The Reporter

Reprinted from Volume 14, No. 4, 1995

T295034

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Analyzing Citrus Oil Using SPB™-5 and SPB-Octyl Capillary Columns

L. Sidisky

Using a single-injection dual-column system provides advantages for analysts studying complex essential citrus oils. Serving as confirmational phases, the SPB-5 and SPB-Octyl capillary columns yield unique fingerprint patterns, allowing for easier identification and quantitation of a number of key compounds.

Flavor and fragrance analyses are challenging to chromato-graphers because of the complexity of the various samples. These samples are comprised of a wide variety of chemical compounds, including terpenes, ketones, aldehydes, alcohols, esters, and hydrocarbons. Capillary gas chromatography provides characterizational fingerprinting of the essential oils and monitoring of samples for adulteration.

Distilled lime oil and cold-pressed lemon oil, important samples in the flavoring industry, are routinely characterized and evaluated for possible adulteration. Distilled lime oil, a by-product of lime juice processing, is recovered by distilling the acid liquors from the crushing of the lime fruit. Cold-pressed lemon oil is obtained by mechanical expression of the oil from the peel of the fresh fruit without using heat. The oil and juice from the fruit are separated during processing to yield the lemon oil.

A number of columns have been examined by flavor chemists for analyzing citrus oils (1). Carbowax® 20M-type columns were once widely used. However, these columns failed to perform a number of key separations. Many analysts switched to using nonpolar phases in the early 1980s. Nonpolar SPB-5 (5% diphenyl/95% dimethylpolysiloxane) and SPB-1 (100% dimethylpolysiloxane) capillary columns traditionally have been used to analyze citrus essential oils. Both columns provide excellent resolution of the citrus oil components, and are used routinely in quality control analyses.

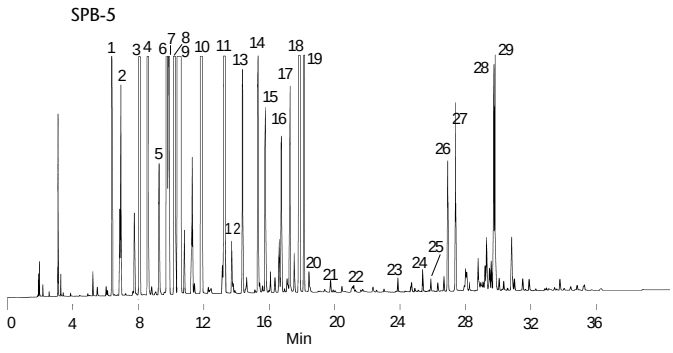
The newly developed SPB-Octyl capillary column, a very nonpolar bonded phase, provides unique resolution of citrus oils not possible on traditional nonpolar columns such as SPB-1 and SPB-5. The SPB-Octyl column is a 50% octyl/50% methyl polysiloxane phase whose polarity is lower than that of traditional nonpolar phases, and approaches that of the most nonpolar phase, squalane. The unique polarity of the SPB-Octyl phase has been shown to benefit analysts in the petrochemical and environmental fields (2).

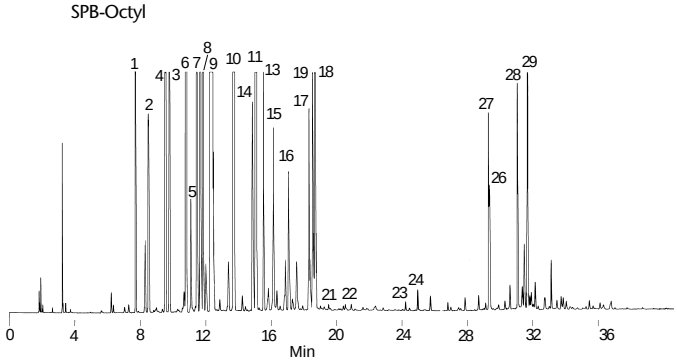
We used an SPB-5 column and an SPB-Octyl column (both 30m x 0.25mm ID, 0.25µm film) in the analysis of citrus oils. Both columns were installed into a single injection capillary split port using a two-hole ferrule. Each column was installed in its own FID detector. Neat injections of the two essential oils were made into the injection port, and the sample was split appropriately onto each

Figure A. Distilled Lime Oil

Column: 30m x 0.25mm ID, 0.25µm film
Cat. Nos.: **24034** (SPB-5)
24218-U (SPB-Octyl)
Oven: 75°C (hold 8 min) to 200°C at 4°C/min (hold 5 min)
Carrier: helium, 25cm/sec, set at 110°C, splitter vent flow 90cc/min
Det.: FID, 260°C
Inj.: 250°C, split 100:1

