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(19)



(54) METHOD AND APPARATUS FOR SOURCE ROCK ANALYSIS

(71) We, CONTINENTAL OIL COMPANY, of 1000 South Pine Street, Ponca City, Oklahoma, United States of America, a corporation of the State of Delaware, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates generally to a process for source rock analysis. More particularly, the invention relates to a process and apparatus for quantitative identification of hydrocarbon constituents which may be present in source rock samples.

There is no prior art known to applicants which has the capability of analyzing and providing quantitative data on organic constituents in fine-grained sediments and rocks in terms of ppm paraffins, aromatics and polar compounds, while performing such analysis rapidly to derive meaningful data. Utilization of the present invention enables complete data compilation relative to the desired source rock constituents and the analysis is carried out with what may be termed small sample input.

The source rock determination is carried out through the utilization of complex and relatively expensive equipmentation, including digital data processing structure, and this factor coupled with the necessary expenditure of man hours in overseeing an analysis procedure tend to make the process performance difficult of attainment for individuals or lesser corporate entities who are unable or unwilling to make the necessary expenditures. Therefore, it has evolved that those entities able to afford such equipmentation, utilize the apparatus and process in order to offer a service whereby drillers, exploration companies and the like merely need to supply core, bit cuttings, outcrop specimens, sediments, or soil samples of source rock, in return for which they can receive complete source rock analysis data

indicating hydrocarbon characteristics and indications of the nature and quality of petroleum products relating to the particular source rocks.

Prior art methods of source rock analysis were of a sectional or individualized nature whereby source rock extractions were chemically treated in suitable manner to enable column chromatography methods in order to identify a specific constituent substance of interest. The inherent requirements of such column chromatography limits the versatility of the method and requires numerous and repeated individual chromatographic analysis runs which are yet multiplied by the number of individual samples which are to be examined, column chromatographic procedures are time-consuming and present limitations as to the extent and completeness of final data.

The present invention utilizes specific preparations procedure for a plurality of source rock samples, and sample analysis is performed through the utilization of thin layer chromatography. The process is carried out in a plurality of phases which include specialized preparation of initial samples, extraction of sample material, extract analysis through thin layer chromatography, and compilation and calculation of specialized data relating to specific compounds to be found in the sample rock. Data determined in the source rock analysis is also evaluated relative to data derived through organic carbon determination and paleotemperature determination, ancillary analyses which do not actually constitute a part in the present invention.

Therefore, it is an object of the present invention to provide a method for rapid and accurate determination of specific hydrocarbon make-up within a source rock sample.

It is yet another object of the present invention to provide a source rock analysis procedure wherein a considerable plurality of rock or sediment samples can be quickly

analyzed and evaluated with output of properly integrated information.

It also is an object of the invention to provide a highly accurate data derivation process utilizing thin layer chromatography.

It is still another object of the present invention to provide a source rock analysis system which yields more complete data relative to the source rock subterrain.

Finally, it is an object of this invention to provide a digital data processing system operating in coaction with thin layer chromatographic equipment in order to generate most complete data pertaining to the source rock situs.

These objects of the present invention are hereinafter more fully explained in reference to the accompanying drawings wherein:

Fig. 1 is a flow diagram illustrating the steps and operations of the source rock analysis system;

Fig. 2 is a plan view of a thin layer chromatographic plate;

Fig. 3 is a top plan view of a sample spotting block assembly;

Fig. 4 is a plan view of an indexing cover plate;

Fig. 5 is an end view of the spotting assembly with TLC plate, indexing cover plate and solvent trough in operative position;

Fig. 6 is an enlarged plan view of a single sample track, a plurality of which are transversely disposed across the plate of Fig. 2;

Fig. 7 is a block diagram illustrating the thin layer chromatographic equipment in combination with output recording and data analysis equipment;

Fig. 8 is a graphic representation of densitometer time-analog output;

Fig. 9 is a flow diagram of a TLC analysis data processing program;

Fig. 10 is a flow diagram of continuation of the program;

Fig. 11 is a flow diagram of continuation of the program;

Fig. 12 is a flow diagram of continuation of the program;

Fig. 13 is a flow diagram of continuation of the program;

Fig. 14 is a flow diagram of continuation of the program;

Fig. 15 is a flow diagram of continuation of the program;

Fig. 16 is a flow diagram of a sub-routine part of the program; and

Fig. 17 is a flow diagram of a sub-routine part of the program.

