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Application News

LCMS-2050 High Performance Liquid Chromatograph Mass Spectrometer

Analysis of Organic EL Materials and Impurities Using Single Quadrupole Mass Spectrometer

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User Benefits

- The synthesis of organic EL materials can be easily confirmed using a single quadrupole mass spectrometer in the same space as an HPLC system.
- Equipped standard with a heated DUIS™ unit, the system can even ionize compounds with low polarity, such as polycyclic aromatics.
- By combining a single quadrupole mass spectrometer with the HPLC unit, the system can be used to check for known impurities or easily predict the molecular weight of unknown impurities.

■ Introduction

Organic EL materials represent a family of mostly polycyclic aromatic light-emitting compounds used to make organic light-emitting diode displays and other products. Because high-quality organic EL materials are required for developing high performance displays, the concentration of impurities contained in the materials must be controlled and evaluated.

This article describes an example of using a high performance liquid chromatograph and single quadrupole mass spectrometer to check the synthesis of organic EL materials and predict the molecular weights of impurities they contain.

Nexera™ High Performance Liquid Chromatograph and LCMS™-2050 Single Quadrupole Mass Spectrometer

■ Checking the Synthesis of Organic EL Materials by LC/MS

One of the typical techniques used to determine whether or not an organic EL material has been synthesized is to check molecular weights with a mass spectrometer. If the composition of the intended substance being measured is already predicted, such as when checking synthesis, then accurate mass measurements are not necessarily required. This article describes an example of using a single quadrupole mass spectrometer to check molecular weights in organic EL materials.

■ Target Compounds being Measured

In this example, laboratory reagents were used to measure seven kinds of commercial organic EL materials (Fig. 1). A standard sample of each compound was prepared by dissolving the compound in tetrahydrofuran to a concentration of 1 mg/mL and then diluting that solution by a factor of ten (tetrahydrofuran/methanol = 1/1 (v/v)).

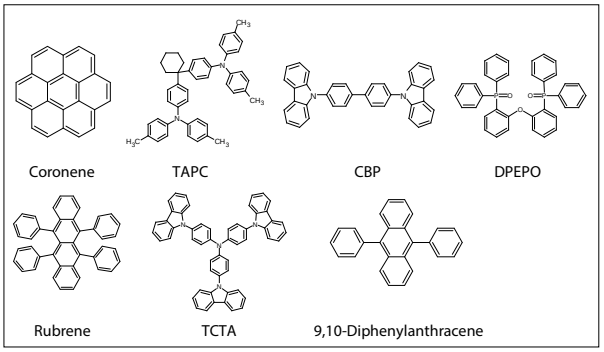

Fig. 1 Target Compounds being Measured

■ Instruments and Analytical Conditions

The conditions used for analysis are listed in Table 1.
The LCMS-2050 single quadrupole mass spectrometer unit and LC unit are about the same size. They can be included in existing Nexera™ series or i-Series LC systems.

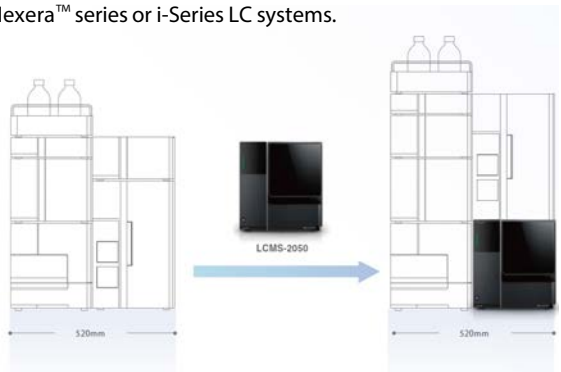

Illustration of Integrating an LCMS™-2050 Unit into a Nexera™ Series HPLC System

Table 1 Analytical Conditions

[HPLC conditions] (Nexera X3)	
Column:	Shim-pack Scepter™ C18-120*1 (100 mm × 2.1 mm I.D., 1.9 µm)
Mobile Phases:	Methanol
Flowrate:	0.4 mL/min
Column Temp.:	40 °C
Injection Volume:	1 µL
Detection:	PDA 210-500 nm
[MS conditions] (LCMS-2050)	
Ionization:	ESI/APCI (DUIS), Positive and Negative mode
Mode:	Scan (*m/z* 250-800)
Interface Voltage:	+3.0 kV / -2.0 kV
Corona Needle Voltage:	+3.0 kV / -2.0 kV
DL/QA Voltage:	+20 V / -20 V
Nebulizing Gas Flow:	2.0 L/min
Drying Gas Flow:	5.0 L/min
Heating Gas Flow:	7.0 L/min
DL Temperature:	200 °C

*1 P/N: 227-31012-05

■ Results and Discussion

Each compound was analyzed in the scan mode based on the analytical conditions listed in Table 1. Mass spectra of the main peaks detected are shown in Fig. 3.

