Rapid Analysis of Spearmint Oil by GC-TOFMS

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Key Words: GC-TOFMS, Flavor, Minor Components

1. Introduction

Spearmint oil is widely used as a flavoring additive for a variety of manufactured foods. Minor variations in spearmint oils from different sources can significantly alter the flavor and ultimately the quality of a product. Consequently, careful analysis of the oil components is important to ensure the manufacture of consistent products over time.

Essential oil analyses are commonly performed using gas chromatography (GC) with flame ionization detection (FID). However, this approach can require lengthy data acquisition of an hour or more to allow for complete chromatographic resolution of the individual components.

Alternatively, spearmint oil analyses using fast GC techniques combined with the LECO Pegasus II Time of Flight Mass Spectrometer (TOFMS) are finished in one-tenth the time. Fast spectral acquisition rates (up to 500 spectra per second) allow for unique and automated spectral deconvolution of overlapping chromatographic peaks. Even minor components coeluting with the major analytes in an oil are detected. The resulting mass spectra coupled with retention index information are used to identify individual components.

2. Experimental Conditions

General GC-TOFMS conditions were established for a variety of essential oil extracts. These conditions, outlined below, were used without further optimization to analyze a sample of spearmint oil[1], resulting in a total acquisition time of 2.3 minutes. Automated data processing and library searching against both the NIST and Terpene Essential Oil[2] mass spectral databases resulted in 61 identified components.

Table 1. GC and MS Conditions for a 2.3 Minute Analysis of Spearmint Oil.

Detector:	LECO Corporation Pegasus II Time-of-Flight Mass Spectrometer
Transfer Line:	300°C
Source:	200°C
Acquisition Rate:	30 spectra/sec
Stored Mass Range:	35 to 400u
GC:	Hewlett Packard® 6890*
Column:	DB-5 4 m x 0.1 mm ID, 0.1 µm phase film
Oven:	40°C for 0.5 min., then to 280°C at 75°C/min., hold for 1 min.
Injector:	290°C
Carrier Gas:	Helium, 2.0 ml/min. constant flow
Sample:	No preparation required. 0.1 µL split (200:1) injection.

**HP6890 GC is equipped with fast oven temperature ramp capabilities and a high pressure EPC module.*

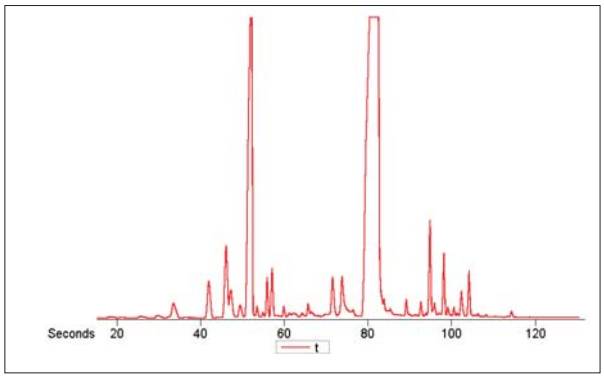

Figure 1. Total Ion Chromatogram (TIC) for a 2.3 Minute Analysis of Spearmint Oil.

3. Results

The data file was processed immediately after acquisition using automated Peak Finding, spectral deconvolution and library searching of the analytes. The unique Peak Finding algorithm locates even minor components coeluting with the primary component of spearmint oil. Figure 2 below shows mass chromatograms for three peaks on the tailing edge of the Carvone peak. The peaks are located and their spectra are automatically deconvoluted by the Peak Find and Deconvolution algorithms. The resulting deconvoluted spectra match the library spectra with similarities greater than 700 for even the most minor component.

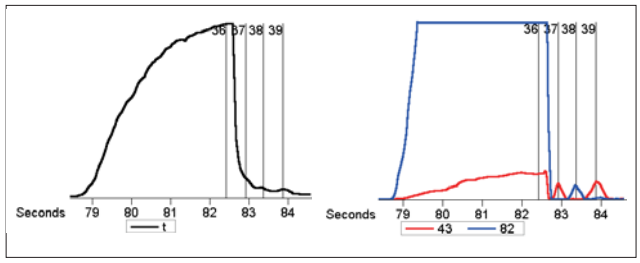

Figure 2. Automated Peak Finding Locates Three Minor Components on the Tailing Edge of Primary Component Carvone in Spearmint Oil. The Total Ion Chromatogram (TIC) on the left and selected ion chromatogram on the right show the peak positions.

