

## **Determination of Organic Matter Maturity by Vitrinite Reflectance**

### **1. Introduction**

Vitrinite reflectance, established over 50 years ago, is a microscope technique used primarily in the oil and gas industry to determine the degree of thermal maturation of source rocks and potential source rocks (Stach et al., 1982). It is a laboratory analytical method performed with a microscope fitted with incident (vertical) white light illumination. Analyses are done on samples of highly polished organic matter (OM) within microscope preparations. These preparations can be comprised of either whole rock or of concentrated organic matter cast in epoxy.

The technique is widely used to determine the degree of thermal maturation (time-temperature index) based on reflectance of type III organic matter or vitrinite. The premise of the technique is to measure and record a number reflectance values, up to about 25, on individual pieces (macerals) of type III organic matter or vitrinite (humic matter) from a single sample preparation. The statistical mean value ( $R_m$ ) of these measurements is considered to be the degree of thermal maturation of that sample.

The vertical illumination microscope is also commonly used for the purpose of merely identifying additional types of organic macerals by using ultraviolet light instead of white light to take advantage of the fluorescence characteristics of these OM types otherwise less evident or completely undetectable in white light.

### **2. Interfaces with Other Methods**

Vitrinite reflectance can be a stand-alone analytical tool. Since its establishment it has been correlated fully or partially with additional thermal-maturity sensitive parameters derived from other analytical methods. Some of these parameters are from RockEval, (a programmed open-system pyrolysis technique), and include Tmax, hydrogen index, oxygen index, and production index (Espitalié et al., 1985). Other related parameters are from proximate and ultimate analyses collectively used to characterize and rank humic coals (Stach et al., 1982). Major coalification ranks include peat, lignite, subbituminous, bituminous, or anthracite (Stach et al., 1982, Table 4).

### 3. Materials and Equipment

Equipment required for sample prep:

- Mortar and pestle
- Plastic cap-plugs, 7/8 inch, for casting of plugs
- Low viscosity epoxy
- An engraver to inscribe identifying number on each
- Wet and dry grit paper: 60 grit and 600 grit
- Alpha -alumina polishing powder: 0.3 and 0.05 micron
- Low-knap polishing cloths
- Polishing machine capable of rotating from 10 to 300 rpm

Equipment for microscope work:

- Reflected light microscope with 500 X oil-immersion lens, green light (546 nm) filter, immersion oil, photometer to measure reflected light, controller to operate shutters and photometer
- Spread sheet program to record reflectance data

### 4. Procedure

Vitrinite reflectance is performed on type III organic matter, or vitrinite. This material is examined in polished preparations comprised of OM either from concentration from OM-bearing rocks, such as a shale or siltstone, by means of an acid treatment and heavy liquid separation technique, or within pieces of rock as “whole rock” preparations. Samples of coal can be considered as whole rock preparations; only significantly less material is required for examination, and no preparation is required other than crushing to a small particle size.

The microscope preparation is made by mixing, in a small sample cup, the concentrated OM or the crushed rock with a two-part, low viscosity epoxy until all the material is wetted. After this, the preparation is allowed to cure overnight. Next the preparations are removed from the cups one at a time and the proper identification is engraved with a vibro engraver. Then they are taken to a grinding/polishing machine, where the preparations are first ground with two sandpaper steps and then polished with two polishing steps. The grinding process requires two sizes of grit paper. A 60 grit paper is used for the first or coarse grinding, followed by the same procedure with a 600 grit paper to remove the coarse grinding marks and to make a smooth surface.

The grinding is done on a heavy duty machine which features a variable speed platen. Both grinding steps are done at a platen speed of 300 RPM, and a stream of water is added to cool the pellet and to carry away the debris. These steps require only that a minimum amount of material be removed to expose the sample material within the epoxy. This is usually about 30 seconds for the first step and about 15 seconds for the second step. Due to the high level of hardness and abrasiveness of some rock samples, the 600 grit paper requires frequent changing, sometimes lasting for only one sample.

The sample preparation is held against the grit paper, taking care to keep the sample flat or parallel to the surface, and pressure is applied; up to about 2-3 lbs (1 kg). The coarse paper removes material quickly so 3 to 5 second intervals are recommended between inspections to follow the process. Only enough material is removed to reveal an edge-to-edge surface of the enclosed material.

The polishing is done with the same equipment as the grinding, though it is performed at a platen speed of 40 RPM, and without running water. A nap-free cloth is used with these steps, and this cloth is first made wet and then a small amount, about 2-3 cc, of polishing solution is squirted onto the platen. For each polishing step, the samples are held onto the platen for about 10 seconds. The prepared pellet is held, by hand, flat against the platen and rotated against the rotation direction of the platen. After rinsing in a beaker of water, the sample is blown dry with a stream of air and examined by eye for scratching or other abnormalities. If the sample looks good, and the surface is uniform, then the procedure is repeated until the sample suite is completed.

