Excellence in Science

Supercritical Fluid Chromatograph – Nexera™ UC

Development of Novel Analytical Method for Organic Light-Emitting Diode Materials

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User Benefits

- Analytical SFC provides shorter analysis time and reduces the possibility of target compound degradation.
- SFC utilizes lower organic solvent consumption than conventional HPLC method.

Introduction

An organic light-emitting diode (OLED) is made from luminescent materials and classified as polycyclic aromatics compounds. Effective analytical and preparative method are required to identify the structure of the compounds and to analyze impurities in high-quality OLED materials. In this article, we established a new analytical method for analysis of OLED materials using supercritical fluid chromatography (SFC).

Organic Light-Emitting Diode Materials

OLED is widely used to create digital display in smartphones, televisions, and PC monitors due to its luminescent properties. High luminescent efficiency and stability of the OLED materials are essential to improve the performance of OLED displays.

OLED materials are conventionally analyzed by HPLC with high concentration of organic solvent condition because of their low water solubility. Table 1 and Fig. 1 show analytical conditions and chromatogram of three OLED materials by HPLC.

Table 1 Analytical conditions (HPLC)

Column	: Shim-pack™ VP-ODS *1 (250 mm × 4.6 mm I.D., 5 µm)
Mobile phase	: A: Acetonitrile/water= 9:1 B: Tetrahydrofuran
Flow rate	: 3 mL/min
Time program	: B conc. 5 % (0 min)→ 30 % (10 min) → 30 % (10.01-15 min)
Column temp.	: 40 ℃
Injection Vol.	: 10 µL
Vial	: SHIMADZU LabTotal™ for LC 1.5 mL, Glass *2
Detection	: PDA 350 nm

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

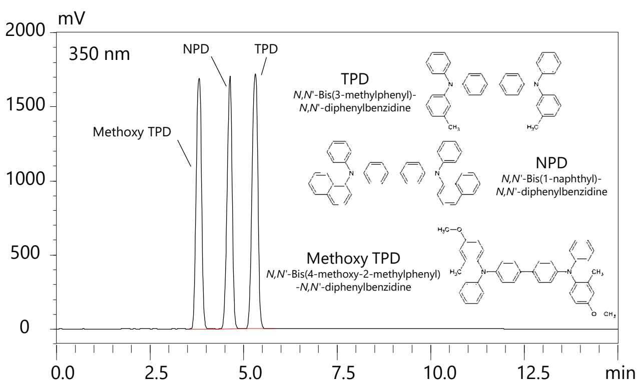

Fig. 1 Chromatogram of OLED materials by HPLC (TPD, NPD, Methoxy TPD)

Features of SFC

SFC is one of the chromatography with supercritical fluid (i.e., supercritical carbon dioxide) as mobile phase. The low cost of carbon dioxide can significantly trim the total running cost of HPLC analysis. Since carbon dioxide is inert, degradation risk of target compounds is lower than that of conventional HPLC approach. In this study, SFC method was developed for analysis of OLED materials.

Method Scouting Solution

Method Scouting Solution, dedicated software for method scouting, can create multiple methods for optimization using different analytical conditions.

A total of 24 analytical conditions using six columns and four organic solvents as modifiers were investigated (Fig. 2). Table 2 and Fig. 3 display analytical conditions and chromatograms of OLED materials.

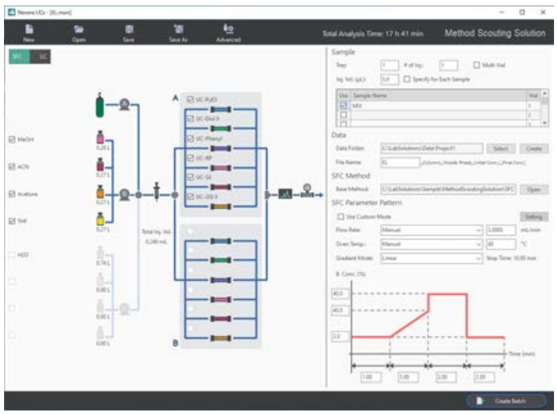

Fig. 2 User interface of Method Scouting Solution

Table 2 Analytical conditions (SFC)

Column	: Shim-pack UC-PyE 3*, Diol II 4*, Phenyl 5*, RP 6*, Sil 7*, GIS II 8* (250 mm × 4.6 mm I.D., 5 µm)
Mobile Phase	: A: CO_2 B: Methanol or acetonitrile or acetone or tetrahydrofuran
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 ℃
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)

3* P/N: 227-32604-02 4* P/N: 227-32606-02 5* P/N: 227-30427-02
6* P/N: 227-30403-02 7* P/N: 227-30415-02 8* P/N: 227-30407-02

