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Application News

Supercritical Fluid Chromatograph Nexera™ UC

Preparative Purification of Aroma Components Using a Supercritical Fluid chromatograph

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User Benefits

- Supercritical fluid chromatography (SFC) enables preparative purification of volatile compounds such as aroma components.
- The analytical fraction system enables a smooth transition from analysis to preparative purification.
- The unique "LotusStream" gas-liquid separator enables recovery of target components with high purity and high yield.

■ Introduction

Many aroma components in foods, beverages, personal care products, and essential oils are volatile compounds with low molecular weights. Many of them are chiral compounds with enantiomers. Generally, enantiomers have different pharmacological effects in pharmaceuticals. Similarly, since enantiomers of aroma components can smell different, their ratio can affect the quality and intensity of fragrances. Thus, enantiomeric separation and preparative purification are required in the development process to understand such differences between enantiomers.

Conventionally, volatile compounds, such as aroma components, are separated and fractionated by gas chromatography (GC). GC offers high resolution, but the small loading volume per injection and the long analysis time can be problems. Preparative purification by supercritical fluid chromatography (SFC) enables faster loading and analysis of the same volume as conventional liquid chromatography (LC). Furthermore, carbon dioxide used as the eluent for SFC is vaporized at room temperature and pressure, leaving only a small amount of organic solvent in the separated fraction. That enables easy concentration of samples in fractions.

This article describes an example of preparative purification of linalool in a lavender essential oil using the Nexera UC analytical fraction collection system.

Table 1 Analytical Conditions

System:	Nexera UC
Column:	CHIRALPAK® IG-3 (100 mm × 3.0 mm I.D., 3 µm)
Mobile Phase:	CO_2 / Methanol = 95:5
Flowrate:	1.5 mL/min
Column Temp.:	40 °C
Injection Volume:	5 µL in methanol
Vial:	SHIMADZU LabTotal for LC 1.5 mL, glass*1
BPR Pressure:	15 MPa
Detection:	UV 220 nm (PDA with a high-pressure flow cell)

*1 P/N :227-34001-01

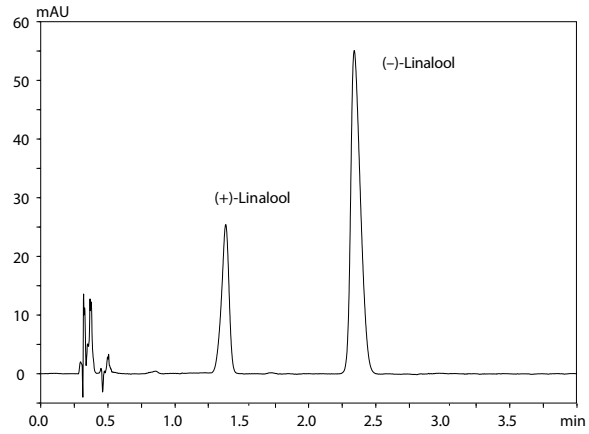

Fig. 2 Chromatogram of Linalool

■ Separation of Linalool Enantiomers

Application News 01-00435 described a workflow for establishing analytical conditions for analyzing linalool enantiomers using the Nexera UC chiral screening system. The optimized analytical conditions for enantiomeric separation are shown in Table 1 and the chromatogram in Fig. 2.

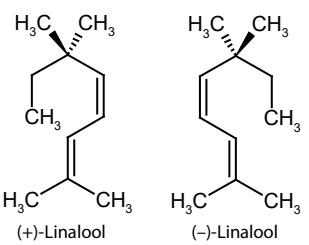

Fig. 1 Chemical Structures of Linalool Enantiomers

■ Nexera UC Analytical Fraction System

The Nexera UC supercritical fluid chromatograph can be used for preparative purification by connecting an FRC-40 SF fraction collector (Fig. 3). The Nexera UC chiral screening system can also be upgraded to an analytical fraction system. That enables a smooth transition to preparative purification.

Fig. 3 Nexera™ UC Analytical Fraction Collection System

■ LotusStream Gas-Liquid Separator

In fractionation using SFC, diffusion of eluent from the column caused by CO_2 expansion as it transitions from a supercritical state to a gas state (about 500 times the SF volume) contributed to poor recovery rates. The unique gas-liquid separator can reduce diffusion and contamination of samples. Samples can be collected in small containers, such as 1.5 mL vials or 96-well microplates.

