

Separation of citral isomers from lemongrass oil

Pure C-850: Purification with flash chromatography followed by a prep HPLC isolation of citral isomers

Lemongrass is cultivated around the world. It's mainly grown for its oils, which have a number of uses including for vitamin A, perfumes, insect spray, cosmetics, perfumes, food and drinks. Lemongrass is also enjoyed as a tea throughout the world. Lemongrass oil contains several monoterpenes, with citral being the major component, present at levels between 65-85%. Citral (3,7-dimethyl-2,6-octadienal) is the name given to a natural mixture of two isomeric acyclic monoterpene aldehydes: geranial (trans-citral, citral A) and neral (cis-citral, citral B). In addition to citral, the lemongrass oil consists of small quantities of geraniol, geranylacetate, and monoterpene olefins, such as myrcene [1].

1. Introduction

The aim of this application note was to isolate geranial and neral from lemongrass oil (Figure 1). The isolation of citral was performed on a normal phase flash column. The citral fraction was concentrated to 5 mL with a Rotavapor® R-300. Subsequently, the 5 mL of concentrated solution was injected and run on a prep HPLC column to separate the isomers. A PureC-850 system, shown in Figure 2, was used for the flash and the prep HPLC separations.

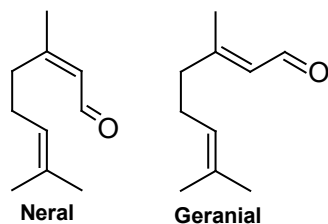


Figure 1: The two isomers of citral.

Table 1: Chromatography conditions for the flash and prep HPLC column.

	Flash Mode	Prep Mode
Column	FlashPure Silica cartridge 4g	BUCHI PrepChrom Silica 250 x 21.2mm, 10 µm
Solvent A	Petroleum ether	n-Hexane
Solvent B	20% Ethyl acetate in Petroleum ether	20% Ethyl acetate in n-Hexane
Flow rate	12 mL/min	15 mL/min
Gradient	1 % to 18 % B in 10 min; 18 % to 100 % B in 0.5 min; 100 % B for 3 min	10 % to 20 % B in 15 min; 20 % B for 30 min
Detector	ELSD and UV 210 nm, 240 nm, 254 nm	ELSD and UV 254 nm, 265 nm, 280 nm
Sample load	1 mL lemongrass oil	5 mL concentrated solution from prepurification



Figure 2: Pure C-850 Purification System.

2. Results

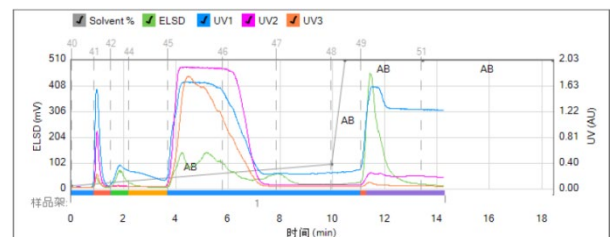


Figure 3: Chromatogram obtained during flash purification of lemongrass oil.

Figure 3 shows the chromatogram obtained by flash chromatography. The citral fraction is separated, it eluted between 4 and 7 minutes. From the ELSD signal, it can be seen that the peak is split, suggesting that it could be possible to isolate the citral isomers.

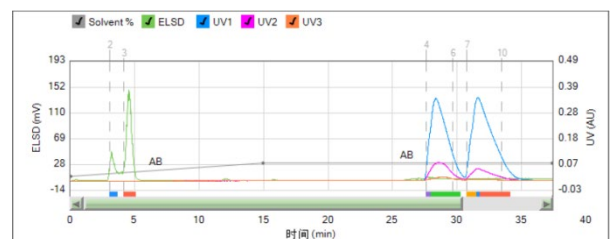


Figure 4: Chromatogram obtained in prep HPLC purification of the citral fraction.

Figure 4 shows the result of prep HPLC purification of the isolated citral fractions. The two isomers geranial and neral eluted after 27 minutes and were separated by a prep HPLC column on the same system. Due to the smaller particle size of the silica particles in the prep HPLC, a much greater separation and purification performance can be obtained.

3. Conclusion

Citral can easily be separated from a crude lemongrass oil mixture by flash chromatography on the Pure C-850 system. A second purification step using the prep HPLC capabilities of the same system enables the separation of two citral isomers. This application demonstrates the combination of flash chromatography and prep HPLC in a single system for the isolation of pure compounds from natural products.

4. References

[1] Govindarajan, V.S.; Sathyanarayana, M.N.; Capsicum. Production, technology, chemistry, and quality. Part V. Impact on physiology, pharmacology, nutrition, and metabolism; structure, pungency, pain, and desensitization sequences. Crit. Rev. Food Sci. Nutr., 29, p. 435-475,1991.