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Table 2. Compound Name, Retention Time (RT), Forward and Reverse Spectral Similarity Indices, and Library Hit Number for Spearmint Oil Analysis.

Peak	Name	R.T. (sec.)	Similarity	Reverse	Hit
1	Butanal, 3-methyl-	5.98	838	840	1
2	Furan, 2-ethyl-	7.08	893	897	1
3	1-Butanol, 2-methyl-	8.68	773	793	1
4	Butanoic acid, 2-methyl-, methyl ester	10.93	839	902	1
5	2-Hexenal	18.23	757	883	1
6	3-Hexen-1-ol	19.03	884	904	1
7	2-Hexen-1-ol, (E)-	20.83	720	801	1
8	n-Hexanol*	21.33	767	851	1
9	Furan, 2,5-diethyltetrahydro-	25.63	744	859	1
10	α-Pinene	33.53	933	936	1
11	Camphene	36.38	853	909	1
12	Thuja-2,4(10)-diene*	37.78	595	825	1
13	β-Pinene	41.98	924	925	1
14	1-Octen-3-ol*	44.18	739	776	2
15	β-Myrcene	46.13	899	899	1
16	3-Octanol	47.23	858	897	1
17	Octanal	48.08	756	841	1
18	Limonene	52.38	842	857	1
19	Benzene acetaldehyde*	53.13	837	866	1
20	cis-β-Ocimene*	53.53	899	925	1
21	trans-β-Ocimene*	54.93	896	929	1
22	γ-Terpinene	55.93	876	899	1
23	cis-Sabinenehydrate	57.13	853	931	4
24	Terpinolene	59.93	901	922	1
25	trans-Sabinenehydrate	61.28	833	913	4
26	Linalool*	62.13	751	871	1
27	cis-p-Menth-2-en-1-ol	64.28	820	830	3
28	Octanol acetate*	65.73	795	846	1
29	cis-Verbenol	67.38	733	747	1
30	Menthone	68.33	830	841	2
31	Pinocarvone*	69.18	788	832	1

Peak	Name	R.T. (sec.)	Similarity	Reverse	Hit
32	4-Terpineol	71.58	874	874	1
33	Dihydrocarveol	76.53	875	887	1
34	trans-Piperitol	78.13	542	843	1
35	Carvone	79.73	905	926	1
36	Carvone oxide, trans-	82.93	676	683	1
37	p-Mentha-1,8-dien-3-one	83.33	789	832	1
38	Carvone oxide, cis-	83.78	863	873	1
39	Dihydrocarvyl acetate	89.18	885	907	1
40	Piperitenone*	89.88	748	889	1
41	Carvyl acetate	90.08	716	735	3
42	Eugenol	91.83	823	848	2
43	cis-Carvyl Acetate	92.73	890	892	1
44	α-Copaene*	93.93	864	907	1
45	β-Bourbonene	94.88	886	892	1
46	β-Elemene*	95.63	881	926	1
47	cis-Jasmone	95.88	854	862	1
48	α-trans-Bergamotene*	96.93	804	868	1
49	Caryophyllene	98.18	880	880	2
50	γ-Muurolene*	99.13	842	886	6
51	α-Humulene*	101.38	862	904	1
52	β-Farnesene	102.38	871	872	1
53	Germacrene D	102.63	867	899	1
54	cis-γ-Cadinene*	104.23	814	849	8
55	Butanoic acid, 3-methyl-, 2-phenylethyl ester	104.83	759	816	3
56	Bicyclogermacrene*	105.68	872	920	1
57	Germacrene A*	106.43	857	903	5
58	α-Farnesene	107.28	796	816	1
59	δ-Cadinene*	108.28	826	885	1
60	Caryophyllene oxide	113.43	701	871	1
61	Viridiflorol	114.33	856	858	2

Analyte found in the Essential Oil MS library. All other analytes were identified in the NIST MS Database.

