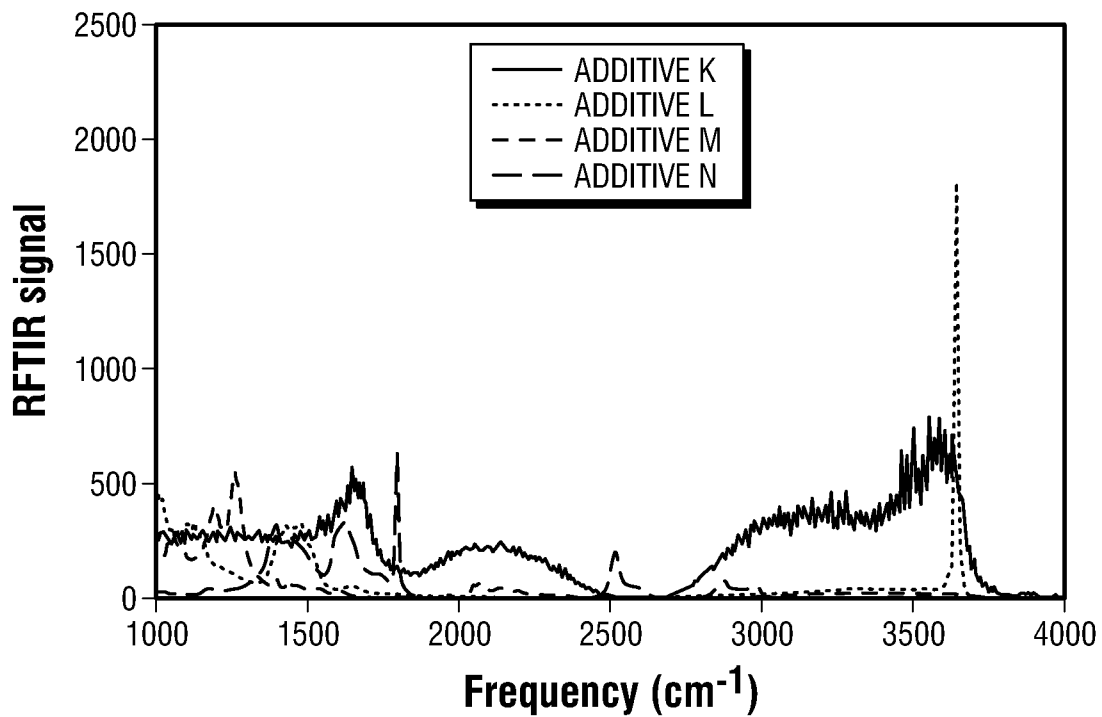
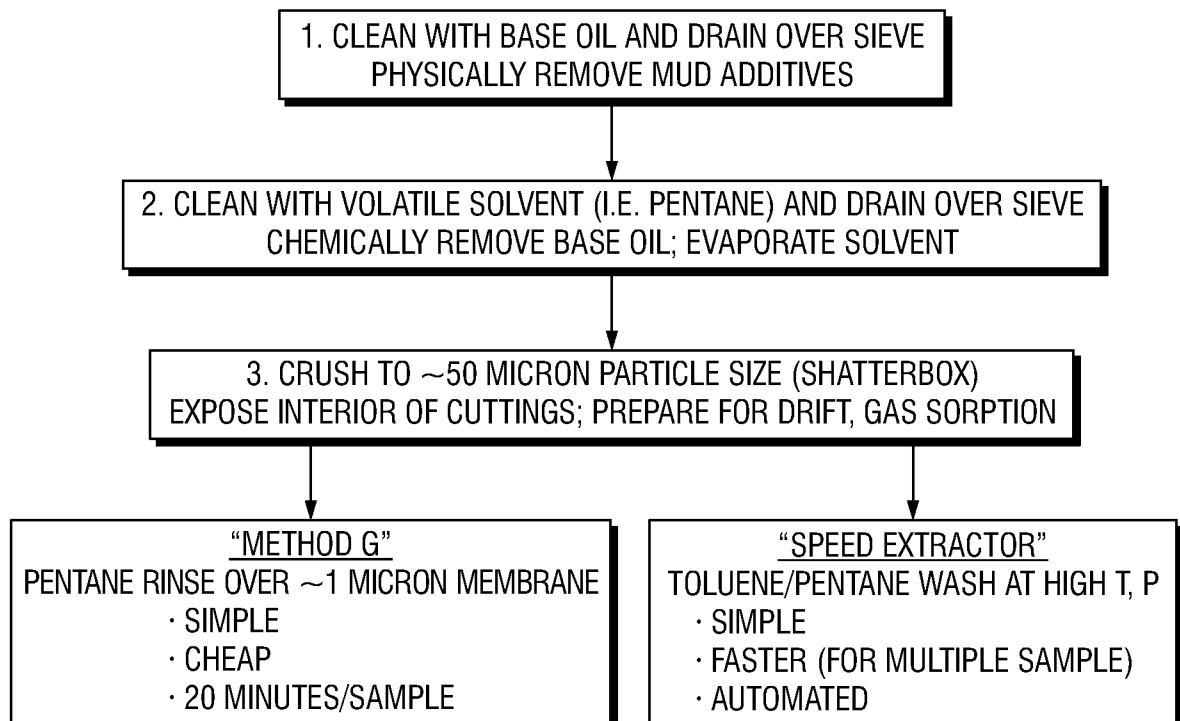
1. A method for recovering hydrocarbons from a formation, comprising:
collecting a formation sample;
forming the sample into particles;
exposing the sample to a cleaning fluid; and
analyzing the sample.
2. The method of claim 1, further comprising exposing the sample to a second cleaning fluid.
3. The method of claim 1, wherein the forming the particles comprises forming the sample to a diameter of about 10 micron.
4. The method of claim 1, wherein the forming comprises crushing, grinding, shaking or a combination thereof.
5. The method of claim 1, wherein the analyzing a sample is selected from a group consisting of infrared spectroscopy, TOC analysis by acidization, Rock Eval, Fischer Assay, XRD, XRF, WDX, EDX, gas sorption, pyconometry, and porosimetry or a combination thereof.
6. The method of claim 1, wherein the formation sample is a solid collected while drilling the formation.
7. The method of claim 1, wherein the sample comprises cuttings.
8. The method of claim 1, wherein the cleaning fluid comprises a base oil of a drilling fluid.
9. The method of claim 1, wherein the cleaning fluid is selected from the group consisting of pentane, hexane, heptane, acetone, toluene, benzene, xylene, chloroform, dichloromethane, and a combination thereof.
10. The method of claim 1, wherein the cleaning fluid comprises surfactant.
11. The method of claim 10, wherein the surfactant comprises ethylene glycol monobutyl ether.