Referring now to Fig. 1, sample selection stage 10 is the first undertaking of the process and this stage should include sample cutting and selection as would be done by a skilled geologist or technician. It is desirable that an E-log or lith-log be used when describing and identifying the litho-

logy of cutting samples in order that cavings and other anomalous variances can be recognized and discarded.

An "E-log" is common nomenclature which refers to an electrical log which is the measure of resistivity of a formation as plotted against the depth the measurement is taken in a bore hole. The log is taken by inserting into the bore hole electrodes which have voltage applied between them. A comparison of the voltage and the current which flows will give the resistivity of the formation at that particular depth.

A "lith-log" is a lithologic or geological description of a formation with respect to depth. The log is obtained by sampling the cuttings from the drill bit as it progresses into the hole. Examination of the samplings will give some idea as to the particular formation the drill bit is presently penetrating. The results of the analysis of the samples are plotted with depth, resulting in a "lith-log".

It is also important that sample contaminants such as indigenous tarballs, gas-ket materials from well-drilling equipment, drilling muds and additives, salt, urea and other unique additives also be recognized and removed from the sample materials. These source rock samples may constitute such as core or bit cutting samples as removed from down hole at a drilling site, or rock specimens collected from outcrops, and the locations at which each of the plurality are taken will be determined by a geologist or one skilled in the art so that the sample material will give best overall indication.

After sample selection and delivery for the analysis process, sample preparation takes place as shown in stage 12 and this step includes sample grinding and weighing. Rapid sample grinding may be performed utilizing a laboratory grinder such as the Bleuler Grinder, a well-known type of Swiss grinding machine. This type of grinder can perform rapid grinding to render a relatively large amount of sample to sixty mesh size, i.e. 246 microns. After grinding, a portion of sample (200 mg is sufficient) may be supplied via flow line 14 to stage 16 wherein organic carbon determination is performed by a commercially known process. Additional ground sample is also supplied for a collateral process wherein paleotemperature determinations are made, that is, paleotemperature analysis stage 18. Data obtained for both organic carbon determination 16 and paleotemperature analysis 18 are applied for later data processing, as will be further described.

Returning to source rock analysis procedure, the samples are weighed within stage 12, ten grams per sample as required for the extraction procedure which is effected

in extraction and evaporation stage 20. The ten gram sample is extracted with a Soxhlet extractor using redistilled benzene and methanol for the solvent. The background value of the solvent should not exceed ten ppm of total extractables. After extraction, the samples are then subjected to repetitive evaporation procedures.

Extraction and evaporation stage 20 is carried out in accordance with standard and accepted laboratory practices. The weighed sample quantities are subjected to extraction using redistilled, reagent grade benzene and methanol solvent, a two:one benzene-methanol solution. The extraction procedure is highly exacting requiring precise ten gram plus or minus .0005 gram per sample quantities and, after extraction with the extracting solvent, initial data recordings are made as to observed abnormalities, e.g. visible salt, any sediment in extracting solvent or shale presence.

A next following evaporation procedure is utilized with tetrahydrofuran solvent (THF). Precisely 15 milliliters of freshly distilled (no peroxide inhibition) THF is pipetted into each extract sample whereupon the sample solutions are filtered through coarse sintered glass to reject salts and other THF insoluble materials. Precisely 10 milliliters of the THF solvent is used to complete the transfer and the combined filtrate is subjected to evaporation in vacuum until all liquid THF has been evaporated. Extremely pure solution product and continual recordation of gross and net weights of the tared flasks utilized is continually maintained through the process.

The extract samples from stage 20 are then applied to a thin layer chromatographic process in order to establish the characterization of the stabilized rock extracts and crude oils. Thin layer chromatographic procedure takes place through plate spotting stage 22, plate developing stage 24, plate charring stage 26 and finally the densitometer examination stage 28. The thin layer chromatography (TLC) plates are prepared by placing a uniform layer of silica gel on a glass plate which measures about eight inches by eight inches and has a thickness of 1.5 millimeters. The illustration of Fig. 2 shows a base portion of a glass plate 30 after tracking and spotting, as will be further described below.

