SHIMADZU
Excellence in Science

Application News

Supercritical Fluid Chromatograph Nexera™ UC

Enantiomeric Separation of Flavor and Aroma Components Using a Supercritical Fluid Chromatograph

Yusuke Masuda

User Benefits

- Enantiomers of aroma components can be separated in several minutes.
- Analytical conditions for new compounds can be easily configured with the software for assisting analytical method development.
- Using carbon dioxide as a mobile phase, which is less expensive than organic solvents, can be expected to reduce operating costs.

■ Introduction

Many aroma components in foods, beverages, personal care products, and essential oils have small molecular weights that volatilize easily. 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, the separation of enantiomers is important in the process of developing fragrances.

This article describes examples of enantiomeric separation of aroma components using the Nexera UC chiral screening system.

■ Nexera UC Chiral Screening System

The Nexera UC chiral screening system can automatically switch between up to 12 columns and blends of up to 4 modifiers during analysis, to greatly reduce the overall workload. With the software for analytical method development*1, columns and modifiers can be managed in a database and various analytical conditions can be applied by simply selecting them in the graphical user interface (GUI) (Fig. 1). In addition, the software can be used to easily determine optimal analytical conditions based on screening results.

*1 For this article, Method Scouting Solution was used as the software for analytical method development. Labsolutions™ MD does not support SFC.

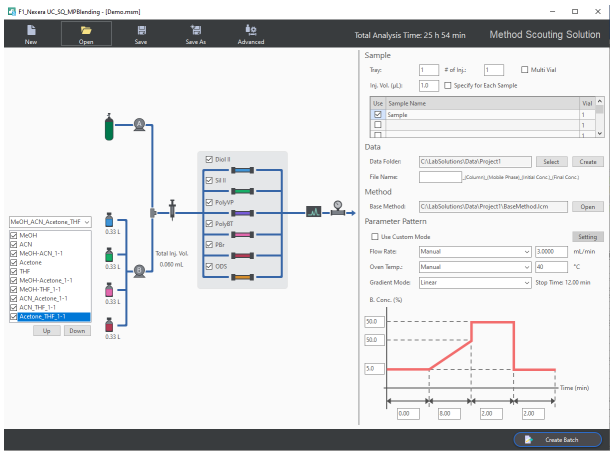


Fig. 1 GUI of the Software for Analytical Method Development

■ Screening of Analytical Conditions for Linalool Enantiomers

Linalool has two enantiomers. The chemical structures of those enantiomers are shown in Fig. 2.

Of the enantiomers of linalool, (+)-linalool has a sweet fruity aroma and is abundant in orange oil. In contrast, (-)-linalool has a woody lavender-like aroma and is abundant in lavender and lemon oils. Both enantiomers have soothing floral aromas that are used in various applications, such as cosmetics, perfumes, and foods.

In this article, a total of 48 analytical conditions using 4 modifiers and 12 columns were screened. The screening conditions are shown in Table 1. Some chromatograms obtained by the screening are shown in Fig. 3.

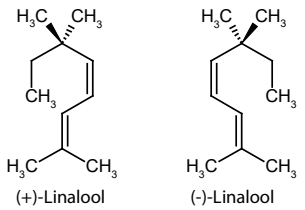


Fig. 2 Chemical Structures of Linalool Enantiomers

Table 1 Modifier/Column Scouting Conditions

System:	Nexera UC chiral screening system
Column:	CHIRALPAK® IA-3 (100 mm × 3.0 mm I.D., 3 µm) CHIRALPAK® IB-3 (100 mm × 3.0 mm I.D., 3 µm) CHIRALPAK® IC-3 (100 mm × 3.0 mm I.D., 3 µm) CHIRALPAK® ID-3 (100 mm × 3.0 mm I.D., 3 µm) CHIRALPAK® IE-3 (100 mm × 3.0 mm I.D., 3 µm) CHIRALPAK® IF-3 (100 mm × 3.0 mm I.D., 3 µm) CHIRALPAK® IG-3 (100 mm × 3.0 mm I.D., 3 µm) CHIRALPAK® AD-3 (100 mm × 3.0 mm I.D., 3 µm) CHIRALPAK® AS-3 (100 mm × 3.0 mm I.D., 3 µm) CHIRALPAK® AY-3 (100 mm × 3.0 mm I.D., 3 µm) CHIRALCEL® OD-3 (100 mm × 3.0 mm I.D., 3 µm) CHIRALCEL® OJ-3 (100 mm × 3.0 mm I.D., 3 µm)
Mobile Phase A:	CO_2
Mobile Phase B:	Methanol Acetonitrile Ethanol 2-Propanpl
Flowrate:	1.5 mL/min
Time Program:	B. conc. 5 % (0 min) → 30 % (5.01-6.0 min) → 5 % (6.01-8.0 min)
Column Temp.:	40 °C
Injection Volume:	2 µL in methanol
Vial:	SHIMADZU LabTotal™ for LC 1.5 mL, glass*2
BPR Pressure:	15 MPa
Detection:	UV 220 nm (PDA with a high-pressure flow cell)

