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METHOD AND APPARATUS FOR PHYSICO-CHEMICAL ANALYSIS

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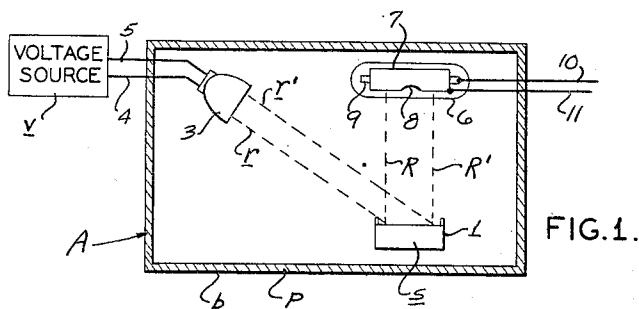


FIG. 1.

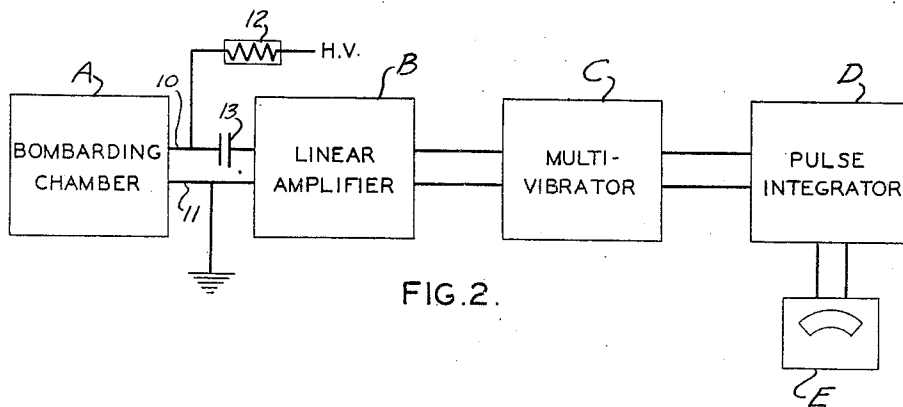


FIG. 2.

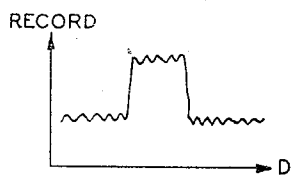


FIG. 3a.

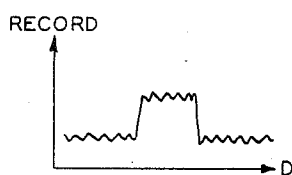


FIG. 3d.

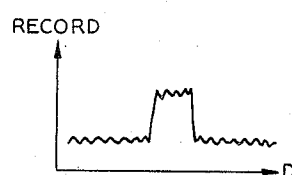


FIG. 3f.

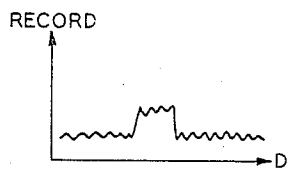


FIG. 3b.

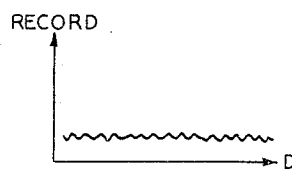


FIG. 3e.

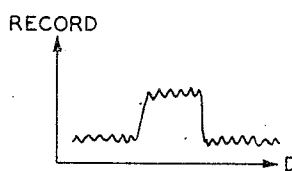


FIG. 3g.

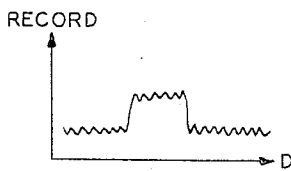


FIG. 3c.

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METHOD AND APPARATUS FOR PHYSICO-CHEMICAL ANALYSIS

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This invention relates in general to certain new and useful improvements in methods and apparatus for physico-chemical analysis and, more particularly, to methods and apparatus for the detection of radio-active and hydrogenous materials in soil samples and the like.

It has been experimentally shown that the radio active content of a surface soil sample as well as its hydrocarbon content may be correlated with subterranean petroleum deposits. Soil and similar substances containing minute quantities of radio active and hydrogenous materials cannot, however, be accurately analyzed by ordinary schemes of analysis in the absence of time-consuming attenuated procedures, and, furthermore, entirely separate analytic procedures and apparatus must be employed for the determination of radio-active materials on the one hand and hydrogenous materials on the other hand. Thus, when prospecting for petroleum and the like by analyzing the radio-active and hydrogenous content of a surface soil sample, it is more convenient and economical to use a single apparatus for performing both of the requisite analytical determinations.

It is, therefore, a principal object of the present invention to provide a simple, rapid, and definitive method for the analytical determination of radio active and hydrogenous materials, and apparatus for carrying out the same.

It is also an object of the present invention to provide compact and economical apparatus for analytically determining the radio active content of a sample.

It is a further object of the present invention to provide compact and economical apparatus for analytically determining the content of hydrogenous material in a given sample.