12. The method of claim 1, wherein the collecting further comprises a first rinse comprising a handheld sieve.
13. The method of claim 12, wherein the first rinse involves an automated shaker such as a rock tumbler or mixer mill.
14. The method of claim 1, where the analyzing occurs in less than an hour.
15. The method of claim 1, wherein the collecting, analyzing, estimating and performing occur in less than 24 hours.
16. The method of claim 1, wherein the analyzing occurs before recovering hydrocarbons begins.
17. The method of claim 1, wherein the analyzing occurs after producing hydrocarbons begins.
18. The method of claim 1, wherein the analyzing occurs during reservoir characterization during production.
19. The method of claim 1, wherein the collecting and analyzing use equipment within 500 meters of a wellbore.
20. The method of claim 1, wherein the analyzing occurs while drilling the formation.
21. A method for recovering hydrocarbons from a formation, comprising the steps of:
collecting a formation sample;
first exposing the sample to a cleaning fluid;
forming the sample into particles;
exposing the sample to a second cleaning fluid; and
analyzing the sample.
22. The method of claim 21, wherein the exposing comprises a volatile solvent.
23. The method of claim 21, wherein the exposing the sample to a second cleaning fluid comprises vacuum filtration.
24. The method of claim 21, wherein the exposing the sample to a second cleaning fluid comprises solvent extraction.

25. The method of claim 24, wherein the extraction occurs at higher temperature and/or higher pressure than the sample temperature and pressure.
26. The method of claim 24, wherein the exposing the sample to a second cleaning fluid comprises a final rinse with a volatile solvent.

1/2**FIG. 1A****FIG. 1B**

2/2

**FIG. 1C****FIG. 2**

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US2013/036520**A. CLASSIFICATION OF SUBJECT MATTER****E21B 49/02(2006.01)i, E21B 49/00(2006.01)i**

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

E21B 49/02; G01N 25/14; E21B 21/00; E21B 49/00; G01N 33/24; E21B 21/06; B01D 11/02; E21B 37/06; C10G 1/00

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Korean utility models and applications for utility models

Japanese utility models and applications for utility models

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

eKOMPASS(KIPO internal) & Keywords:: sample, preparation, clean, rinse, pentane, solvent, and petrophysical

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 0915331 B1 (SAUDI ARABIAN OIL COMPANY) 02 November 2011 See paragraphs [0002], [0006], [0034]–[0038] and claims 17, 18.	1,3–7,12–20
Y		2,8–11,21–26
Y	WO 91–13232 A2 (CONOCO INC.) 05 September 1991 See page 2 line 20 – page 3 line 29, page 6 lines 22–35, page 7 lines 12–23, page 9 lines 30–34.	2,9–11,21–26
Y	WO 01–73253 A1 (BJ SERVICES A.S.) 04 October 2001 See abstract and claim 4.	8
Y	WO 2011–082209 A1 (EXXONMOBIL UPSTREAM RESEARCH COMPANY et al.) 07 July 2011 See paragraph [0205].	23
A	US 4979393 A (LEUNG et al.) 25 December 1990 See abstract and claims 1–7.	1–26



Further documents are listed in the continuation of Box C.



See patent family annex.

* Special categories of cited documents:

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"P" document published prior to the international filing date but later than the priority date claimed

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"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search

12 July 2013 (12.07.2013)

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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.

PCT/US2013/036520

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