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Analysis of Organic Light Emitting Diode Materials Using a Novel Single Quadrupole LC/MS

<u>Vikki Johnson¹</u>; Miho Kawashima²; Yuka Fujito¹; Yusuke Inohana² ¹Shimadzu Scientific Instruments, Columbia, MD²Shimadzu Corporation, Kyoto, Japan

1. Overview

This poster utilized LC/MS for the analysis of OLED materials. In this example, a single quadrupole was used to check the synthesis of organic EL materials and to predict the molecular weights of impurities they contain.

2. Introduction

Organic Light Emitting Diode (OLED) materials are used in the LED display industry. For the development of high-quality displays, quality control and evaluation of impurities must be controlled and evaluated. HPLC-UV is primarily used for impurity analysis, and LC/MS is used for molecular weight confirmation of synthesis products.

Most OLED materials are classified as polycyclic aromatics. Due to their low polarity characteristic, APCI is often used in LC/MS analysis. This study shows an example of impurity analysis of OLED materials in combination with molecular weight confirmation using PDA and MS detection. A single quadrupole equipped with a heated dual ion source (DUIS[™]) with both ESI and APCI capability was employed (Fig. 1).



Figure 1. Nexera[™] HPLC and fully integrated LCMS[™]-2050 Single Quadrupole Mass Spectrometer

3. Experimental Methods

Seven commercially available organic electro-luminescence materials (Coronene, TAPC, CBP, DPEPO, Rubrene, TCTA, and 9,10-Diphenylanthracene) were used as compounds (Fig. 2). Each compound was dissolved in a tetrahydrofuran/methanol solution to a concentration of 0.1mg/mL.



Figure 2. Structure of OLED Analytes

4. Analytical Method

A newly developed single quadrupole mass spectrometer (LCMS-2050, Shimadzu) coupled to an HPLC (Nexera X3, Shimadzu) equipped with a pump, autosampler, column oven, and PDA detector was used for analysis (Fig. 1).

The LCMS-2050 employs DUIS (Dual Ion Source) that combines ESI and APCI functions. This technology enables the measurement of a wide range of compounds in a single analysis. Low-polarity analytes that cannot be detected by ESI can be detected with high sensitivity when measured with DUIS. Fig. 3 show a schematic representation of the ionization portion that comes standard on the LCMS-2050.

LC and MS analytical conditions are summarized in Table 1 and Table 2.



Figure 3. Ionization structure of LCMS-2050 (left). Schematic diagram of the ionization section (ESI/APCI) of LCMS-2050 (right)

Table 1. HPLC Conditions (Nexera X3) Shim-pack Scepter™ C18-Column 100 mm ×2.1 mm I.D., 1.9 Column Dimensions Mobile Phases Methanol 0.4mL/min Flow rate Injection Volume PDA, 210-500nm Detection

Table 2. MS Conditions (LCMS-2050)

Ionization	ESI/APCI (DUIS), (+) and (-) 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

high voltage application

5. Results

5-1. Mass Spectra of Main Peaks

is already predicted, then accurate mass measurements are not necessarily required.



Figure 4. MS Spectra of Main Peaks in the PDS Chromatograms

5-2. Analyzing Impurities in Organic EL Materials

The development of organic EL materials also requires checking for and controlling impurities, such as unreacted compounds and decomposition products from synthesis or a mixture of compounds from tools and regents used from manufacturing processes. Peaks were confirmed for ultra-trace impurities in TAPC. The UV and MS chromatograms are shown in Fig. 5 and Fig. 6, respectively. Characteristic ions detected from the impurity peaks are shown in Table 3.



6. Conclusion

This study suggests that ionization by the DUIS mode, which incorporates both ESI and APCI capabilities simultaneously, is effective in detecting low-polarity compounds as well as compounds with a wide range of chemical properties whose chemical nature is unknown. 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.



Mass spectra of the main peaks in the PDA chromatogram are shown in Fig. 4. If the composition of the intended substance being measured

	Table 3. lor	ns Detected from Imp	ourity Pea	k
ain component	Peak No.	Retention time (min)	m/z	
	1	2.41	663.6	
	2	4.43	627.5	
	3	4.90	627.5	
7,8	4	5.21	627.5	
9	5	6.46	717.5	
	6	6.79	625.5	
min	7	7.76	717.5	
natogram	8	7.86	717.5	
	9	8.21	717.5	