Gas Chromatograph-Mass Spectrometry / GCMS-QP2020 NX

Application News

Simultaneous Analysis of 19 Organic Solvents and Additives in Lithium-Ion Battery Electrolytes Using GC-MS

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User Benefits

- Simultaneous analysis of 19 organic solvents and additives reduces analytical time and improves efficiency.
- This method is suitable for the characterization of commercial LIB electrolyte formulations.

■ Introduction

Lithium-ion batteries (LIBs) are eco-friendly, rechargeable energy storage devices that provide a sustainable alternative to fossil fuels. They are widely used to power electric vehicles and store energy generated from renewable sources, thereby reducing dependence on fossil fuels and lowering greenhouse gas emissions. As a result, LIBs play a essential role in the transition toward low-carbon and sustainable energy systems.

The electrolytes in LIB consists of a lithium salt dissolved in a mixture of organic solvents and formulated with various functional additives. The organic solvents act as the vehicle for lithium-ion transport between electrodes, while the additives improve electrolytes stability, safety, and overall performance by enabling stable operation under various conditions. Both organic solvents and additives play an essential roles in the function and performance of LIBs.

Accurate control of electrolytes composition is critical, as even minor impurities in the solvents or additives can result in significant safety risks and degradation of battery performance. Therefore, the analysis of electrolytes composition and its operational degradation products is essential for ensuring the safety, stability, and quality of LIBs throughout their development and production. In a previous Application News, the analysis of seven compounds used as organic solvents and additives in LIB electrolytes using GC-FID was proposed.[1]

In this study, an analytical method of 19 organic solvents and additives commonly used in LIBs electrolytes was developed. The analysis was carried out using the Shimadzu Nexis™ GC-2030 gas chromatograph coupled with the QP2020NX single quadrupole mass spectrometer. The developed method was successfully applied to commercial LIB electrolyte sample.

Figure 1. Nexis GC-2030 + QP2020 NX

■ Materials & Method

Analytes and Analytical Conditions

In this study, 19 target compounds mainly used as organic solvents and additives in the LIB electrolytes were selected and then analyzed for simultaneous analysis using SHIMADZU GC-2030 + QP2020NX (Figure 1).

All 19 standard compounds were obtained from TCI Chemical (Tokyo, Japan) and dichloromethane (DCM) was purchased from Tedia (Fairfield, OH, USA). Detailed analytical conditions of the instrument are shown in Table 1 and 2.

Table 1. Analytical method of GC-MS

GC System	Nexis GC-2030
Carrier Gas	: He (99.999 %)
Flow Control Mode	: Constant linear velocity
Liner Velocity	: 36 cm/s
Injector Temp.	: 250 °C
Injection Mode	: Split (10:1)
Column Flow	: 1.0 mL/min
Analytical Column	: SH-1701 (30 m x 0.25 mm ID, 0.25 μm)
Column Temp.	: 35 °C (3 min) → 10 °C/min → 240 °C (5 min)
MS System	**QP2020NX**
Ionization Method	: EI Mode
Ion source Temp.	: 250 °C
Interface Temp.	: 300 °C
Acquisition Mode	: SIM mode (refer to Table 2)

Table 2. The retention times and the selected ions used for quantitative and qualitative analysis of the 19 target compounds.

No.	Compounds	Abbreviation	Cas No.	Retention time (min)	Quantitative ion (m/z)	Qualitative ion (m/z)
1	Ethyl acetate	EA	141-78-6	2.855	43	29, 45
2	Tris(trimethylsilyl) Phosphite	TMSP	1795-31-9	2.865	147	73, 131
3	Methyl propionate	MP	554-12-1	3.08	57	59, 88
4	Dimethyl carbonate	DMC	616-38-6	3.147	45	31, 59
5	Fluorobenzene	FB	462-06-6	3.608	96	70, 50
6	Ethyl propionate	EP	105-37-3	4.486	57	29, 74
7	Ethyl methyl carbonate	EMC	623-53-0	4.677	45	29, 77
8	Diethyl carbonate	DEC	105-58-8	6.36	45	31, 91
9	n-Propyl propionate	PP	106-36-5	6.526	57	75, 43
10	Vinylene carbonate	VC	872-36-6	7.694	86	29, 42
11	Fluoroethylene carbonate	FEC	114435-02-8	11.045	29	62, 33
12	1,1–dimethylpropylbenzene (tert-Amylbenzene)	TAB	2049-95-8	11.428	119	91, 41
13	Ethylene carbonate	EC	96-49-1	13.106	29	43, 88
14	Propylene Carbonate	PC	108-32-7	13.191	57	43, 30
15	Vinyl ethylene carbonate	VEC	4427-96-7	14.082	42	39, 40
16	Succinonitrile	SN	110-61-2	14.198	53	40, 79
17	Ethylene sulfate (1,3,2-Dioxathiolan-2,2-oxide)	DTD	1072-53-3	15.309	31	30, 29
18	1,3-Propanesultone	PS	1120-71-4	16.652	58	29, 57
19	1,4-Dicyanobutane (Adiponitrile)	ADN	111-69-3	16.976	41	68, 54

Preparation of standard solutions

A total of 19 target compounds were used as standard materials. The individual stock solutions were prepared by diluting each standard compound in DCM to a concentration of 1,000 µg/mL.