The TLC plate is formed by spreading a slurry of silica gel G over the surface thereof. The silica gel G slurry is applied to uniform thickness with a gate setting of 250 microns using a Desaga Blinkmann "Slurry Applicator". The layered plates are allowed to dry for a period of several hours and then placed in a dust-free standby container. The TLC plates having been air-dried are then further dried and acti-

vated under heat, e.g. extended drying at 120°C with a filtered, room air sweep-breathing vacuum oven; and thereafter, the activated TLC plates are stored in a desiccated standby container.

The activated TLC plates are then pre-eluted several times by edge submersion in a mixture of benzene, diethyl ether and acetic acid (approximately 50:50:2) in order to remove most foreign absorbents. The TLC plates are disposed in vertical attitude with one edge of the plates submerged to about one-quarter inch in the solvent mixture and maintained in that attitude until the mixture has travelled, through capillary attraction, up the full expanse of the coated TLC plate. Next, the pre-eluted layers are tracked and edge-stripped to prepare the synthesized surface for the subsequent spotting operation. As needed, the pre-eluted TLC plates are placed upon a scribing jig whereupon the layer is scribed to produce 25 spaced tracks 32 (Fig. 2) each 5 millimeters wide. Also at this time, the plate is edge-stripped to provide a three-eighth inches border along both side edges running parallel to the tracks. For this application, it is best to pre-elute *with* the direction of intended development. Layer impurities are moved to the top portion of tracks where they do not contribute to the char background of chromatograms. The TLC plate 30 is then ready for sample spotting upon reactivation through short presence within purified, heated air.

The spotting process is a highly exacting procedure and the equipment utilized is illustrated in Figs. 3, 4 and 5. Fig. 3 illustrates a spotting block assembly 36 having a plate receiving surface 38 of proper dimension to receive a TLC plate 30, as positioned between opposite edge plates 40 and 42. Two pair of manually releaseable clamps 44, 45 and 46, 47 provide means for clamping a TLC plate 30 and indexing cover plate 48 (Fig. 4) in proper position through the spotting process. A trough, 49 having an upper surface 50 and hollowed out trough portion 52 is secured in juxtaposition to one end of the spotting block 36, and trough portion 52 serves to contain a sufficient amount of spotting solvent, a mixture of cyclohexane and benzene in the ratio of 9:1 by volume. The trough 49 is constructed for vertical movement to be clamped down during preparation, for release and spring tensioned upward position during operation in contact with the underside of coverplate 48. Cooling water may be circulated through cooling block connectors 53 to keep TLC plate 30 cooler than coverplate 48 thereby to prevent solvent vapor condensation under coverplate 48.

In the spotting process, the TLC plate 30 is placed within plate receiving surface 130

38 with the edge placed at the trough edge or line 54. Thereafter, a clean wick, shown by dash lines 56, of selected filter paper, is placed with one edge submerged within solvent trough 52 and one edge overlaid in contact with the edge of the TLC plate 30 which is undergoing spotting. The indexing cover plate 48 is then placed in superposition over TLC plate 30, wick 56 and trough 52, and securely clamped by means of manual clamps 44, 45, 46 and 47. Fig. 5 illustrates such position as indexing cover plate 48 is securely clamped over TLC plate 30. This then places a spotting slot 58 (Fig. 4) over the TLC plate 30 at a reference position. A first index line 60 is scribed on track index plate 48 at a position thirty-five (35) millimeters up from the center of spotting slot 58, while a second index line 62 is transversely scribed parallel to and one hundred fifteen (115) millimeters disposed from the first index line 60.

With TLC plate 30 and indexing cover plate 48 properly positioned and clamped as shown in Fig. 5, the plate is ready for spotting of samples. The source rock samples to be spotted, and this particular TLC plate makes provision for twelve such sample spottings, are arranged in order for spotting on tracks 3, 4, 6, 7, 11, 12, 14, 15, 19, 20, 22 and 23 (see Fig. 2). Similar order of sample listing is maintained throughout the procedures in order to insure that proper values are entered in the log record of the particular TLC plate as they relate to referenced samples. Further, in preparing the spotting solutions, it is required to calculate and record for each sample the volume solvent required for the preparation of spotting solutions containing 12.5 micrograms of solvent per one microliter.

