LAAN-A-MS-E030A

SHIMADZU

Application News

Gas Chromatography Mass Spectrometry No.M264A

Analysis of Electrolyte Solution in Lithium Ion Rechargeable Battery (LIRB) and Evolved Gas from LIRB

The electrolyte solution in a lithium ion rechargeable battery consists of an organic solvent (consisting chiefly of carbonates), the electrolyte, and additives. Evaluation of the electrolyte solution and analysis of its deterioration due to repetitive charge/discharge cycles is important for the further development of the lithium ion rechargeable battery. GC-MS is applicable for composition analysis of the electrolyte solution, and for analysis of components in the electrolyte solution that have been generated due to repeated charging and discharging.
This Application News introduces examples of analysis of the electrolyte solution of a lithium ion rechargeable battery and of the gas evolved from a cell that has been maintained at a high temperature.

■ Analytical Conditions

Analysis of the electrolyte solution was conducted using the analytical conditions of Table 1, and analysis of gas evolved from the cell was conducted using the conditions of Table 2. The evolved gas to be measured was collected from an aluminum-laminated lithium ion battery that had been stored for 5 days at 80 °C. The needle tip of a gas-tight syringe was inserted directly into the cell, and the gas phase was aspirated into the syringe.

Table 1 Analytical Conditions for Electrolyte Solution Analysis

Model	: GCMS-QP2010 Ultra		
Column	: Rtx-200MS (RESTEK Corporation) [30 m × 0.25 mm I.D. df = 1 µm]		
-GC-		-MS-	
Injector Temp.	: 250 °C	Interface Temp.	: 250 °C
Injection Method	: Split	Ion Source Temp.	: 200 °C
Split Ratio	: 1 : 100	Ionization Mode	: EI
Carrier Gas	: He (Constant Linear Velocity Mode)	Acq. Mode	: Scan
Linear Velocity	: 40 cm/sec	Scan Range	: *m/z* 35-500
Column Temp.	: 40 °C (3 min) - 8 °C/min - 280 °C (5 min)	Event Time	: 0.3 sec
Injection Volume	: 1 µL		

Table 2 Analytical Conditions for Evolved Gas Analysis

Model	: GCMS-QP2010 Ultra		
Column	: Rt-Q-BOND (RESTEK Corporation) [30 m × 0.32 mm I.D. df = 10 µm] + Guard column (RESTEK Corporation) [3 m × 0.32 mm I.D.]		
-GC-		-MS-	
Injector Temp.	: 200 °C	Interface Temp.	: 200 °C
Injection Method	: Split	Ion Source Temp.	: 200 °C
Split Ratio	: 1 : 30	Ionization Mode	: EI
Carrier Gas	: He (Constant Linear Velocity Mode)	Acq. Mode	: Scan
Linear Velocity	: 61.6 cm/sec	Scan Range	: *m/z* 10-300
Column Temp.	: 35 °C (3 min) - 10 °C/min - 260 °C (5 min)	Event Time	: 0.3 sec
Injection Volume	: 500 µL		

■ Results of Electrolyte Solution Analysis

Fig. 1 shows the results of analysis of the lithium ion rechargeable battery electrolyte solution. The dimethyl carbonate, ethyl methyl carbonate and ethylene carbonate used as the solvent were identified from a mass spectral library search. In addition, vinylene carbonate used for the additive was also identified.