A multi-standard solution was prepared by adding 500 µL of each individual stock solution to a 10 mL volumetric flask, resulting in a final concentration of 50 µg/mL for each of the 19 target compounds in DCM.

For calibration curve preparation, the multi-standard solution was further diluted with DCM to obtain standard solutions with final concentrations of 0.1, 0.25, 0.5, 1, 2.5, 5, and 10 µg/mL.

Sample preparation

The method was applied to commercial electrolyte sample containing lithium salts (LiFSI and $LiPF_6$), three organic solvents (EC, EMC, DMC), and four additives (VC, FEC, PS, SN).

For analysis, liquid electrolyte samples were diluted by transferring 0.1 mL of the sample into a 100 mL volumetric flask, resulting in a 1000-fold dilution. Subsequently, to accurately quantify compounds detected at high concentrations, additional dilutions (10-fold or 100-fold) were performed using the initial diluted solution. The final diluted samples were analyzed quantitatively using GC-MS.

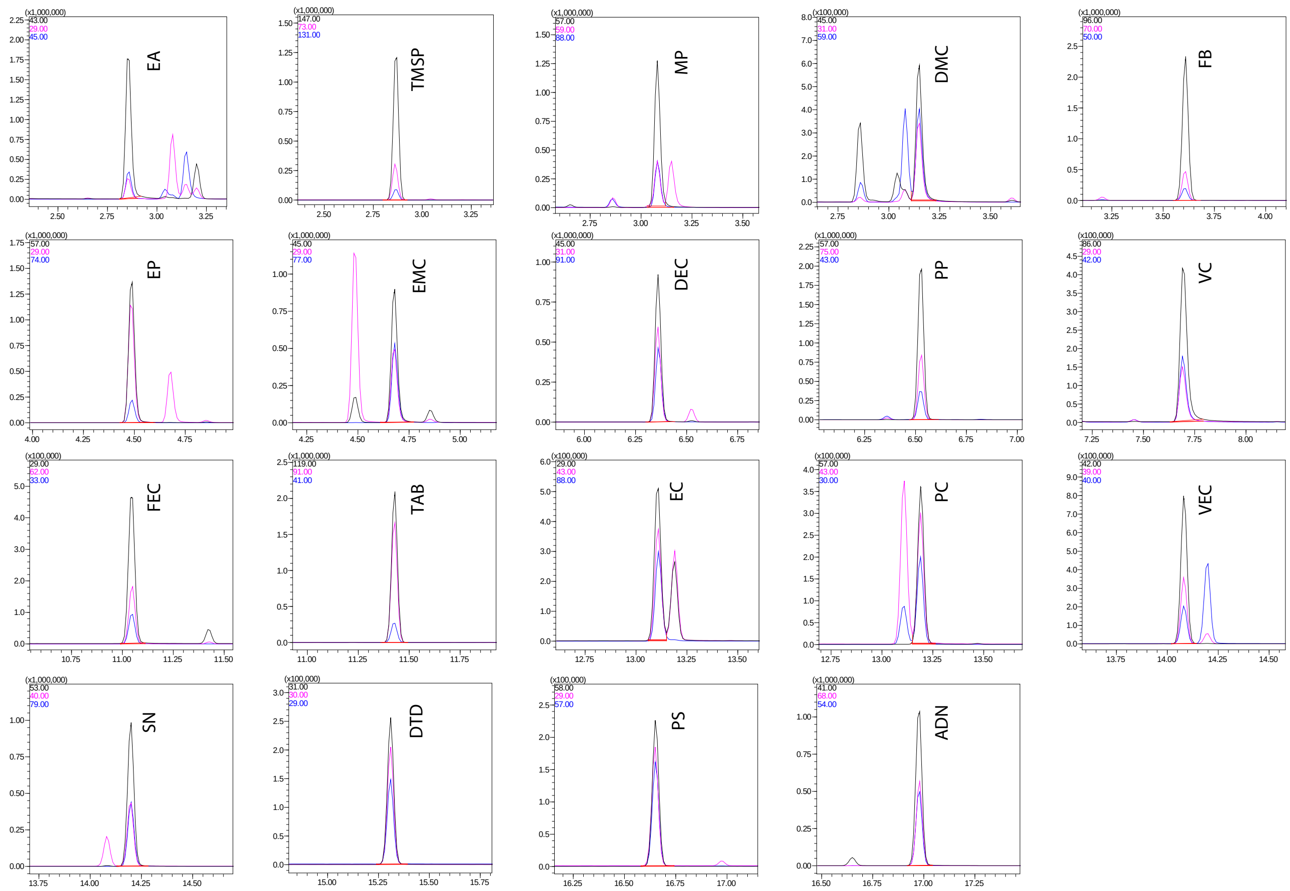

Figure 2. SIM chromatograms of 19 organic solvents and additives (10 µg/mL)

