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Application News

Semi-Preparative Supercritical Fluid Chromatograph – Nexera™ UC Prep

Purification of Organic Light-Emitting Diode Materials

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User Benefits

- Analytical SFC system enables seamless small-scale purification.
- Dedicated SFC columns assist effortless scale up from analytical scale to preparative scale.
- Nexera UC Prep provides good peak shape even in large volume injection.

■ Introduction

An organic light-emitting diode (OLED) is made from luminescent materials and classified as polycyclic aromatic compounds. Effective analytical and preparative methods are required to identify the structure of the compounds and to analyze impurities in high-quality OLED materials. This article introduces Nexera UC analytical fraction system and Nexera UC Prep for small-scale and large-scale preparative purification of OLED materials, respectively.

■ OLED materials analysis by SFC

Published application Note (01-00135-en) introduces a novel SFC method for quantitative analysis of OLED materials (Fig. 1) using Nexera UC and Method Scouting Solution software. Table 1 and Fig. 2 show the analytical conditions and the acquired chromatogram of three OLED materials by SFC system.

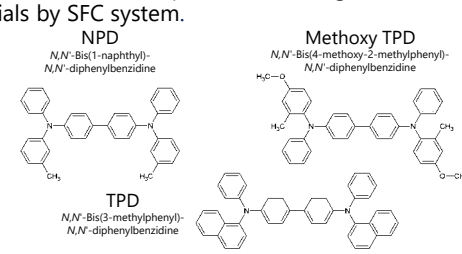

Fig. 1 Structure of OLED materials (NPD, Methoxy-TPD, TPD)

Table 1 Analytical conditions (SFC)

Column	: Shim-pack™ UC-Diol II *1 (250 mm × 4.6 mm I.D., 5 µm)
Mobile phase	: A: CO_2 B: Acetonitrile
Flow rate	: 3 mL/min
Time program	: B conc. 2 % (0 min-1 min) →40 % (6 min-8 min) →2 % (8.01 min-10 min)
Column temp.	: 40 °C
Injection vol.	: 5 µL in tetrahydrofuran (containing 250 mg/L for each compound)
Vial	: SHIMADZU LabTotal™ for LC 1.5 mL, Glass *2
BPR setting	: 10 MPa
Detection	: PDA 350 nm (reference 400 nm)

*1 P/N: 228-34937-92 *2 P/N: 227-34001-01

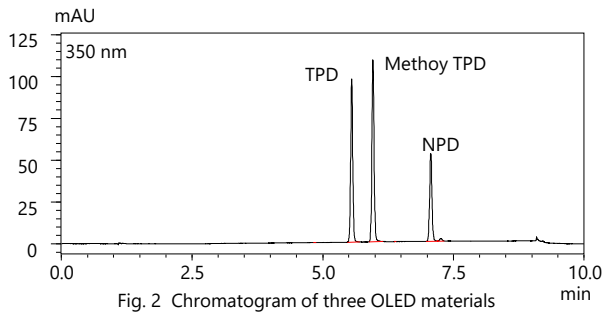

Fig. 2 Chromatogram of three OLED materials

■ SFC analytical fraction system

Nexera UC, supercritical fluid chromatograph, has capability to couple with FRC-40 SF, which is fraction collector, and performs as an analytical scale preparative system (Fig. 3). It facilitates smooth transition from method development to sample purification.

Fig. 3 Nexera UC™ analytical fraction system

■ Small-scale purification and purity check of OLED materials

Fig. 4 shows a preparative chromatogram of three OLED materials by analytical method displayed in Table 1. Sample solution was prepared at 5 g/L concentration. Nexera UC analytical fraction system can prevent contamination by purge function, which washes both the dispense nozzle and the gas-liquid separator after fractionation (Fig. 4). Nexera UC allows re-injection of the collected fractions and subsequently measures the fraction purity. Results are displayed on Fig. 5 and Table 2. Nexera UC analytical fraction system achieved high-performance purification.

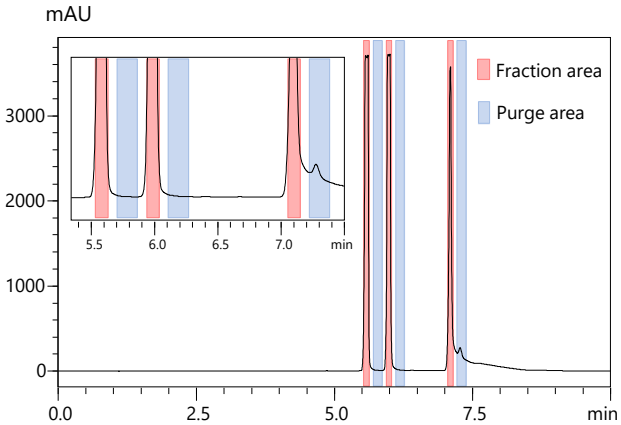

Fig. 4 Preparative chromatogram of SFC (Nexera UC analytical fraction system)