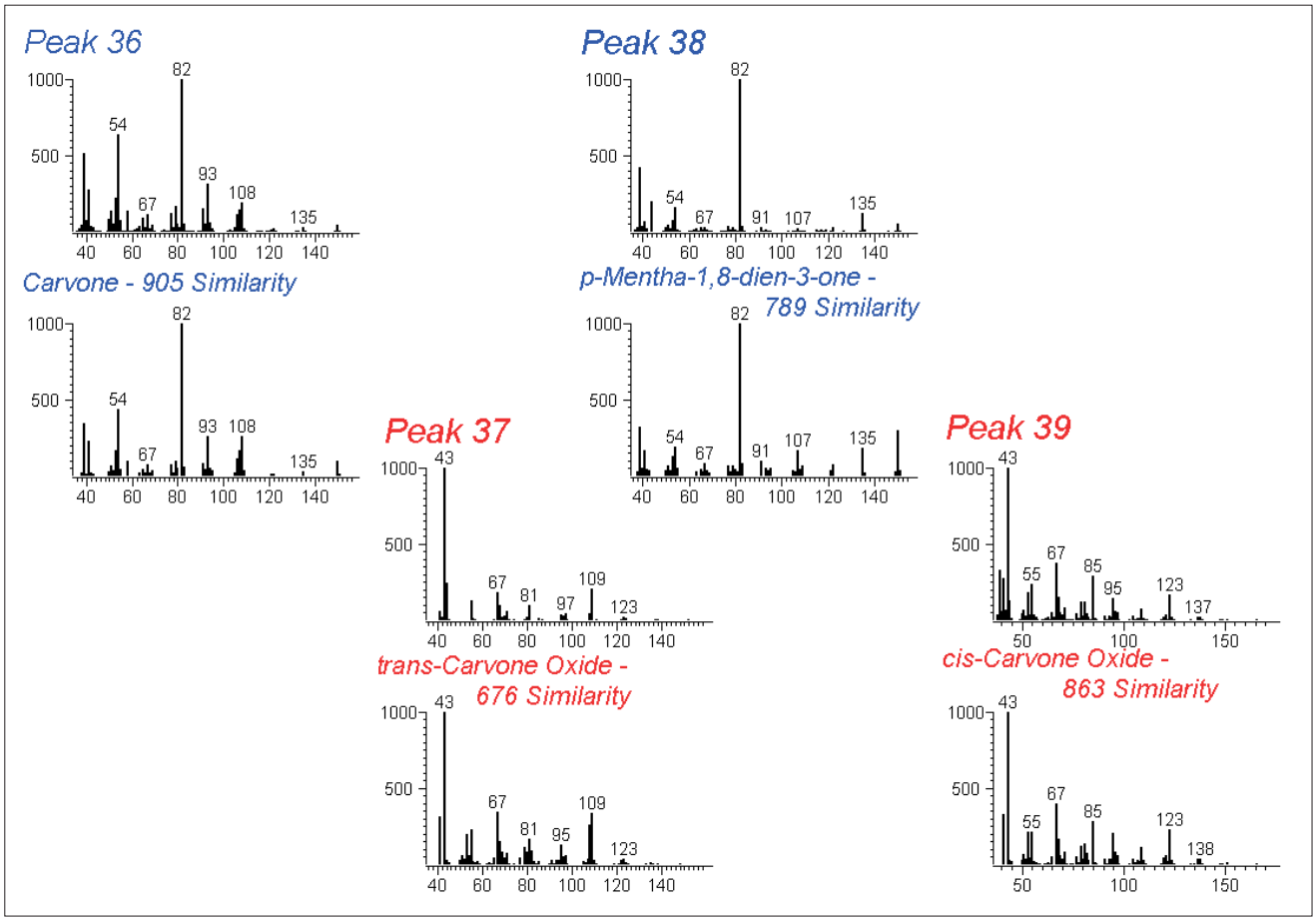

Figure 3. Automated Spectral Deconvolution Allows the Spectra of Overlapping Chromatographic Peaks to be Determined Individually Even When the Peaks Are Minor Components Coeluting With Major Components in the Oil. The top spectrum for each peak is the Pegasus II acquired spectrum. The bottom spectrum is the library spectrum.

4. Conclusions

General conditions for rapid flavor and fragrance analyses were used to analyze a sample of spearmint oil in just 2.3 minutes. The Peak Find and Spectral Deconvolution algorithms unique to the Pegasus II locate even minor components in the oil and significantly reduce data processing time. The entire analysis is complete in less than one-tenth the time required using traditional approaches.

5. Acknowledgements

[1]Thanks to Kathleen K. Webb and A.M. Todd Company for providing a sample of spearmint oil.

[2]Robert P. Adams. *Identification of Essential Oil Components by Gas Chromatography/Mass Spectroscopy.* Allured Publishing Corporation, Carol Stream, IL. 1995.

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