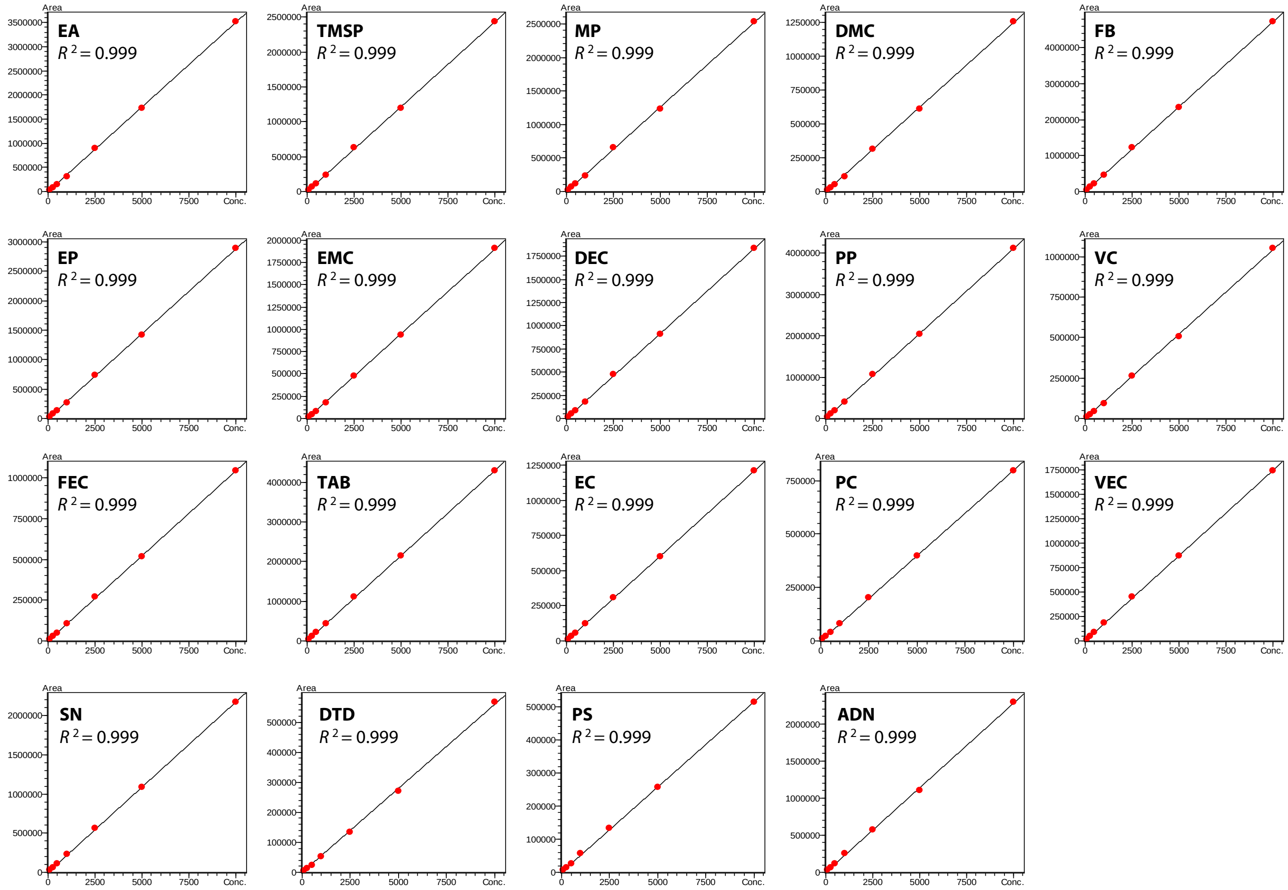

Figure 3. Linearity of calibration curve for 19 target compounds.

■ Results

The retention times and selected ions listed in Table 2 were determined by analyzing individual standard solutions under optimized GC-MS conditions. For quantification, the ions exhibiting the highest intensity and selectivity were selected. The SIM chromatograms of 19 organic solvents and additives are shown in Figure 2. The calibration curves for all standard compounds are excellent linearity, with correlation coefficients (R^2) greater than 0.999 (Figure 3).

The developed analytical method was successfully applied to actual LIB electrolyte samples, confirming the presence and quantifying the concentrations of three organic solvents (EC, EMC, DMC) and four additives (VC, FEC, PS, SN), as summarized in Table 3.

Table 3. Detected concentrations of organic solvents and additives in commercial LIB electrolyte samples

Compounds	Concentration (mg/mL)
DMC	471.1
EMC	497.4
EC	193.7
VC	30.6
FEC	13.5
PS	3.9
SN	13.4

■ Conclusions

In this study, a simultaneous GC-MS method was developed and validated for the quantitative analysis of 19 organic solvents and additives in LIB electrolytes. The calibration curves demonstrated excellent linearity for all target compounds. Furthermore, The method was successfully applied to actual LIB electrolyte samples. This analytical method provides a reliable approach for lithium-ion battery research and quality assessment.

■ Reference

1. Analysis of Carbonic Esters and Additives in Lithium Ion Battery Electrolytes, [Application News No.01-00708-EN]

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