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Application News

TOC-L Total Organic Carbon Analyzer + SSM-5000A Solid Sample Combustion Unit

Evaluation of Oil Residues on Aluminum Foil Surfaces

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User Benefits

- The quality of metal foils, such as aluminum foil, can be managed swiftly and effortlessly.
- Organic contaminants, including oils that don't dissolve in extraction solutions with traditional methods, can also be measured.
- Given each measurement takes about 5 minutes, the quick and easy analysis is also suitable for handling large number of samples.

■ Introduction

Aluminum foil is integral to a variety of applications, especially in determining the quality of certain electronic components like aluminum electrolytic capacitors. One of the critical factors influencing foil quality is residues of oil applied as a lubricant on its surface during the rolling process. To ensure optimal product performance, it is essential to manage the oil levels on the surface of aluminum foil.

The Shimadzu TOC-L total organic carbon analyzer combined with an SSM-5000A solid sample combustion unit serves as a system for measuring carbon content in solid samples. Unlike traditional methods, which often require laborious extraction with reagents, this method can identify organic impurities, such as non-soluble oils in the extraction solutions. That results in a swift and straightforward approach to quality control of aluminum foil.

This article describes an example of measuring the oil on the surface of aluminum foil using the Shimadzu TOC solid sample measurement system.

■ TOC Solid Sample Measurement System

Fig. 1 shows the Shimadzu solid sample measurement system, which is composed of a TOC-L total organic carbon analyzer and an SSM-5000A solid sample combustion unit. When employing this system to measure the total carbon (TC) content of a solid sample, the sample is heated to 900 °C in an oxygen-rich environment, thereby converting its carbon content into carbon dioxide (CO_2). By quantifying the generated CO_2, the amount of total carbon can be determined. Owing to its capability to burn and oxidize the oil residues on the aluminum foil's surface directly, the solid sample measurement system bypasses the need for cumbersome preliminary processes like extraction. This facilitates a rapid and efficient analysis of oil residues.

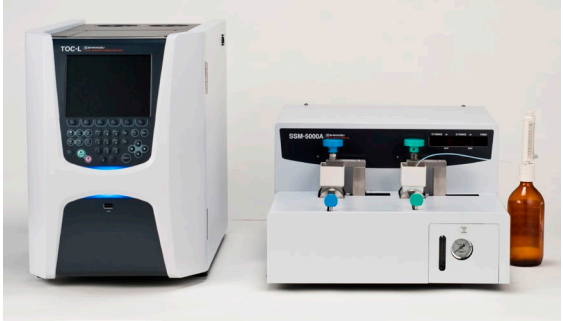


Fig. 1 TOC Solid Sample Measurement System
TOC-L Total Organic Carbon Analyzer (left) and SSM-5000A Solid Sample Combustion Unit (right)

■ Analysis Method

The base oil (made by Conostan, 20 cSt, with an 86 % carbon content) was diluted with ethanol to create a simulated sample of oil residues.

This diluted solution was spread onto an aluminum foil of dimensions 3 cm × 10 cm and left to dry in a fume hood for approximately an hour to evaporate the ethanol (Fig. 2). As shown in Fig. 3 and Fig. 4, the aluminum foil was then rolled and positioned into the sample boat of the TOC solid sample measurement system for TC analysis.

Three distinct simulated samples were prepared, representing theoretical carbon values of 43, 86, and 258 µg.

Fig. 2 Aluminum Foil during Drying

Fig. 3 Aluminum Foil Placed in the Sample Boat

Fig. 4 Sample Boat Set in the TC Sample Loading Section

The measurement conditions for the solid sample measurement system are detailed in Table 1.

Considering the requirement to quantify trace amounts of carbon in this experiment, the "Cell Switching Valve Set - Specification A" (an optional feature) was utilized, setting the absolute carbon quantitation range between 15 and 300 µgC. Using this option, modifications to the system's internal flow can be made to boost the TC detection sensitivity.

A 1 % carbon-concentrated glucose solution (30 µL), corresponding to an absolute carbon amount of 300 µg, was used to establish the one-point calibration curve.

Table 1 Measurement Conditions

Instrument	TOC solid sample measurement system (TOC-L_{CPH} total organic carbon analyzer + SSM-5000A solid sample combustion unit)
Cell Length	Short cell
SSM Carrier Gas	400 mL/min oxygen gas
TC Measurement Method	Combustion catalytic oxidation (TC furnace: 900 °C)
Measurement Item	TC (total carbon)
Calibration Curve	One-point calibration curve using a 1% glucose solution (30 µL with 300 µg absolute carbon)

■ Measurement Results

The results of the TC measurements of the oil residues on the aluminum foil surface are presented in Table 2, while Fig. 5 shows the associated TC measurement data. Recovery rates ranged from 95.5 % to 104 % for all samples. The coefficient of variation (CV) remained less than 2 %, indicating reliable measurement repeatability.