The spotting solution for each sample is prepared by adding the required volume of spotting solvent, tetrahydrofuran (THF), with further addition of a one microliter disposable capillary into each spotting solution. Additionally, fresh spotting solutions are prepared for standard reference samples which standard samples will be spotted on tracks 1, 9, 17 and 25 (see Fig. 2) to provide a relative reference for densitometer readings obtained subsequently in the operation. The processing authority maintains various samples of crude oil and the like for use as standard reference samples, and such standard samples may be any of the various, selected samples of known analysis which will provide a relative reference point for later calculation and evaluation. The tracks 2, 5, 8, 10, 13, 16, 18, 21 and 24 are maintained blank in order to provide further background reference relative to the densitometer readings and evaluation.

Thus, each of the samples is rapidly spot-

ted in order through the spotting slot 58 of indexing cover plate 38. This spotting is effected when the solvent front (from trough 52 and wick 56) reaches the first index line 60, and at that time the samples are spotted rapidly from left to right in proper order. The spotting slot 58 is maintained covered with a 185 mm square, 1.5 mm thick track indexing glass plate during the solvent movement to provide most effective capillary action or movement of the solvent front upward along the multiple tracks 32 of TLC plate 30. The developing solvent is allowed to move therealong until a first portion of the solvent front reaches the second index line 62, whereupon the spotting assembly is disassembled and the developed TLC plate 30 is removed.

After development, the TLC plate is transferred to a 120°C vacuum oven for drying with filtered air flow in order to remove the developing solvent and any surface moisture which may have been absorbed from room air during preceding operations.

As shown in Fig. 1, a stage of TLC plate charring 26 is then effected in order to bring out the visual characteristics of the solvent and sample effects to the thin layer tracks. In this process, the developed TLC plates are enclosed in a heated atmosphere of dry sulphur trioxide and allowed to char for about twenty minutes. This is done by placing the developed TLC plate on a smooth, uniformly heated metal charblock at a temperature of about 150 degrees centigrade, and covering the TLC plate with a heat-resistant glass charlid wherein its sand blasted concave surface is coated with 30-33 percent pyrosulphuric acid. After removal of the TLC plate 30 from the charblock, the plate is ready for overnight storage in a light-tight container and the subsequent densitometer reading process in densitometer 28, (Fig. 1).

Fig. 6 illustrates a representative form of thin layer track 32 wherein a measured amount of spotting solution has been placed at spot point 64 in contact with the developing solvent wet track. During the developing process, different adsorbed sample materials progressed upward along track 32 to leave distinct char-color demarcations. Such demarcations occur on each track as a wick mark 66, polar compounds 68, aromatic hydrocarbons 70, saturated hydrocarbons 72, and solvent front layer impurities 74. The TLC plates are then transversely scribed with four scribe lines, 76, 78, 80 and 82 in order to mark the division between the adsorbent substances 66 through 74. After scribing, the TLC plate is then ready for application to the densitometer reading stage 28, refer again to Fig. 1.

Output from densitometer stage 28 is 130

applied through digital readout circuitry 84 whereupon the data is output to data recording system 86. The TLC data from data recording system 86 is applied to a standard form of computer 88, as is also the ancillary data derived from paleotemperature analysis stage 18 and organic carbon determination stage 16, both processes having been commercially utilized prior to this time to derive specific information. The computer 88 is utilized to process the TLC data in conjunction with other input data in accordance with a specialized TLC calculation program 90, to be further described below, and the computer output in the form of interpretative data relative to the initial samples input is provided with various parameters analyzed.

The output information from data interpretation stage 92 will serve to provide the initial supplier of samples with data as to the likelihood of finding petroleum products in the area from which the supplier had derived the samples. Such samples may be derived in various patterns. The plurality of supplied samples might have been taken from successive strata points downward along a single bore hole, or the samples might be taken from one or more strata as specified for a plurality of bore holes which are located in related disposition at some situs of earth terrain.

As shown in Fig. 7, the developed and charred TLC plate 30 is applied to a scanning densitometer 94 which serves to continuously scan each successive track 32 of TLC plate 30 to derive density versus track position readings. The scanning densitometer 94 may be a commercially available transmission type such as the Automatic TLC Densitometer as manufactured by Photovolt Corporation of New York, New York.