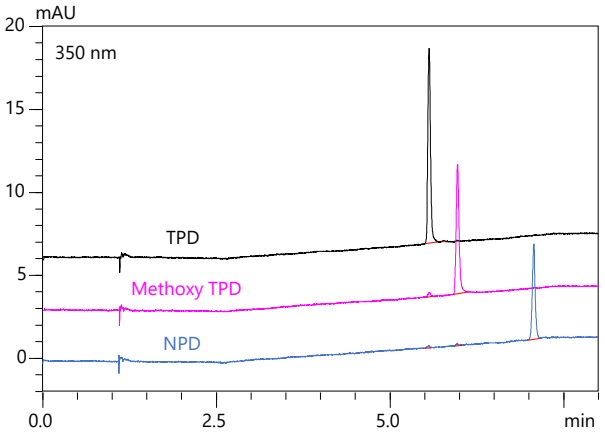

Fig. 5 Re-analysis chromatograms of each fraction

Table 2 Purity check results
(Area normalization method, UV 350 nm)

	Area %
TPD	99.7
Methoxy TPD	97.2
NPD	96.7

■ Upscaling the preparative method from small-scale to large-scale

Nexera UC analytical fraction system purified approximately 25 µg of OLED materials at one injection. Moreover, we developed a large-scale preparative method to purify higher amount of crude sample. Upscaling from small-scale to large-scale can be achieved easily by Shim-pack UC columns. Shim-pack UC columns affords a simple upscaling from small-scale to large scale by changing only the flow rate setting because both scale columns contain identical packing materials (See Technical Report C190-E246).

In this article, a 20 mm I.D. column and Nexera UC Prep system were utilized. By using method described in Table 3, Nexera UC Prep achieved 40 times more loading amount in one injection than a small-scale preparation (Fig. 6).

Table 3 Analytical conditions (preparative SFC)

Column	: Shim-pack UC Diol II *3 (250 mm × 20 mmI.D., 5 µm)
Mobile Phase	: A: CO_2 B: acetonitrile
Flow rate	: 60 mL/min
Make up Flow	: 5 mL/min (tetrahydrofuran)
Time program	: B conc. 2 %(0-1 min)→ 40 % (6-8 min) →0 %(8.01-10 min)
Column temp.	: 40 °C
Injection vol.	: 200 µL in tetrahydrofuran (containing 5 g/L for each compound)
Vial	: 10 mL screw vial *4
BPR Parameter	: 10 MPa
Detection	: PDA 350 nm (reference 400 nm) Prep cell

*3 P/N: 227-32606-04 *4 P/N: 18 09 1306-1

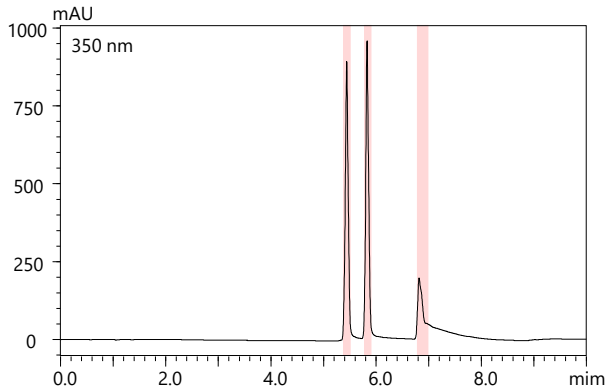

Fig.6 Chromatogram of preparative SFC (Nexera UC Prep)

■ Reduction of sample solvent effect

Large volume loading is important to achieve a large amount of purification at one time. However, peak separation and peak shape can be deteriorated when using high volume injection. Nexera UC Prep has an on-column dilution function to dilute sample solvent during injection. This function reduces sample solvent effect and maintains peak shape better in large injection volume. Experiments were carried out by changing injection volume from 200 to 1800 µL. Good peak separation and shape were observed by using 1800 µL injection (Fig. 7).

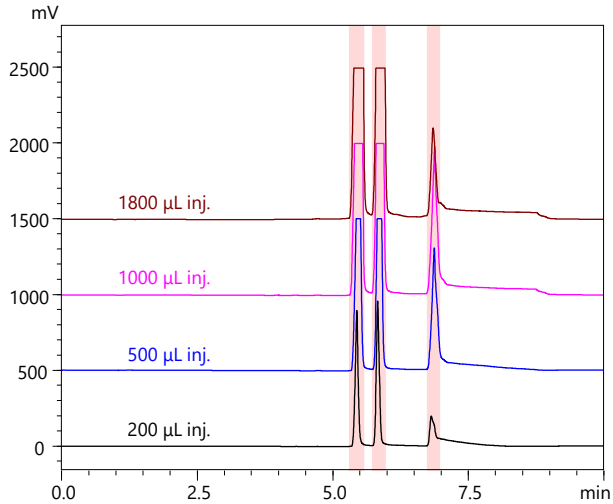

Fig.7 Preparative chromatogram with large injection volumes

■ Conclusion

This article introduces small-scale and large-scale preparative methods for OLED materials. Nexera UC and Nexera UC prep provide comprehensive workflow for new compounds research including method development, quantitative analysis, small-scale purification, and large-scale purification. Both systems are expected to be resourceful for research of OLED materials from the R&D stage to the quality control stage in electronic industries.

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