*2 P/N :227-34001-01

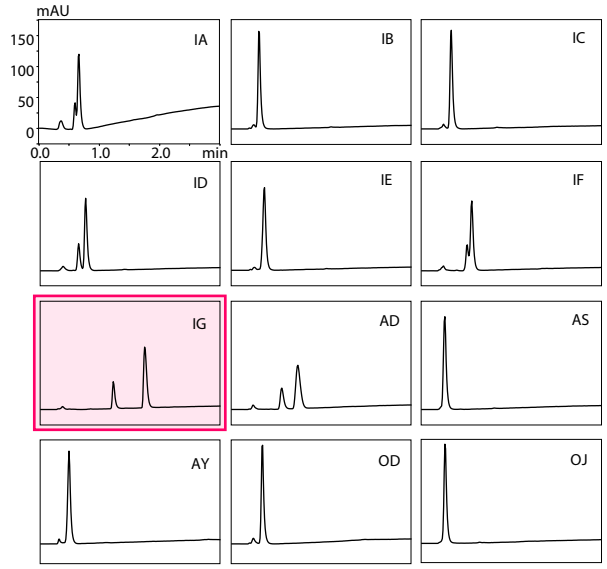


Fig. 3 Chromatograms Obtained by Scouting (Modifier : MeOH)*3

*3 These scales are identical.

■ Optimization of Analytical Conditions for Linalool Enantiomers

The screening results showed that the best separation was achieved using MeOH as the modifier and the CHIRALPAK® IG-3 as the column, as indicated with a red box in Fig. 3.

In addition, analytical conditions were optimized by including isocratic conditions in conditions used for screening. As a result, linalool enantiomers were separated in only 2.5 min. The optimized analytical conditions are listed in Table 2 and the chromatogram in Fig. 4.

Table 2 Optimized Analytical Conditions

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
BPR Pressure:	15 MPa
Detection:	UV 220 nm (PDA with a high-pressure flow cell)

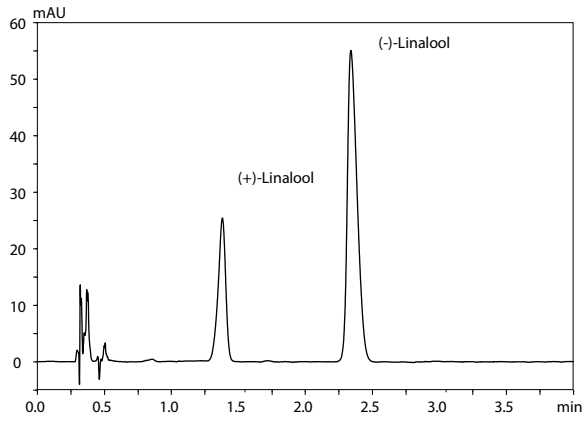


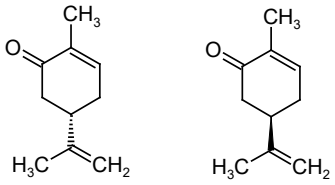
Fig. 4 Chromatogram of Linalool under Optimized Conditions

■ Separation of Carbone Enantiomers

Carvone has two enantiomers. The chemical structures of the enantiomers are shown in Fig. 5.

Carvone is the main ingredient in spearmint, which has a refreshing aroma and is used in foods and toothpaste.

As with linalool, the analytical conditions were optimized based on the screening results. Carvone enantiomers were separated in only 1.7 min. The optimized analytical conditions are shown in Table 3 and the chromatogram in Fig. 6.



(+)-Carvone (-)-Carvone

Fig. 5 Chemical Structures of Carvone Enantiomers

Table 3 Analytical Conditions for Carvone

Column:	CHIRALPAK® IG-3 (100 mm × 3.0 mm I.D., 3 µm)
Mobile Phase:	CO_2 / Acetonitrile = 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
BPR Pressure:	15 MPa
Detection:	UV 230 nm (PDA with a high-pressure flow cell)

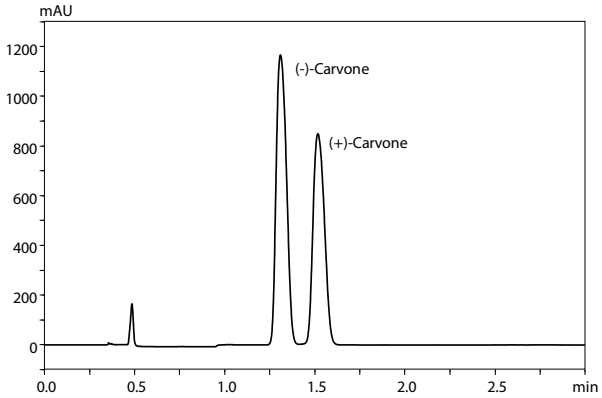


Fig. 6 Chromatogram of Carvone

■ Conclusion

This article described examples of enantiomeric separation of aroma components using the Nexera UC chiral screening system.

The system was able to separate the enantiomers of aroma components in several minutes. The software for analytical method development can reduce the overall workload. Furthermore, CO_2 is less expensive than many organic solvents used in HPLC and waste disposal is also cheaper. SFC can be applied not only for analysis but also for preparative purification.

01-00435-EN First Edition: Feb. 2023

SHIMADZU

Shimadzu Corporation

www.shimadzu.com/an/

› Please fill out the survey

Related Products Some products may be updated to newer models.

› Nexera UC

Supercritical Fluid Extraction / Supercritical Flu···

Related Solutions

› Evidence-based

› Price Inquiry

› Product Inquiry

› Technical Service / Support Inquiry

› Other Inquiry