Instantaneous densitometer output is measured in terms of voltage via a digital voltmeter 96, and data is supplied to a strip chart recorder 98 as well as to an analytical data acquisition system 100. Strip chart recorder 98 is a variable response recorder also supplied through Photovolt Corporation, and which provides a chart such as that shown in Fig. 8. The digital voltmeter 96 is an integrating digital voltmeter as supplied by Hewlett-Packard and digital read out is provided through such as the Perkin-Elmer Read Out Model No. 2.

Referring to Fig. 8, the output strip chart provides a positive and negative calibrated amplitude indication relative to distance along the thin layer track. A strip chart line 104 is shown as indicating the variable density read out and negative indications 106 and 108 would be caused by scribe lines 80 and 78 of Fig. 6. Thus, the variable area 110 would be caused by paraffins, the

variable area 112 by aromatics, and the variable area 114 by the asphaltenes or resins, as the constituents were adsorbed along the TLC track.

As shown in Fig. 1, instantaneous digital readings of the densitometer record or chart line 104 would be applied through digital read out to the computer 88 as controlled by the TLC calculation program 90. The digital read out provides an integrated read out, and the voltages are then pre-normalized in order to give a percentage output indication for each adsorbent component, i.e. those major components of interest in the analysis.

All TLC data is then made available to the computer 88 under control of the TLC calculation program in order to derive maximum interpretive data from the densitometer readings as well as other data compiled for the same sample substances through ancillary measures. The TLC program is designed to calculate normalized areas from data acquired from densitometric scanning of the TLC plates, such areas as delineated by the scribe lines 76, 78, 80 and 82 (Fig. 6). The TLC program Figs. 9 through 17, operates on the data obtained from the analytical data acquisition system 100, i.e. the digitized output from densitometer 94 through digital volt meter 96. The TLC program scans each track 32 successively to integrate data as it is read therefrom until a scribe mark is encountered. At the point of the scribe mark, the area is stored and integration begins on the next successive area or until occurrence of the next scribe mark. Scribed delineation is made on a voltage basis as the output signal will increase to a predefined constant value. Interpolated area across a scribe mark is evenly divided as between the two adjacent areas. When the end of track is reached, integrated areas for that particular track are stored and integration is started on the next successive track. This continues until all tracks are scanned in succession and the data run is concluded.

After conclusion of a data run, a plate format card is read through the computer, which card lists the position by track number of all blank runs on the plate. A background correction is then made by subtracting from each sample area the corresponding area of the nearest blank, i.e. data read from the blank or "B" tracks of the plate as shown in Fig. 2. After correction is made, the sample areas are normalized by track, ignoring the first area (solvent front 74) and the last area (wick mark 66), and percentage areas are calculated for the remaining regions therebetween. The program may be made to function with any number of areas; however, five regions are usually delineated, as shown in Fig. 6, so that the three center areas can be isolated

and used in the calculation relative to ascertaining the percentages of paraffins, aromatics and asphaltenes present within the particular samples.

- 5 Throughout operation of the TLC program, the TLC tracks are continually identified, in terms of the samples supplied, by cards which are input to the program. These cards contain a sample number, plate number, and a track number. The program then matches up the plate and track number on the card with its stored information, and prints out the final data, area percent values and corrected areas, with the correct sample number attached. In addition to print out of final data, which would be made available in the service to the sample supplier, the program can also output the same information on punch cards; whereupon, the entire assemblage would be available for input to the computer under control of another specific program.

- The total processing program includes three specific subroutines. Thus, a subroutine INTEG is called when a complete track has been recorded. This subroutine integrates the area under the curve between scribe points along a track. When a scribe point is recognized, integration is stopped, the area evaluation is stored, and then the data is corrected for the area of the scratch. A second subroutine TLCERR is called whenever a new plate number is recognized before the 25 tracks have been found. This routine outputs all area information which has been stored for the present TLC plate. It then ignores the input cards for that plate and resets the necessary parameters so that the new plate can be calculated. Yet another subroutine entitled TIMEIT serves to set time parameters for use in a subsequent accounting routine as might be necessitated by any of various additional processing requirements.

