

Application News

No. 062

Total Organic Carbon Analysis

Measurement of Oil Residues on Metal Pipes Using the TOC Solid Sample Measurement System

Various metalworking fluids are used in the process of working metal materials. The fluids used vary according to the type of metal and functionality that is in need. Their main components include oils, surfactants, and pH control agents to achieve functions such as lubricity, durability, and cleanability. Depending on the residual amount of such fluids, the quality of the product in which the metal parts are incorporated may be adversely affected, which is why the evaluation of the residual content of metalworking fluids is considered important in quality control.

The TOC-L Total Organic Carbon Analyzer and SSM-5000A Solid Sample Combustion Unit comprise a system which can measure the amount of organic substances contained in a solid sample. In this research, we prepared stainless steel pipes as metal material samples, and used this system to measure the oil residues on the surface of the stainless steel pipes.

Y. Ikezawa

TOC Solid Sample Measurement System

The TOC solid sample measurement system (Fig. 1) comprises the Shimadzu TOC-L Total Organic Carbon Analyzer and SSM-5000A Solid Sample Combustion Unit. It oxidizes organic substances in a solid sample by combustion, and quantifies the organic substances by detecting carbon dioxide generated from the reaction. Simple and fast organic substance analysis is possible because the sample is combusted directly and measurement is done, thereby eliminating the need for sample pretreatment such as extraction. Table 1 lists the measurement conditions.

Table 1 Measurement Conditions

Analyzer	: TOC Solid Sample Measurement System TOC-L _{CPH} Total Organic Carbon Analyzer and SSM-5000A Solid Sample Combustion Unit (IC circuit bypassing on a system with a cell switching valve set added)
Cell length	: Short cell
SSM carrier gas	: 400 mL/min oxygen gas
TC oxidation method	: Combustion catalytic oxidation (TC furnace at 900 °C)
Measurement item	: TC
Calibration curve	: Single point calibration curve using 1 % C glucose solution
Sample	: Stainless steel pipes with an O.D. of 1.5 mm

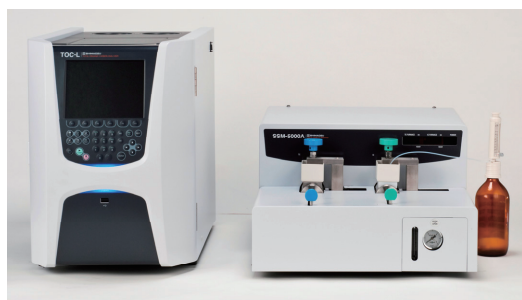


Fig. 1 TOC Solid Sample Measurement System

Measurement Procedure

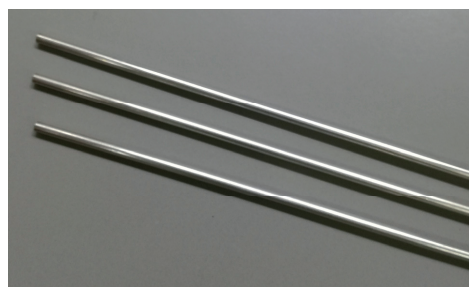


Fig. 2 Stainless Steel Pipes

Stainless steel pipes (Fig. 2) were prepared as samples. As indicated below, pipes without cleaning were used as sample A, and cleaned pipes were used as sample B.

Sample A: Pipes in the state that they were obtained without treatment

Sample B: Pipes cleaned with a laboratory cleaning detergent and rinsed with water

Measurement was done by the following procedure.

- (1) Cut the stainless steel pipe into segments approx. 3 cm long using diagonal cutting pliers with cutting edges cleaned with ethanol.
- (2) Place the pipe segments in the sample boat of the SSM-5000A (Fig. 3) and weigh using an electronic scale.
- (3) Set the sample boat in the sample port of the SSM-5000A and start TC measurement.



Fig. 3 Stainless Steel Pipes in the Sample Boat

■ Calibration Curve Creation

The analyzer was calibrated by generating a calibration curve using 20 µL of 1 % C (1 % carbon concentration) glucose solution, by adding the solution to heat-treated quartz wool placed in the sample boat and performing TC measurement.