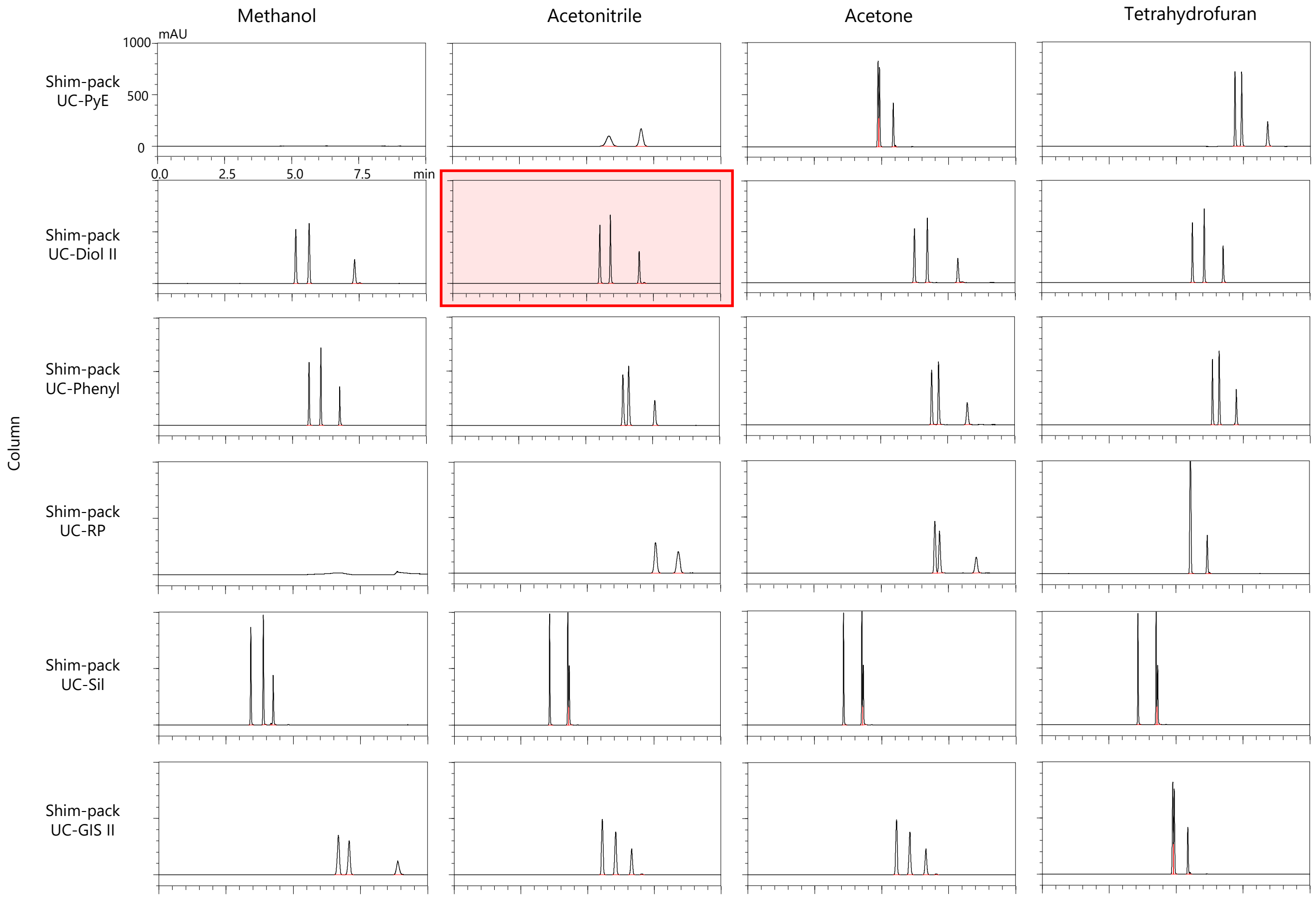

Fig. 3 Chromatograms of OLED materials using Method Scouting Solution
The highlighted one was the best, and the method was used for quantitative analysis.

■ Quantitative Analysis by SFC

Analytical method using a Shim-pack Diol II column and acetonitrile as modifier was chosen based on the method scouting results for quantitative analysis of OLED materials (Fig. 4). Calibration curves of three OLED materials were prepared ranging from 1 mg/L to 250 mg/L. Repeatability, linearity of the calibration curves, and LOD(limit of detection) are summarized in Table 3.

Fig. 5 shows the comparison of solvent consumption between conventional HPLC and SFC method. Taken together, these results demonstrate the capability of SFC method for quantitative analysis of OLED compounds with much lower solvent consumption.

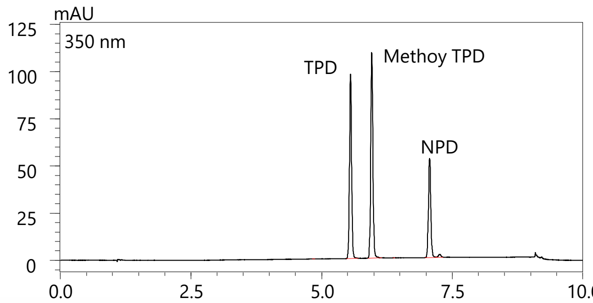

Fig. 4 Chromatogram of three OLED materials

Table 3. Repeatability, linearity, and LOD of OLED materials

	TPD	Methoxy TPD	NPD
%RSD (peak area, n=6 at 50 mg/L)	0.65	0.66	0.63
Linearity of calibration curve (R^2) (1, 2, 5, 10, 25, 50, 250 mg/L)	0.9999	0.9999	0.9999
LOD (mg/L, calculated values)	0.13	0.16	0.28

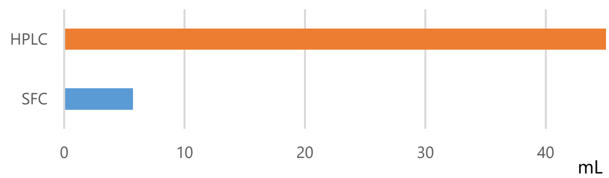

Fig. 5 Comparison of organic solvent consumption per one analysis

■ Conclusion

A novel SFC method was developed for quantitative analysis of OLED materials using Nexera UC and Method Scouting Solution. Method Scouting Solution provides a seamless method optimization. SFC significantly reduces organic solvents consumptions than conventional HPLC. Additionally, SFC is proven to be effective for preparative purification workflow (application note 01-00136-en).

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