- 45 The TLC program as presently utilized is written in S/360Fortran H execution on an IBM Model 360/65 computer with full operating system. The complete TLC program including subroutines is illustrated in flow diagram form in Figs. 9 through 17. The operating dictionary for the TLC program is as follows:

- 55 **AREA**
An array which contains the area values for the present track.
- BASE**
A baseline voltage which is set when program is written.
- 60 **ICUT**
A calculated variable which determines a frequency filter cut-off for eliminating water spots, etc., bands.
- IFLAG**

A flag which is set when the end of a track is recognized.

- INTEG**
A subroutine which integrates the delineated areas. 70
- IRATE**
Data rate in readings/sec.
- ITEST**
A temporary variable which indicates the amplitude difference between two adjacent data points. 75
- NAREA**
An array which contains any notations which are appended to area answers on output.
- NBLK**
The number of blanks on a plate. 80
- NBLANK**
The name of a variable containing two blank characters.
- NØP**
Variable on sample input card which indicates plate number. 85
- NØT**
Variable on sample input card which indicates track number. 90
- NPERR**
An array which contains any notations which are appended to percent answers on output.
- NPLAT**
Plate number of the present plate. 95
- NPNT**
An experimental point counter.
- NTRACK**
Track number of present track. 100
- PAREA**
Array where corrected areas stored by track and delineated area for one entire plate.
- STAR**
Variable containing two asterisks (**). 105
- STPM**
The stepname of this program (TLC—GC) for accounting purposes.
- TIMEIT**
A subroutine which sets up timing parameters for accounting purposes. 110
- TITLE**
An array which contains the customer sample number from the sample input card. 115
- TLCERR**
A subroutine which outputs area information when a missing track is discovered.
- Y**
An array which contains the raw data as it is read in. 120
- The flow diagram of Figs. 9 through 17 constitute the process matter as printed out by the IBM Model 360/65 computer, and such information in combination with the above-listed dictionary is sufficient to allow the skilled artisan to utilize the programmed 125

data procedure in accordance with the teachings of the present invention. It should be understood, however, that the same data processing measures may be instituted utilizing other existing computational equipment as well as other similar forms of command language.

WHAT WE CLAIM IS:—

1. The process for determining the content of definitive hydrocarbon constituents from particular source rock samples using thin layer chromatography comprising, grinding and extracting source rock samples to provide sample material of finite granular consistency and compound content, including asphaltenes, aromatic hydrocarbons and paraffins, and dissolving the sample material in a solvent for addition to a thin layer track of solvent-saturated silica gel to proceed therealong by capillary attraction for a pre-set distance, whereupon the thin layer track is dried and charred in the presence of sulphur trioxide to provide a variable density track record, and thereafter scanning said track record at a constant rate along the record length with an optical densitometer to derive a time-analog representation of the polar compounds, aromatic hydrocarbons and saturated hydrocarbons contained in said source rock sample material.

2. The process for determining the content of source rock samples as set forth in claim 1, in which a plurality of related source rock samples are prepared, dissolved and each applied in addition to a separate one of a plurality of thin layer tracks, each track to be similarly scanned by an optical densitometer to provide a related family of said time-analog representations.

3. The process for determining the content of source rock samples as set forth in claim 1 or 2, in which the time-analog representations are integrated over specific areas to derive percentage composition of the definitive hydrocarbon constituents for each source rock sample.

4. The process for determining the content of source rock samples as set forth in the previous claims, in which the solvent added for dissolution of sample material, is tetrahydrofuran.

5. The process for determining the content of source rock samples as set forth in previous claims, in which the solvent applied for saturation of the silica gel track is an adsorbed mixture of nine parts cyclohexane and one part benzene.

6. The process for determining the content of source rock samples as set forth in the previous claims, wherein an electronic

data processing machine under control of a specific program processes time-analog data output from the densitometer to integrate the data indications for each thin layer track.

7. The process for determining the content of source rock samples as set forth in previous claim 6, wherein the integrated output data for each thin layer track, each related to a specific source rock sample, is processed in relation to ancillary data derived as to paleotemperature analysis and organic carbon determination for each source rock sample.

