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Application News

Total Organic Carbon Analyzer

Evaluation of Organic Impurities in Lithium Carbonate by TOC Analysis

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User Benefits

- TOC analysis enables effective control of organic impurities in lithium carbonate.
- Using the combustion tube kit for high-salt samples can extend the lifespan of the combustion tube and catalyst, reducing maintenance frequency.
- With the ASI-L autosampler, multiple samples can be measured automatically, improving throughput and efficiency.

■ Introduction

Lithium carbonate (Li_2CO_3) is an important material used in a wide range of applications in the lithium-ion battery industry. For example, it is used as a lithium source for cathode active material synthesis and electrolyte salt production, as a raw material for oxide-based solid electrolytes, and as a form of lithium commonly recovered in recycling processes.

Because impurities in lithium carbonate can affect downstream processes and final products, appropriate control is essential. In particular, organic impurities require especially careful attention because they may originate from so many sources, such as solvents and processing aids used during purification, residual organics from manufacturing steps, or contamination introduced during handling and storage.

Total organic carbon (TOC) analysis offers an effective way to manage such organic impurities in raw materials. This article presents an example of using a Shimadzu TOC-L total organic carbon analyzer (Fig. 1) to evaluate the TOC content of lithium carbonate.

Fig. 1 TOC-L Total Organic Carbon Analyzer

■ Sample Preparation

Samples for TOC Analysis

Reagent-grade lithium carbonate (1.00 g) was dissolved with pure water and then 3.0 % (v/v) of 4.5 mol/L sulfuric acid to make 100 mL of a 10 g/L lithium carbonate solution.

Sulfuric acid was added to ensure complete dissolution of the lithium carbonate, which has low solubility in water. During preparation, stirring and ultrasonic treatment were applied as needed.

A blank solution was prepared in the same manner, but without lithium carbonate.

Samples for Spike-Recovery Testing

To verify method validity, potassium hydrogen phthalate was added to the 10 g/L lithium carbonate solution described above to prepare spiked samples with TOC concentrations of 1, 2, and 5 mgC/L, respectively.

The measurement samples are listed in Table 1.

Table 1 Measurement Samples

	Sample Description
S0	Pure Water (blank)
S1	10 g/L Li_2CO_3 Solution
S2	10 g/L Li_2CO_3 Solution + 1 mgC/L TOC
S3	10 g/L Li_2CO_3 Solution + 2 mgC/L TOC
S4	10 g/L Li_2CO_3 Solution + 5 mgC/L TOC

■ TOC Analysis

TOC Analysis Methods

Two commonly used methods are available for TOC analysis:

(1) TC - IC method: Total carbon (TC) and inorganic carbon (IC) are measured separately, and then TOC is calculated as the difference (TOC = TC – IC).

(2) NPOC method: TOC is measured as non-purgeable organic carbon (NPOC). After acidifying the sample to pH < 3, IC is subsequently removed by sparging and the remaining carbon is measured as TC, which is regarded as TOC. The acidification and sparging steps are performed automatically by the instrument.

For lithium carbonate solutions, the amount of carbonate-derived IC is typically very high. As a result, the TC–IC method tends to yield calculated TOC values with a larger relative error. Therefore, the NPOC method is recommended.

In lithium carbonate solutions prepared as described above, IC is expected to be present mainly as bicarbonate (HCO_3^-) and carbonic acid (H_2CO_3). If IC is not fully removed during NPOC pretreatment, the TOC result may be overestimated. Therefore, thorough IC removal is critical for samples with high IC content. For actual samples, sparging parameters, such as sparge gas flow rate and sparge time settings, can be adjusted.

Combustion Tube for High-Salt Samples

The prepared lithium carbonate solution contains a large amount of salts. When a large number of salt-containing samples are analyzed, salts may accumulate inside the combustion tube, which can lead to catalyst clogging, reduced sensitivity, and poorer repeatability.