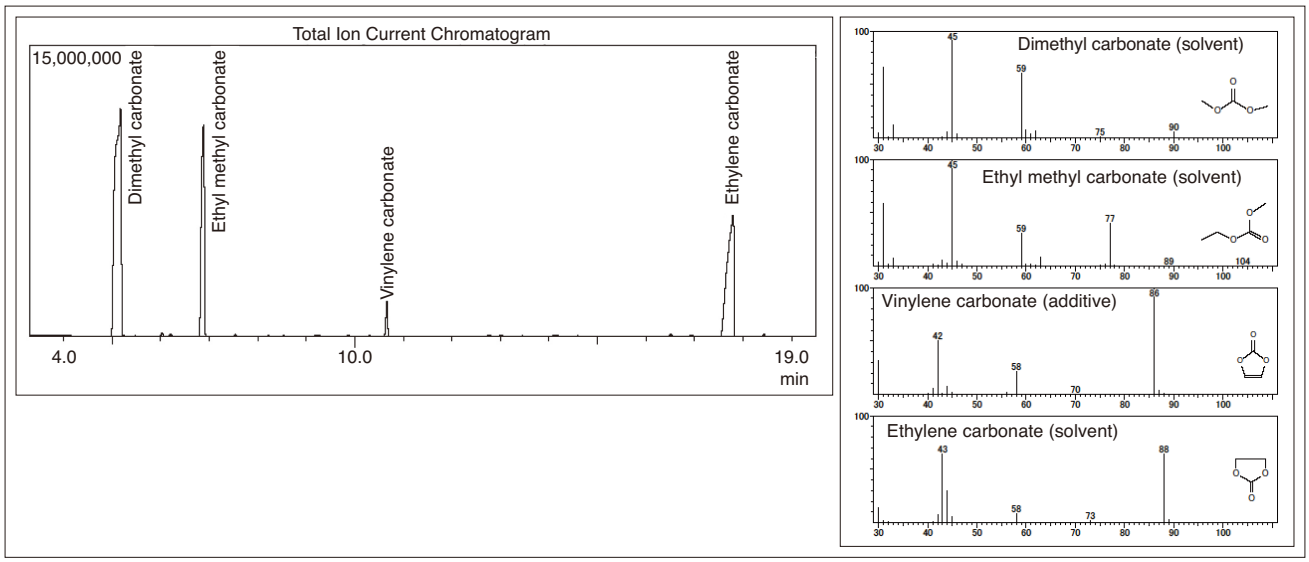

Fig. 1 Total Ion Current Chromatogram (TICC) of Electrolyte Solution and Mass Spectra of Target Compounds

■ Analytical Results for Evolved Gas from LIRB

The results obtained from analysis of the gas evolved from the cell are shown in Fig. 2. Many substances that originated from the degenerated solvent and additive, as well as from the electrolyte, were identified. Moreover, a fluorinated compound of electrolyte origin was also identified in the library search.

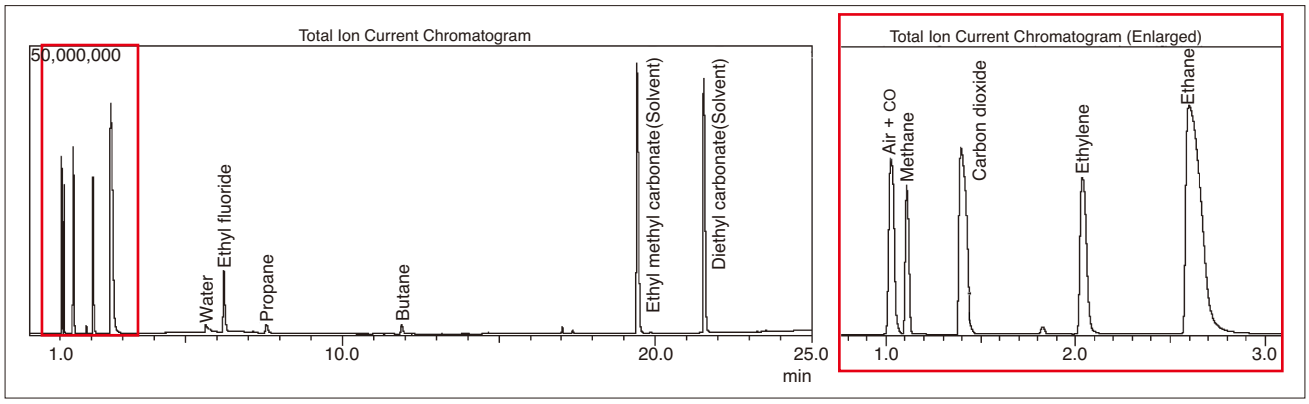

Fig. 2 Total Ion Current Chromatogram (TICC) of Evolved Gas from Lithium Ion Rechargeable Battery

■ Analytical Note

A PLOT column solid phase consisting of an adsorbent or porous polymer is suitable for analysis of gases and low-boiling compounds. However, compared to a capillary column that uses a liquid phase, there is a greater risk of particle scattering, resulting in clogging or ion source contamination. It is therefore necessary to connect a 2 to 3 m guard column at the interface end of the PLOT column to lessen the risks associated with particle scattering.

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