The microscope used for reflectance is a Zeiss<sup>1</sup> with a programmable controller box. A PC with a spreadsheet program, such as Microsoft Excel™, is opened to directly type reflectance data into at the time of a reflectance value acquisition.

The polished preparation of rock chips or concentrated organic matter is first gently pressed onto a glass petrographic slide with modeling clay, and then placed onto a leveling press to make the preparation level. The slide is then placed on the microscope stage and a drop of immersion oil is put onto the polished sample. The sample is brought into focus and the sample is moved in a stepping fashion across the width of the prep. During the scanning portion all the organic material is observed and particles of type III OM are identified, if present. When a quality piece of OM is seen, and deemed appropriate for measuring, a measurement is then made. This involves positioning the photomultiplier sensing spot on a precise, polished location of the OM particle and then pushing the key on the controller box which represents the data acquisition channel. At the time of this action, the reflectance is shown in the LCD readout window of the controller box; this number is then typed into the spreadsheet opened for the current sample suite.

---

<sup>1</sup> Any use of trade names is for descriptive purposes only and does not imply endorsement by the U.S. Government.

This action is repeated until at least 25 reflectance measurements are recorded or the end of the sample pellet is reached. Samples with a lean OM content often will have fewer than the suggested number above. After completing the scan of the preparation, the average reflectance value is calculated.

When the samples from a particular job are completed the data are transferred to the database for storage.

## **5. Calibration and Quality Control Samples**

At the beginning of the microscope work, the microscope is calibrated several times throughout the day. If the microscope light is adjusted in any way, the calibration is redone.

Calibration is performed by programming the MSP-25 controller with a short series of commands, which ends with entering the value for a reflectance standard and running the programming step with the standard in place. The standard should be altered to approximate the expected level of reflectance (there are several standards of increasing reflectance available) depending on the maturation of the organic material being examined. Keeping the standard closer to the reflectance of the measured material assures a more accurate response from the photomultiplier.

## **6. Limits, Precautions and Interferences**

Limits, precautions, and interferences are not relevant to the reflected light analytical technique if only organic matter, prepared in an appropriate manner, is to be examined and measured. Almost any material can be observed with the scope.

## **7. Acceptance of Data**

There is no consensus regarding the number of data points per sample that is acceptable. It is desirable to attain perhaps, twenty five reflectance values for any sample, but due to the high variability, i.e., organic matter content, of any two samples, this is rarely possible. One exception, though, is with a coal sample, relating to the abundance of vitrinite in coal. In the absence of OM particles to measure, there is the compromise to attain as many readings as possible. This minimum number can be 2 or 20. Though two measurements would be insufficient for statistical purposes, it does retain relevance if the data points are obtained rigorously. Instead of measuring all identifiable OM and allowing for a statistical sorting, the organic matter is selected by the microscopist with high confidence. Only that material deemed to be the indigenous OM population is

measured; all other OM is noted, but not measured. The OM not measured is addressed in accompanying notes, but does not enter into the computation of a mean value for the sample.

The matter of calibration drift is addressed by checking the equipment response several times during the day. The calibration standard is put into place on the microscope and a reading is performed. A difference of 0.02 percent reflectance is not considered significant. Any more than that number and adjustments are made to bring the calibration back to compliance.

## **8. Data Handling and Transfer**

At the start of the examination of a sample, information such as the job number and the sample submitter field number are entered into the spreadsheet. Thereafter, an identifying value or name or number representing each sample is entered into the spreadsheet as column headings and individual reflectance readings are entered, manually, next to these until the sample is completed. Statistical values, such as mean and standard deviation, are calculated and added for each sample at the bottom of these data columns.

At the completion of a suite of samples, the spreadsheet is copied and electronically sent to the LIMS coordinator, and then entered into the database.

## **9. References**

Stach, E., Mackowsky, W., Teichmuller, M., Taylor, G.H., Chandra, D., and Teichmuller, E., 1982, Stach's textbook of coal petrology: Gebruder Borntraeger, Berlin, 535 pp.

Espitalie, J., Deroo, G., and Marquis, F., 1985, La pyrolyse Rock-Eval et ses applications: Revue de L'Institut Français du Pétrole, v. 40, No. 6, pp. 755-784.

## **10. Attachments**

None

## **11. History of Changes**

R0: Initial Issue