In the drawing:

Figure 1 is a vertical sectional view of a bombarding chamber for detecting the radio active content of a sample and for exposing said sample to radiations of selected wave length;

Figure 2 is a diagrammatic representation of a preferred form of apparatus for the analytical determination of radio active and hydrogenous materials in accordance with the methods of the present invention.

Figures 3^a, 3^b, 3^c, 3^d, 3^e, 3^f, and 3^g are graphs or charts showing examples of the analytical results obtainable with the methods and apparatus of this invention.

Referring now in more detail and by reference characters to the drawings, A designates a bombardment chamber comprising a preferably light-

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proof rectilinear box *b* having walls *p* formed of a relatively thick plywood or other similar material. Suitably disposed within box *b* is an open-topped sample container *1* for holding a sample *s*. Suitably mounted within the box *b* is an ultra-violet light source *3* having leads *4* and *5* connected to voltage source *v*. Also disposed within box *b* to receive fluorescence rays *R* and *R'* given off by the sample *s* upon bombardment by ultra-violet light rays *r* and *r'*, is a detector tube *6* comprising a self-extinguishing tube of the Geiger-Müller type, consisting of a cylindrical element *7* having a small aperture *8* centered midway along its length, and a wire *9* extending co-axially through element *7*. Lead *10* extends through the walls of box *b* and connects to wire *9*. Lead *11* extends through the walls of box *b* and connects to element *7*. The detector tube *6* is constructed of ultra-violet transparent glass, that is to say, a glass which passes or transmits a relatively large quantity of ultra-violet as well as visible light. Element *7* is coated on its inner surface with a material which freely gives up electrons when bombarded by radiations of the correct wave length.

The lead *11* issuing from the chamber *A* is connected to one input lead of a high-gain linear amplifier *B* and to the ground. The lead *10* is connected through resistor *12* to a source of high voltage and is also connected through the capacitor *13* to the other input lead of the amplifier *B*. This source of high voltage is maintained just above the threshold voltage of the detector tube *6* so that such tube *6* is in its most sensitive operating condition for the detection of radiant energy entering therein. The output leads of the linear amplifier *B* are connected to the input leads of a multi-vibrator *C*, the output leads of which are connected to the input leads of a pulse integrator *D*. The output leads of pulse integrator *D* are connected to the input leads of a suitable type of direct reading instantaneous recorder *E*, all as best seen in Figure 2.

In performing an analysis, a sample of soil or other material containing radio active or hydrogenous material, or both, is carefully milled or ground to a substantially uniform state of fineness and dried under a carefully controlled moderate temperature so as to remove all traces of moisture. It is essential that the sample be thoroughly dried or that all the samples in a single survey be dried to the same degree to insure uniform test conditions. Because of the danger of contaminating the sample with water vapor picked up from the atmosphere, it is preferable

that sample *s* be packed in sample containers prior to drying. When dry, sample container 1 together with sample *s* is weighed and then placed within box A.

The ultra-violet light issuing from light 3 in rays *r*, *r'* bombards sample *s*. If sample *s* contains hydrogenous material, such material will fluoresce when so bombarded and rays R, R' will issue toward the detector tube 6 and enter cylinder 7 through aperture 8. Since the inner surface of cylinder 7 is coated with a photo-sensitive substance, electrons are freely given up and attracted by wire 9. The electrons will create impulses which will pass through linear amplifier B, multi-vibrator C, pulse integrator D, and give a visible record on recorder E.

The detector tube 6 actually counts the photo-emitted electrons, and thus, it is obvious that much smaller values of fluorescence can be measured with detector tube 6 because it is more sensitive than an ordinary phototube. Since ultra-violet transparent glass is used in the manufacture of detector tube 6, it is capable of detecting fluorescence in the visible range or near ultra-violet region, as well as the full range of ultra-violet wave lengths.

Furthermore, the detector tube 6 is uniquely capable of detecting radio activity as well as fluorescence. Thus, it can be seen that when light 3 is bombarding sample *s*, and sample *s* contains radio active as well as hydrogenous material, the record on recorder E will be a composite one of both radio activity and fluorescence. Figure 3^a shows a profile taken over an area which contains radio active as well as hydrogenous substances. This composite curve may be resolved into two components, one the contribution of radio active material, and the other the contribution of hydrogenous material, by disconnecting source *v* from light 3 and noting the record on recorder E. The record shown will be that of radio activity only, since no fluorescence will be evident in the absence of ultra-violet light. Figure 3^b shows this profile. If the curve in Figure 3^b is subtracted from the curve in Figure 3^a, the curve in Figure 3^c is the result. This curve is the result of fluorescence only.

Figures 3^d and 3^f show profiles made under the similar conditions to those of Figure 3^a, that is to say, placing different samples in the sample container and bombarding it with rays *r*, *r'*, employing the same hookup employed in the example above discussed. For purposes of discussion, let it be assumed that the curves shown in Figures 3^d and 3^f, respectively, are the type of curves obtained from two different samples and that correspondingly the curves of Figure 3^e and 3^g, respectively, are obtained when the source *v* is disconnected. Scrutinizing Figures 3^d and 3^e, which are companion curves, it becomes evident that the anomaly shown in the curve of Figure 3^d is due to fluorescence only, since no anomaly occurs in the curve of Figure 3^e. Similarly, it is evident that the anomaly of Figure 3^f is due to radio activity only, since the curve of Figure 3^g is identical to it in every respect.