8. The process for determining the content of source rock samples as set forth in claim 2, in which a standard sample material having known characteristics and time-analog representations is dissolved and applied for adsorption on a parallel thin layer track, such that optical densitometer scanning provides relative data for comparison to the time-analog representation derived for each of the sample materials.

9. The process of determining the content of source rock samples as set forth in claims 2 and 8, in which yet another thin layer track is left void of either sample or standard sample adsorbent materials, such that optical densitometer scanning of said yet another track provides background data for comparison to the time-analog representation derived for each of the sample materials.

10. The process of determining the content of source rock samples as set forth in claims 2, 8 and 9, wherein an electronic data processing machine under control of a specific program processes time-analog data output from the densitometer to integrate the data indications for each thin layer track.

11. The process of determining the content of source rock samples as set forth in claims 2, 8, 9 and 10, wherein the integrated output data for each thin layer track, each related to a specific source rock sample, is processed in relation to ancillary data derived as to paleotemperature analysis and organic carbon determination for each source rock sample.

12. The process for determining the content of source rock samples substantially as described with reference to and as shown in the accompanying drawings.

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data procedure in accordance with the teachings of the present invention. It should be understood, however, that the same data processing measures may be instituted utilizing other existing computational equipment as well as other similar forms of command language.

WHAT WE CLAIM IS:—

1. The process for determining the content of definitive hydrocarbon constituents from particular source rock samples using thin layer chromatography comprising, grinding and extracting source rock samples to provide sample material of finite granular consistency and compound content, including asphaltenes, aromatic hydrocarbons and paraffins, and dissolving the sample material in a solvent for addition to a thin layer track of solvent-saturated silica gel to proceed therealong by capillary attraction for a pre-set distance, whereupon the thin layer track is dried and charred in the presence of sulphur trioxide to provide a variable density track record, and thereafter scanning said track record at a constant rate along the record length with an optical densitometer to derive a time-analog representation of the polar compounds, aromatic hydrocarbons and saturated hydrocarbons contained in said source rock sample material.

2. The process for determining the content of source rock samples as set forth in claim 1, in which a plurality of related source rock samples are prepared, dissolved and each applied in addition to a separate one of a plurality of thin layer tracks, each track to be similarly scanned by an optical densitometer to provide a related family of said time-analog representations.

3. The process for determining the content of source rock samples as set forth in claim 1 or 2, in which the time-analog representations are integrated over specific areas to derive percentage composition of the definitive hydrocarbon constituents for each source rock sample.

4. The process for determining the content of source rock samples as set forth in the previous claims, in which the solvent added for dissolution of sample material, is tetrahydrofuran.

5. The process for determining the content of source rock samples as set forth in previous claims, in which the solvent applied for saturation of the silica gel track is an adsorbed mixture of nine parts cyclohexane and one part benzene.

6. The process for determining the content of source rock samples as set forth in the previous claims, wherein an electronic

data processing machine under control of a specific program processes time-analog data output from the densitometer to integrate the data indications for each thin layer track.

7. The process for determining the content of source rock samples as set forth in previous claim 6, wherein the integrated output data for each thin layer track, each related to a specific source rock sample, is processed in relation to ancillary data derived as to paleotemperature analysis and organic carbon determination for each source rock sample.

8. The process for determining the content of source rock samples as set forth in claim 2, in which a standard sample material having known characteristics and time-analog representations is dissolved and applied for adsorption on a parallel thin layer track, such that optical densitometer scanning provides relative data for comparison to the time-analog representation derived for each of the sample materials.

9. The process of determining the content of source rock samples as set forth in claims 2 and 8, in which yet another thin layer track is left void of either sample or standard sample adsorbent materials, such that optical densitometer scanning of said yet another track provides background data for comparison to the time-analog representation derived for each of the sample materials.

10. The process of determining the content of source rock samples as set forth in claims 2, 8 and 9, wherein an electronic data processing machine under control of a specific program processes time-analog data output from the densitometer to integrate the data indications for each thin layer track.

11. The process of determining the content of source rock samples as set forth in claims 2, 8, 9 and 10, wherein the integrated output data for each thin layer track, each related to a specific source rock sample, is processed in relation to ancillary data derived as to paleotemperature analysis and organic carbon determination for each source rock sample.

12. The process for determining the content of source rock samples substantially as described with reference to and as shown in the accompanying drawings.

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