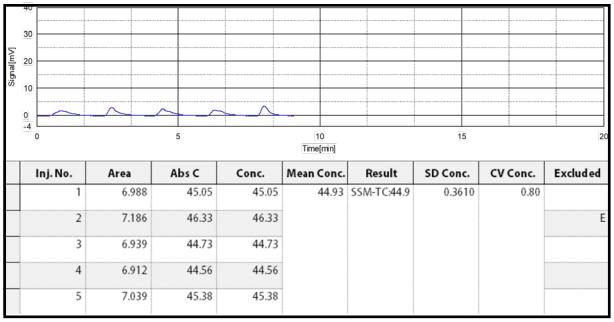
As shown in Fig. 5, the peak shape varied across measurements even for identical samples. Nonetheless, since the TOC solid sample measurement system derives the TC value from the peak area, variations in peak shape don't affect the final results.

Based on these findings, the TOC solid sample measurement system has proven its capability to accurately evaluate the oil present on the aluminum foil surface.

Table 2 TC Measurement Results of the Added Oil

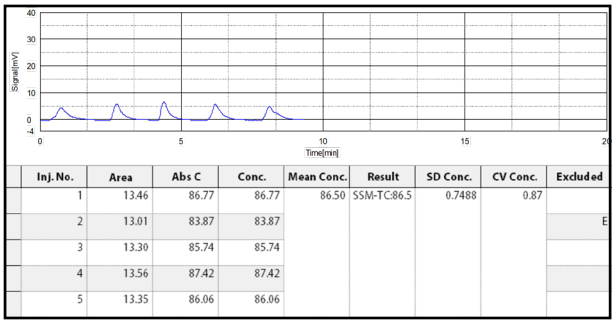
Samples	Theoretical Carbon Content [µgC]	Measured Carbon Content [µgC]	Recovery Rate [%]
Aluminum Foil + Ethanol	0	0	-
Aluminum Foil + Oil (1)	43	44.9 (CV: 0.80 %)	104
Aluminum Foil + Oil (2)	86	86.5 (CV: 0.87 %)	101
Aluminum Foil + Oil (3)	258	247 (CV: 1.85 %)	95.5

CV: Coefficient of variation



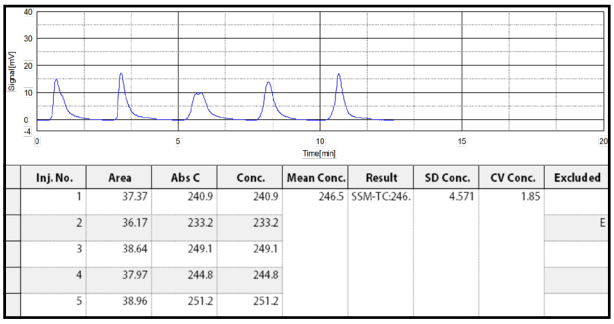
Inj. No.	Area	Abs C	Conc.	Mean Conc.	Result	SD Conc.	CV Conc.	Excluded
1	6.988	45.05	45.05	44.93	SSM-TC:44.9	0.3610	0.80	
2	7.186	46.33	46.33					E
3	6.939	44.73	44.73					
4	6.912	44.56	44.56					
5	7.039	45.38	45.38					

(a) Aluminum Foil + Oil (1)



Inj. No.	Area	Abs C	Conc.	Mean Conc.	Result	SD Conc.	CV Conc.	Excluded
1	13.46	86.77	86.77	86.50	SSM-TC:86.5	0.7488	0.87	
2	13.01	83.87	83.87					E
3	13.30	85.74	85.74					
4	13.56	87.42	87.42					
5	13.35	86.06	86.06					

(b) Aluminum Foil + Oil (2)



Inj. No.	Area	Abs C	Conc.	Mean Conc.	Result	SD Conc.	CV Conc.	Excluded
1	37.37	240.9	240.9	246.5	SSM-TC:246.	4.571	1.85	
2	36.17	233.2	233.2					E
3	38.64	249.1	249.1					
4	37.97	244.8	244.8					
5	38.96	251.2	251.2					

(c) Aluminum Foil + Oil (3)
Fig. 5 TC Measurement Results of the Added Oil

■ Conclusion

Using the TOC solid sample measurement system, the absence of steps like extraction allows for a swift and effortless evaluation of oil residues on the surface of aluminum foil to ensure the quality of the aluminum foil. The direct measurement can capture organic impurities, including oils that are not soluble in the extraction solutions. Moreover, with each measurement taking roughly 5 minutes, it offers an efficient method suitable for a large number of samples.

In addition to aluminum foil, it can also be used to manage organic residues on various other metals and inorganic materials, such as copper sheets and wires. The effectiveness of the TOC solid sample measurement system can be expected to be especially useful for such applications.

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