Characteristic ions were detected from each compound. The results confirmed that the LCMS-2050 is able to easily ionize even low polarity compounds such as polycyclic aromatics.

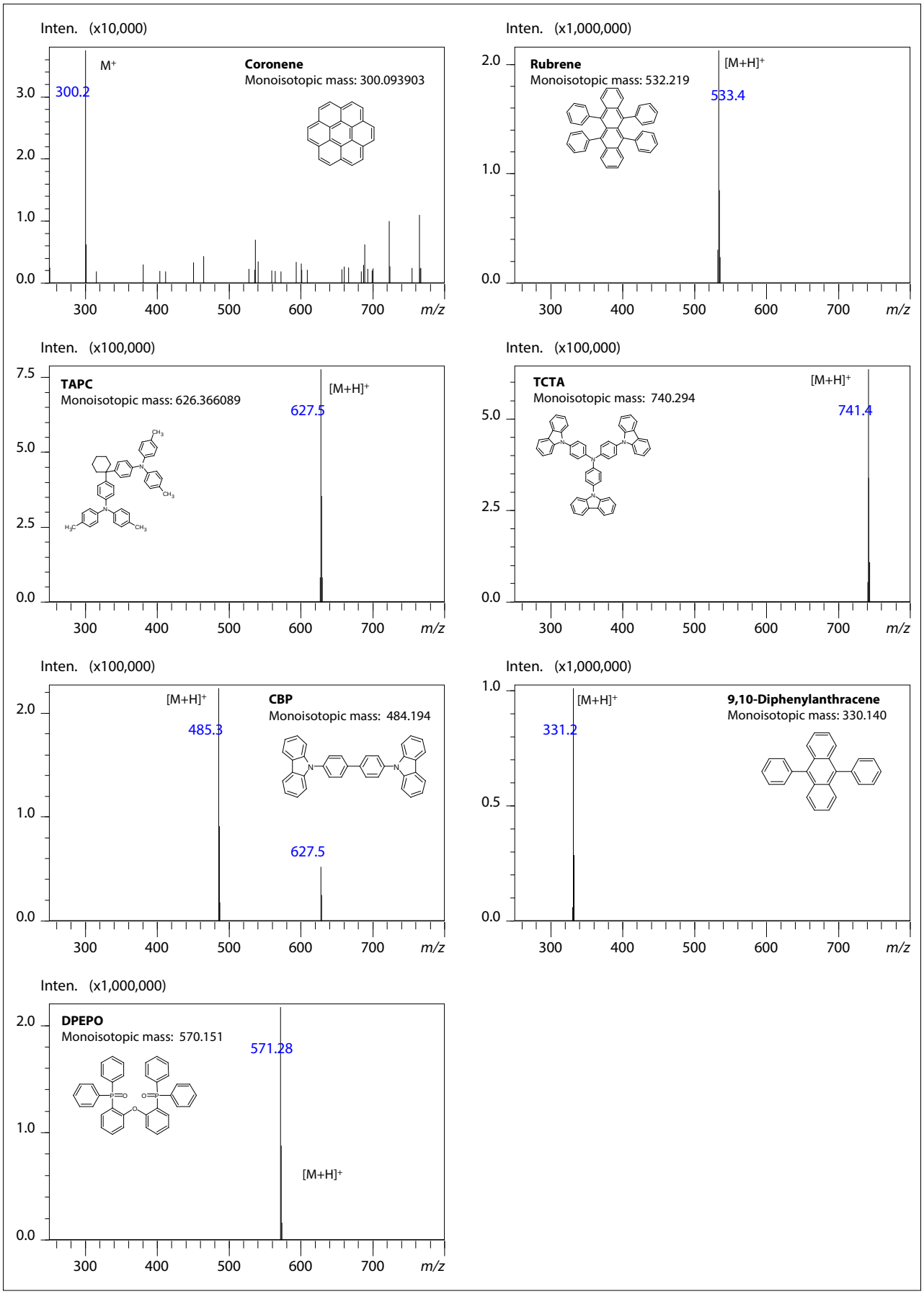

Fig. 3 MS Spectra of Main Peaks in the PDA Chromatograms

■ Analyzing Impurities in Organic EL Materials

In addition to checking for synthesized compounds, the development of organic EL materials also requires checking for and controlling impurities. Organic EL materials can include a variety of impurities, such as unreacted compounds and decomposition products from synthesis or a mixture of compounds from tools and reagents used during manufacturing processes. Because fluorescent or phosphorescent compounds are used in organic EL materials to emit light, it is especially important to reduce the quantities of impurity compounds that are fluorescent or phosphorescent compounds and absorb light in the ultraviolet to visible light range.