Fig. 4 LotusStream Gas-Liquid Separator

■ Recovery Rate of Linalool

The analytical fraction collection system was used to purify a sample solution with about 20 g/L of commercial linalool by preparative purification (Fig. 5) using the same preparative conditions as indicated in Table 1. Both (+)-linalool and (–)-linalool enantiomers were recovered in high yields over 97 % (Table 2). In this example, approximately 100 µg of linalool was injected per analysis, with more than 97 µg successfully recovered as fractions.

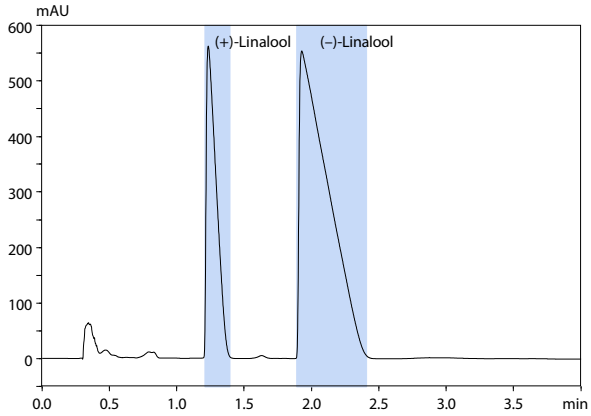

Fig. 5 Preparative Chromatogram of Linalool

Table 2 Recovery Rate of Each Fraction

	Recovery rate (%)
(+)-Linalool	97.6
(–)-Linalool	99.3

■ Fractionation of Linalool in Lavender Essential Oil

Lavender essential oil was diluted with methanol by a factor of 10 and filtered through a 0.2 µm membrane filter. The preparative conditions are the same as those shown in Table 1 and used in the chromatogram in Fig. 6.

The collected fractions of (+)-linalool and (–)-linalool enantiomers were reanalyzed to confirm purity (Fig. 7). The results showed that the purity of both fractions exceeded 99 %, which confirmed that high-purity preparative purification was achieved (Table 3).

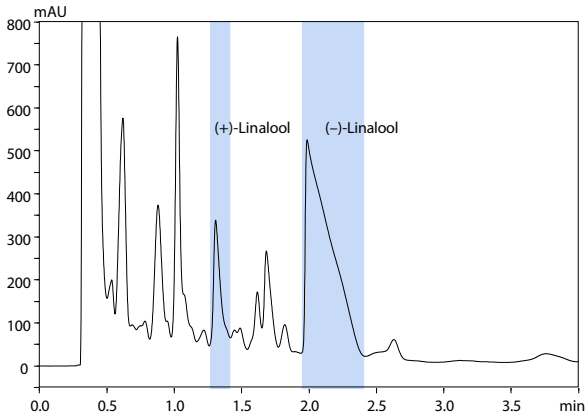

Fig. 6 Preparative Chromatogram of Lavender Essential Oil

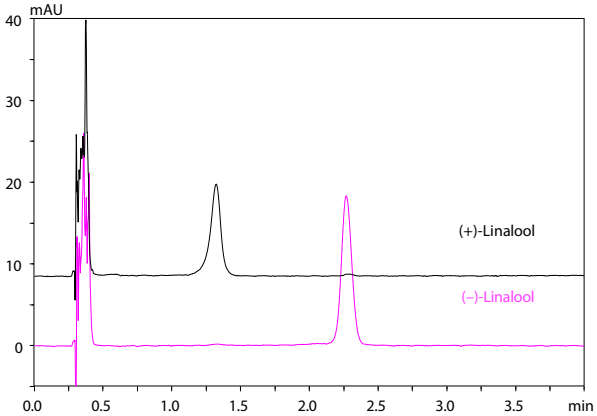

Fig. 7 Chromatograms from Reanalysis of Collected Fractions

Table 3 Purity of Each Fraction (Peak Detection: 0.5 -4.0 min)

	Area (%)
(+)-Linalool	100 %
(–)-Linalool	99.4 %

■ Conclusion

This article described examples of preparative purification of aroma components in a lavender essential oil. Aroma components were recovered with high yield and purity.

Separation and preparative purification of chiral compounds is important in development processes. The Nexera UC analytical fraction collection system, which can be used for applications from analysis to preparative purification, can be expected to improve operating efficiency.

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