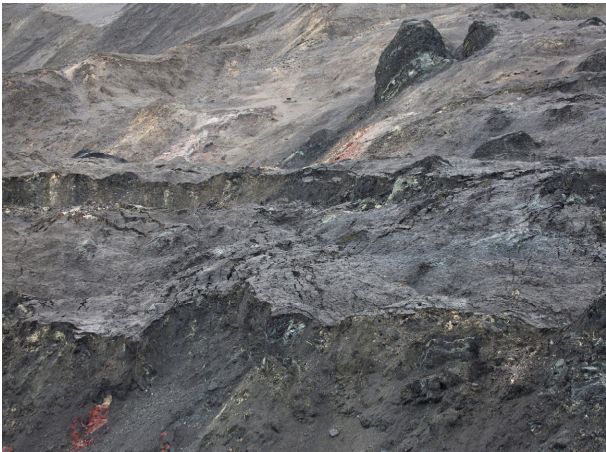


Application News

No. 071

Total Organic Carbon Analysis

Carbon Measurement of Oil Shale



Oil shale, also called kerogen shale, is a type of sedimentary rock that has a high content of hydrocarbons in the stage prior to petroleum. The gas obtained by heating oil shale is called oil shale gas, while the natural gas enclosed in fine cracks in sedimentary rocks is called shale gas. In some cases, oil shale is used for power generation and heating by direct combustion. As high expectations are placed on oil shale as an energy source, the carbon content of oil shale is an important factor.

The system using Shimadzu TOC-L total organic carbon analyzer and SSM-5000A solid sample combustion unit measures the carbon content of solid samples by direct combustion at high temperature. This system does not require extraction or other troublesome pretreatment, and enables measurement of the total carbon content with a high recovery rate and accuracy. This article introduces an example of measurement of the total carbon content of oil shale by using the above-mentioned system.

Y. Ikezawa

■ Sample Preparation

Because natural gas has accumulated in the crevices in the sedimentary rock called oil shale, there is a possibility of explosion due to rapid heating if oil shale is measured in lump form. Therefore, part of the sample material (Fig. 1) was broken up and crushed using an agate mortar (Fig. 2), and the powdery oil shale was placed in sample boat for use as the measurement sample.



Fig. 1 Oil Shale

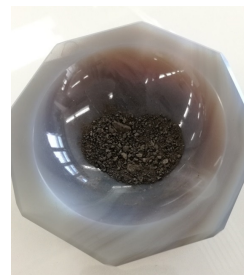


Fig. 2 Crushing Treatment



Fig. 3 Measurement Sample



Direct Combustion Method Using TOC Solid Sample Measurement System

- ✓ Does not require extraction or other pretreatment, and has high recovery rate and accuracy.
- ✓ Does not require chemicals.
- ✓ Quick measurement time of approximately 5 min.
- ✓ Sample boat is reusable.

■ TOC Solid Sample Measurement System

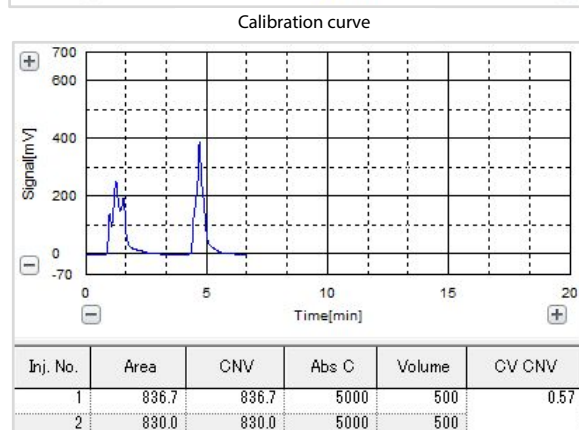
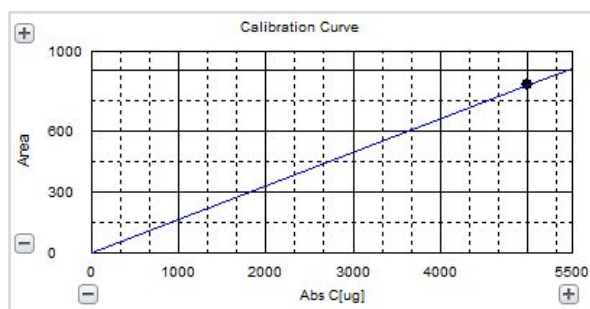
The TOC solid sample measurement system, comprising the TOC-L total organic carbon analyzer and SSM-5000A solid sample combustion unit, quantifies the organic content of solid samples by combustion oxidation of the organic matter in the sample and detection of the formed carbon dioxide (CO₂). Because the organic matter contained in a sample can be measured by direct combustion of the sample, quick and simple organic analysis without extraction or other troublesome pretreatment processes is possible. Table 1 shows the measurement conditions.

Table 1 Measurement Conditions

Analyzer	: TOC solid sample measurement system TOC-L _{CPH} total organic carbon analyzer + SSM-5000A solid sample combustion unit (Cell switching valve set: Specification A)
Cell length	: Short cell
SSM carrier gas	: Oxygen gas, 400 mL/min
TC oxidation method	: Combustion catalytic oxidation (Combustion temperature: 900 °C)
Measurement item	: TC (Total carbon)
Calibration curve	: One-point calibration curve using 1 % C glucose solution

■ Preparation of Calibration Curve

The calibration curve was prepared by adding 500 µL of a 1 % C (carbon concentration: 1 %) glucose solution to ceramic fiber in the sample boat, and performing the TC measurement (Fig. 4).



Measurement of calibration curve with 500 µL of 1 % C glucose solution

Fig. 4 Measurement Data of Calibration Curve

■ Results of Sample Measurement

Approximately 130 mg of the powdered oil shale was weighed out into the sample boat for each measurement, and three measurements were performed. Table 2 shows the measurement results, and Fig.5 shows the peak data obtained by the measurements. From the measurement results, the oil shale prepared for this study contained approximately 3.6 % carbon. The CV value of the concentration obtained from the three measurements was 1.37 %, showing that the measurements were conducted with good repeatability. The carbon content in rock such as oil shale can be quantified if the TOC solid sample measurement system is used.

Table 2 Measurement Results

Measurement No.	Total carbon content (%)
1	3.62
2	3.60
3	3.52
Average	3.58

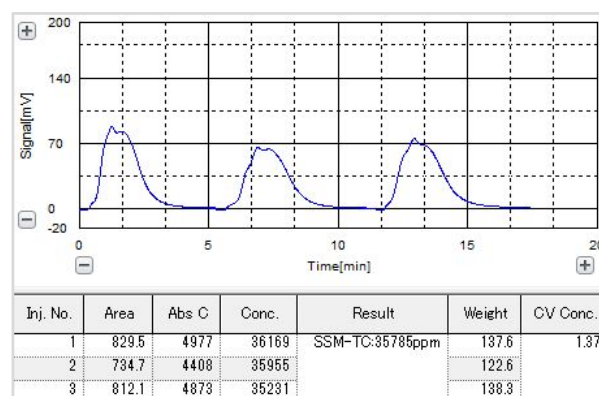


Fig. 5 Measurement Data