Compounds that absorb light in the UV to visible range can be effectively analyzed with a PDA detector, but to identify the impurities it is essential to acquire a UV spectrum and confirm their retention times by analyzing standard samples. However, that requires the time and trouble of performing a separate analysis and identifying impurities based on PDA detector data alone is difficult because standard samples are not available for some impurities. In combination with a mass spectrometer for obtaining mass information about peaks detected in the UV chromatogram, the system can be used to check for known impurities or easily predict unknown impurities. In this example, the molecular weights of impurities detected in TAPC were confirmed from among compounds with confirmed molecular weights.

■ Results and Discussion

Peaks were confirmed for ultra-trace impurities in TAPC. The UV chromatogram for TAPC is shown in Fig. 4. Characteristic ions detected from the impurity peaks are indicated in Table 3. MS chromatograms for those ions are shown in Fig. 5. The mass chromatogram peak retention times are consistent with PDA results and m/z values were confirmed for all major impurity peaks detected.

■ Conclusion

This article describes analyzing organic EL materials and corresponding impurities using a high performance liquid chromatograph and single quadrupole mass spectrometer.

Impurities that absorb light in the UV to visible light range were selectively detected by using a PDA detector to check UV chromatograms at any specific wavelength. Molecular weights of impurities were confirmable using a single quadrupole mass spectrometer to check the mass spectrum for peaks detected in the UV chromatogram.

Using a single quadrupole mass spectrometer in combination with a PDA detector, it was easy to confirm organic EL material synthesis and analyze impurities.

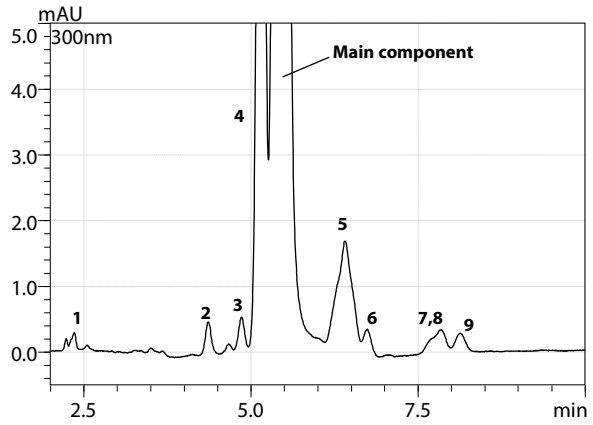

Fig. 4 PDA Chromatogram

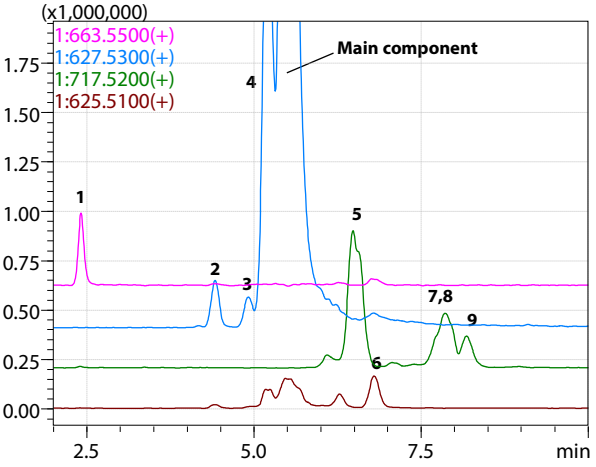

Fig. 5 MS Chromatograms for Impurity Peaks

Table 3 Characteristic Ions Detected from Impurity Peaks

Peak No.	Retention time (min)	*m/z*
1	2.41	663.6
2	4.43	627.5
3	4.90	627.5
4	5.21	627.5
5	6.46	717.5
6	6.79	625.5
7	7.76	717.5
8	7.86	717.5
9	8.21	717.5

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