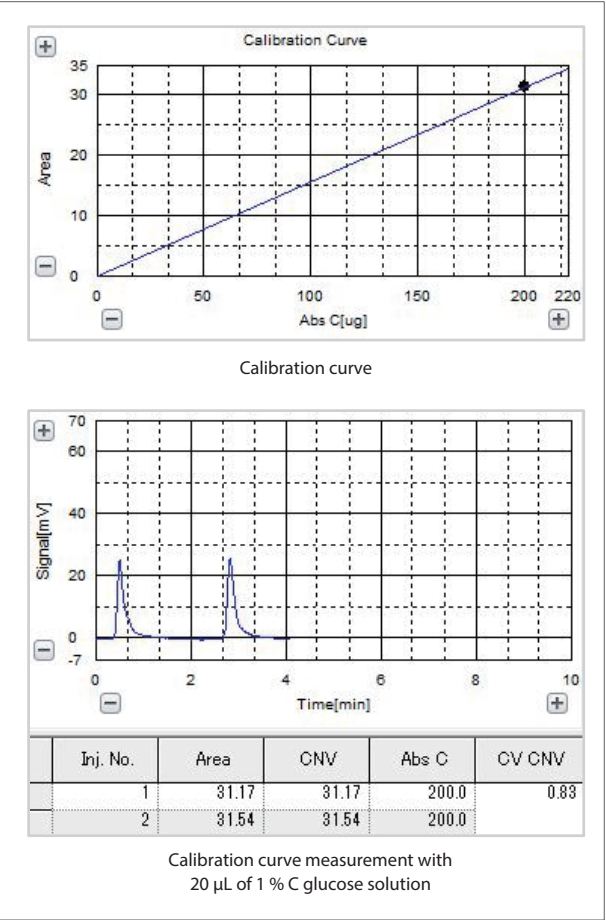


Fig. 4 Calibration Curve Measurement Data

■ Measurement Results of Samples

Measurement results of samples A and B are shown in Table 2, and Fig. 5 shows the data obtained by measurement. The sample A results indicate that oil residues is successfully quantified. From the sample B results, we can quantitatively see the effects of cleaning.

Table 2 Measurement Results

Sample name	TC concentration (ppm)
Sample A (not cleaned)	20.76
Sample B (cleaned)	1.79

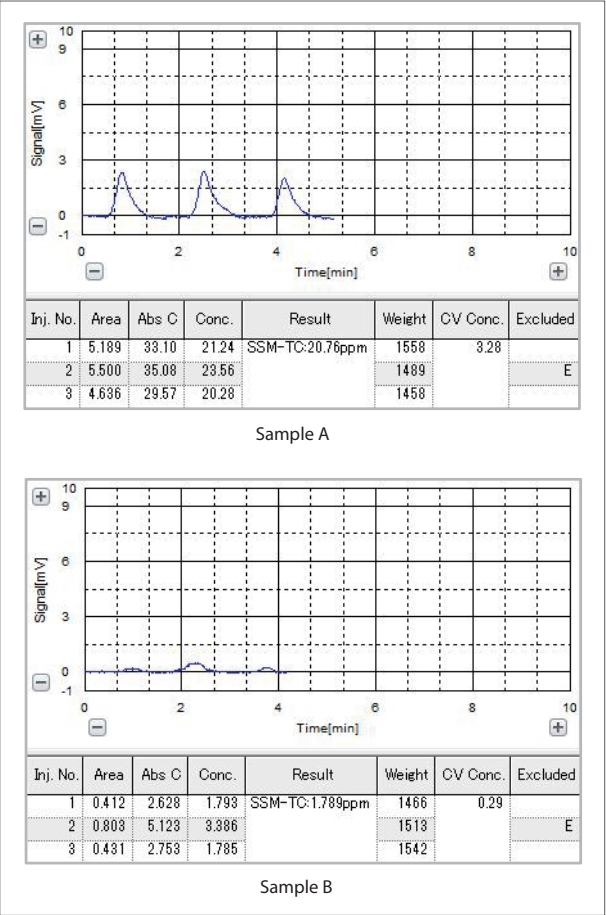


Fig. 5 Measurement Peak Data



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