In order to relate the results to actual figures and percentages, the apparatus must, of course, be calibrated against known samples. A description of the technique of prospecting for oil by examination of surface soil samples is contained in my co-pending application Serial No. 501,499, filed September 7, 1943, now Patent No. 2,462,270.

While I have described my invention in certain specific embodiments, I do not desire to be limited thereto, as for example, the material examined does not have to be a surface soil sample, but may be well cuttings or cores or any material suspected to contain radio active and hydrogenous materials.

It should be understood that changes in the methods, compositions and combinations above set forth may be made without departing from the nature and principle of my invention.

Having thus described my invention, what I claim and desire to secure by Letters Patent is:

1. A method for use in petroleum exploration, which method comprises thoroughly drying a soil sample and reducing it to a substantially uniform state of subdivision, irradiating the sample directly with ultra-violet light, measuring the intensity of the total radiations given off by the sample, thereupon stopping ultra-violet radiation, and measuring the inherent radiations coming from the sample to ascertain the radio activity of the sample.

2. A method for use in petroleum exploration, which method comprises drying a soil sample, directly irradiating the sample with ultra-violet light, measuring the intensity of the total radiations given off by the sample, thereupon stopping ultra-violet radiation, and measuring the inherent radiations coming from the sample to ascertain the radio activity of the sample.

3. Apparatus for analyzing soil samples incident to petroleum exploration to determine the content of radio active and hydrogenous materials in the samples, said apparatus comprising a bombardment chamber having an ultra-violet light source, means in the chamber below and to one side of the light source for exposing a surface of the sample to rays from said source, and an electronic tube positioned outwardly from the sample along a line normal to the sample-surface for sensing the total radiations given off by the sample when irradiated by ultra-violet light.

4. Apparatus for analyzing soil samples incident to petroleum exploration to determine the content of radio active and hydrogenous materials in the samples, said apparatus comprising a bombardment chamber having an ultra-violet light source, means in the chamber below and to one side of the light source for exposing a surface of the sample to rays from said source, and an electronic tube of the Geiger-Müller type positioned along a line normal to said sample-surface for sensing the radiations given off by the sample when irradiated by ultra-violet light.

5. Apparatus for analyzing soil samples incident to petroleum exploration to determine the content of radio active and hydrogenous materials in the samples, said apparatus comprising a bombardment chamber having an ultra-violet light source, means in the chamber below and to one side of the light source for exposing a surface of the sample to rays from said source, an electronic tube positioned along a line normal to said sample-surface for sensing the combined effect of the radiations given off by the sample and the radiations produced by irradiation of the sample with ultra-violet light and an electronic circuit for measuring the radiations sensed by the tube.

6. Apparatus for analyzing samples to determine the content of radio active and hydrogenous materials in the samples, said appara-

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tus comprising a bombardment chamber having an ultra-violet light source, means in the chamber below and to one side of the light source for exposing the sample to rays from said source, and an electronic tube of the Geiger-Müller type positioned above the said means for sensing the combined effect of the radiations given off by the sample and the radiations produced by irradiation of the sample with ultra-violet light in combination with a linear amplifier, a multi-vibrator, a pulse integrator, and an instantaneous recorder.

7. Apparatus for analyzing samples to determine the content of radio active and hydrogenous materials in the samples, said apparatus comprising a bombardment chamber having an ultra-violet light source, means for exposing the sample to rays from said source, and an electronic tube having a central rod-like electrode surrounded by a tubular photo-sensitive electrode having an aperture presented toward said means, said tube being positioned for sensing the combined effect of the radiations given off by the sample and the radiations produced by the irradiation of the sample with ultra-violet light.

8. Apparatus for analyzing samples to determine the content of radio active and hydrogenous materials in the samples, said apparatus comprising a bombardment chamber having an ultra-violet light source, means for exposing the sample to rays from said source, and an electronic tube having a central rod-like electrode surrounded by a tubular photo-sensitive electrode having an aperture through which radiations may reach the rod-like electrode positioned for sensing the combined effect of the radiations given off by the sample and the radiations produced by the irradiation of the sample with ultra-violet light.

9. A method for use in petroleum exploration, which method comprises thoroughly drying a soil sample and reducing it to a substantially uniform state of subdivision, irradiating the sample directly with ultra-violet light while main-

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taining the sample out of contact with moisture and moisture-containing atmosphere, measuring the intensity of the total radiations given off by the sample, thereupon stopping ultra-violet radiation, and measuring the inherent radiations coming from the sample to ascertain the radio activity of the sample.

10. A method for use in petroleum exploration, which method comprises thoroughly drying a soil sample and reducing it to a substantially uniform state of subdivision, placing the dried sample in a substantially moisture-proof chamber, irradiating the sample directly with ultra-violet light, measuring the intensity of the total radiations given off by the sample, thereupon stopping ultra-violet radiation, and measuring the inherent radiations coming from the sample to ascertain the radio activity of the sample.

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