For this analysis, the optional combustion tube for high-salt samples was used, shown in Fig. 2. The high-salt combustion tube has a larger diameter than the standard combustion tube and contains larger catalyst particles to help mitigate salt-related clogging. Thus, the frequency of combustion tube replacement can be significantly reduced.

Fig. 2 Combustion Tube for High-Salt Samples

Acid Solution Used for NPOC Method

Hydrochloric acid or sulfuric acid is commonly used for acidification in the NPOC method. However, the salts formed upon acid addition should be carefully considered. If hydrochloric acid is used for lithium carbonate samples, lithium chloride is formed. Since lithium chloride has a melting point of approximately 610 °C, which is lower than the TOC-L combustion temperature (680 °C), salt melting inside the combustion tube may occur. At high concentrations, molten salts can be carried downstream as mist and affect analytical results. They may also devitrify the combustion tube, potentially causing instrument damage.

For those reasons, sulfuric acid was used in this experiment, both for sample preparation and for acidification in the NPOC measurement. Lithium sulfate formed upon sulfuric acid addition has a melting point of 859 °C, which is higher than the combustion temperature and therefore prevents salt melting.

In addition, because sulfuric acid had already been added during preparation of the lithium carbonate solution, the acid addition rate in the NPOC method was set to a relatively low value of 0.5 %.

Detailed analytical conditions are indicated in Table 2.

Table 2 TOC Analysis Conditions (NPOC Method)

Instrument	TOC-L_{CPH} + combustion tube for high-salt samples + B-type halogen scrubber
Catalyst	TOC catalyst for high-salt samples
Substances Measured	TOC (NPOC method)
NPOC Method	Acid addition: 4.5 mol/L sulfuric acid, 0.5 %
	Sparging treatment: 80 mL/min, 90 seconds
Injections	2 or max. 3 injections
Option	ASI-L autosampler
Calibration Curves	NPOC: 4-point calibration curve using potassium hydrogen phthalate aqueous solutions at 0, 2, 5, and 10 mgC/L

■ Calibration Curve

The 4-point NPOC calibration curve, prepared using potassium hydrogen phthalate aqueous solution at 0, 2, 5, and 10 mgC/L, is shown in Fig. 3. To correct for the carbon contribution from the pure water used to prepare the standards, the calibration curve was adjusted by shifting the origin before use. The correlation coefficient (r) was 0.9999, indicating excellent linearity.

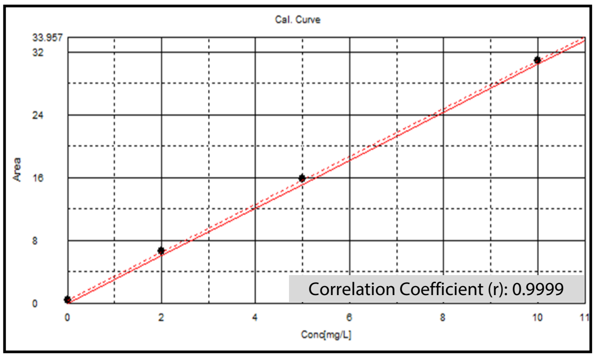


Fig. 3 NPOC Calibration Curve

■ Results

TOC Analysis Results

Table 3 summarizes the measured TOC values for the blank solution and the 10 g/L lithium carbonate solution, as well as the TOC value converted to a solid basis for lithium carbonate.

The results of the spike-recovery test are shown in Table 4. Recovery rates of 100 ±2 % were obtained for all levels, demonstrating excellent recovery.

These results confirm that TOC in the 10 g/L lithium carbonate solution can be measured accurately.