1. α-Pinene
2. Camphene
3. β-Pinene
4. Myrcene
5. α-Phellandrene
6. 1,4-Cineole
7. α-Terpinene
8. p-Cymene
9. d-Limonene
10. γ-Terpinene
11. Terpinolene
12. Linalool
13. α-Fenchyl alcohol
14. Terpinen-1-ol
15. β-Terpineol
16. Borneol
17. Terpinen-4-ol
18. α-Terpineol
19. γ-Terpineol
20. Decanal
21. Neral
22. Geranial
23. Neral acetate
24. Geranyl acetate
25. Dodecanal
26. β-Caryophyllene
27. trans-α-Bergamotene
28. trans-α-Farnesene
29. β-Bisabolene





795-0529, 0530

column. Since the dimensions of the two columns were identical, both columns received equal sample amounts.

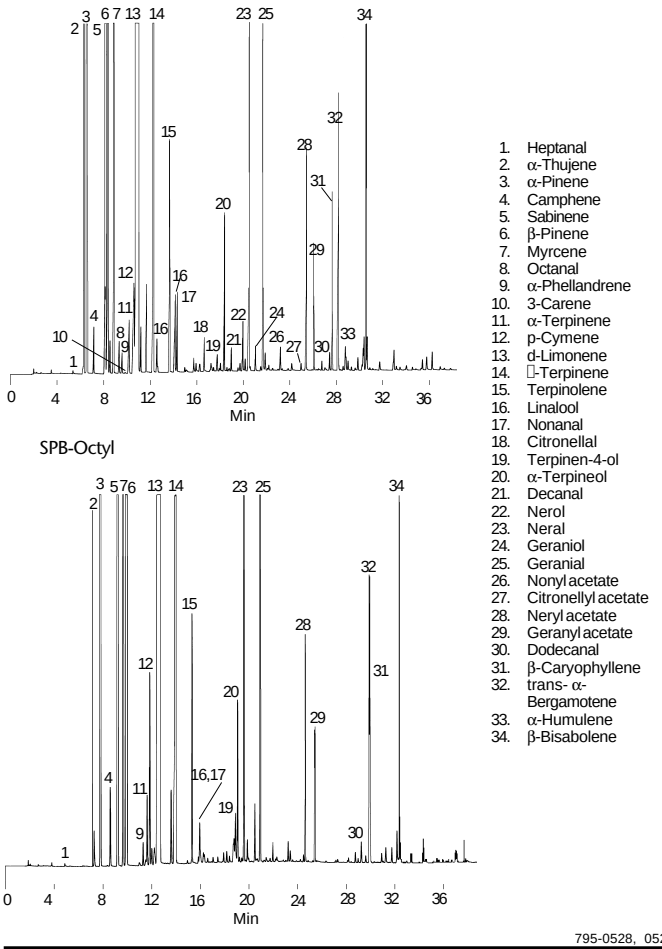
An examination of the fingerprint pattern for distilled lime oil (Figure A) reveals differences throughout the profiles. A shift in the elution pattern of two major quantitative components of lime oil — γ-terpineol and α-terpineol — is observed. Note also the reversal in elution of two other pairs — β-pinene and myrcene, and 1,4-cineole and α-phellandrene. The SPB-Octyl column resolved a large unknown compound after p-cymene that was not observed using the SPB-5 column. Additionally, the SPB-Octyl column provided a better separation of trans-α-farnesene from β-bisabolene.

The major quantitative components of cold-pressed lemon oil — limonene, α-pinene, and γ-terpinene — were well resolved on both columns (Figure B). Similar elution shifts of the common components in lemon and lime oil are demonstrated in this figure. The fingerprint patterns for both columns show a number of differences which add to the complementary nature of the single-injection dual-column analysis.

Both the SPB-Octyl column and the SPB-5 column are useful for the analysis of citrus oils. Each column gives a characteristic fingerprint profile for the oils and provides improved resolution over the other column for a number of key compounds. This allows for easier identification and quantitation of complex essential oil samples.

Figure B. Cold-Pressed Lemon Oil

Conditions: same as in Figure A.



795-0528, 0527

Ordering Information:

Description	Cat. No.
Capillary Columns, 30m x 0.25mm ID, 0.25µm film	
SPB-5	**24034**
SPB-Octyl	**24218-U**
Supeltex™ M-2A Ferrules, 2-hole, 0.4mm ID	
Pk. of 5	**22467**

References

1. The Supelco Reporter, Vol. 2, No. 5, 1983.
2. The Supelco Reporter, Vol. 14, No. 1, 1995.

Acknowledgement

We are grateful to Mr. Bill Faas of the A.M. Todd Company for providing samples and assisting with peak identification.