Table 3 TOC Analysis Results

	Sample	Measured TOC (mgC/L)	TOC in Lithium Carbonate Solid (mgC/kg)
S0	Pure Water (blank)	0.09899	-
S1	10 g/L Li_2CO_3 Solution	0.2912	19.2

TOC in the solid = (TOC of S1 − TOC of S0) × total solution volume / mass of the weighed solid sample

Table 4 Results of the Spike-Recovery Test

	Spiked TOC Level (mgC/L)	Measured TOC (mgC/L)	Recovery (%)
S2	1	1.279	98.8
S3	2	2.318	101
S4	5	5.250	99.2

Recovery (%) = (TOC of spiked sample − TOC of S1) / spiked TOC level × 100

Consistency in Continuous Analysis

To evaluate analytical consistency for lithium carbonate solutions with high salt content, long-term continuous analysis was performed. Consistency was assessed after sample S2 was analyzed continuously for approximately 7 hours.

The summary of the long-term continuous analysis is shown in Table 5 and the normalized TOC values for each sample are shown in Fig. 4. As a result of measuring 47 consecutive samples over approximately 7 hours, a favorable coefficient of variation (CV) less than 5 % was obtained. When the mean TOC of all measurements was normalized to 100 %, the normalized TOC values remained within 100 ±10 %, confirming good consistency.

Table 5 Summary of the Long-Term Continuous Analysis

Duration (h)	Number of Samples	Mean TOC (mgC/L)	CV (%)
Approx. 7	47	1.278	2.6

Example Measurement Data

Examples of TOC analysis data are shown in Fig. 5 and Fig. 6.

An example of peak results obtained from 20 consecutive injections is shown in Fig. 7.

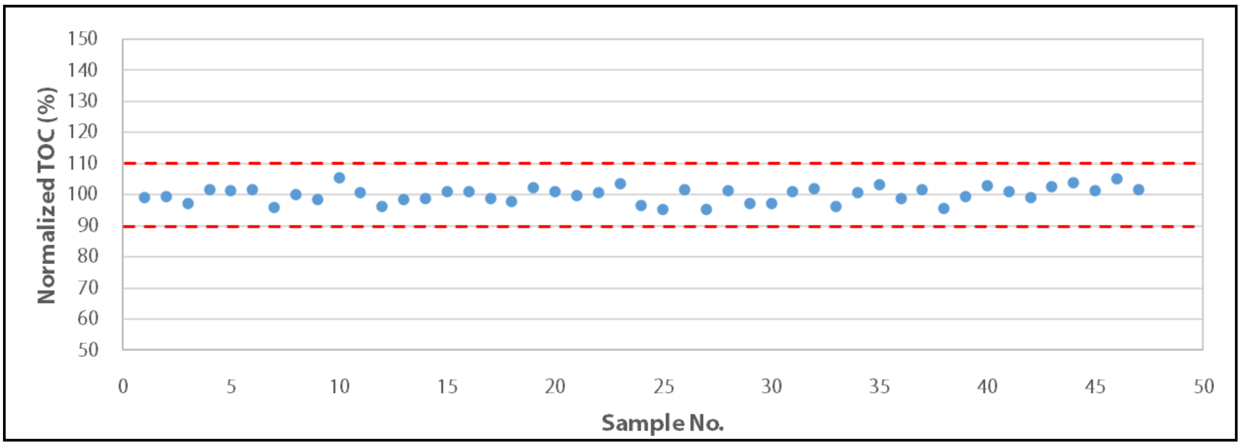
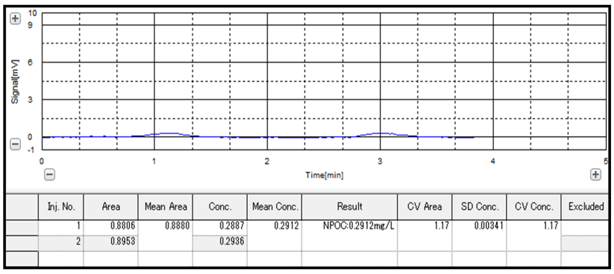


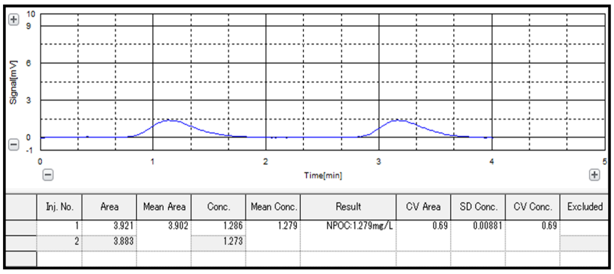
Fig. 4 Normalized TOC (%) during Continuous Analysis

Normalized TOC (%) = (TOC of each sample / mean TOC of all samples) × 100



	Inj. No.	Area	Mean Area	Conc.	Mean Conc.	Result	CV Area	SD Conc.	CV Conc.	Excluded
	1	0.8806	0.8880	0.2887	0.2912	NPOC:0.2912mg/L	1.17	0.00341	1.17	
	2	0.8953		0.2936						

Fig. 5 TOC Measurement Data for S1 (10 g/L Li_2CO_3 Solution)



	Inj. No.	Area	Mean Area	Conc.	Mean Conc.	Result	CV Area	SD Conc.	CV Conc.	Excluded
	1	3.921	3.902	1.286	1.279	NPOC:1.279mg/L	0.69	0.00881	0.69	
	2	3.883		1.273						

Fig. 6 TOC Measurement Data for S2 (10 g/L Li_2CO_3 Solution + 1 mgC/L TOC)

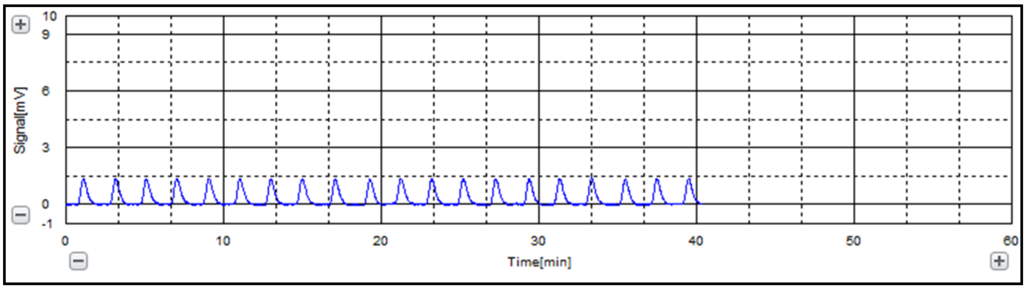


Fig. 7 Example of Peak Results from 20 Consecutive Injections of S2 (10 g/L Li_2CO_3 Solution + 1 mgC/L TOC)

■ Conclusion

In this article, TOC analysis was performed for a 10 g/L lithium carbonate solution using a Shimadzu TOC-L total organic carbon analyzer. By measuring TOC in the prepared solution, the TOC content in the lithium carbonate solid was calculated. The results showed good recovery rates in the spike-recovery test and consistent performance was also confirmed during long-term continuous analysis. These findings demonstrate that the TOC-L is a reliable solution for measuring TOC in lithium carbonate and for effective management of organic impurities.

When applying the NPOC method to lithium salt-containing solutions, careful selection of the acid is essential because salts formed during acidification can affect both data quality and instrument condition. For this example, sulfuric acid is recommended.

In addition, using the optional combustion tube kit for high-salt samples can extend the lifespan of the combustion tube and catalyst, thereby reducing maintenance frequency. This option is especially helpful for samples with high salt concentrations.

In addition to lithium carbonate, the TOC-L can also be applied to TOC analysis of lithium hydroxide solutions, determination of water-extractable TOC in recycled black mass, and analytical needs in lithium refining processes based on direct lithium extraction (DLE). With its broad applicability across various upstream materials, recycling streams, and refining processes, the TOC-L supports both quality control and process optimization operations throughout the lithium-ion battery value chain.

Related Applications

1. Determination of Water-Extractable Total Organic Carbon Content in Recycled Black Mass, Application News No. 01-00919-en
2. TOC and IC Measurements for Lithium Refining Processes, Application News No. 01-01018-en

01-01098A-EN
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