Automated Comparison of Scotch and Midwest Spearmint Oils using GC-TOFMS

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Key Words: GC-TOFMS

1. Introduction

Spearmint oils are widely used to flavor a variety of manufactured foods. Oils from different regions can vary significantly in composition and thus affect the flavors of the foods in which they are used. Two spearmint oil samples, one Scotch and one Midwest, were analyzed using fast GC techniques and the fast data acquisition of the LECO Pegasus® II Time of Flight Mass Spectrometer (TOFMS). The samples were then processed and compared using algorithms unique to the Pegasus II.

The Pegasus II automated data processing algorithms include peak finding and spectral deconvolution. Peak finding effectively locates the positions of all peaks in a sample including multiple components in complex coelutions. Deconvolution then resolves the mixed mass spectra of the coelution into accurate individual mass spectra for each analyte, including the accurate distribution of signal from masses shared by several components in the coelution. Once the peaks are located and the spectra determined for each sample, a comparison is performed based upon peak positions and spectral similarities. Components present in one sample and not in another are identified, as well as components present in both samples, but at widely varying concentrations (based upon a user-defined threshold).

2. Experimental Conditions

Scotch and Midwest spearmint oil samples were analyzed. Automated data processing was performed and the resulting mass spectra were searched against both the National Institute of Standards and Technology (NIST) 1998 Mass Spectral Database and the Terpene Essential Oil Library.[1]

Detector:
LECO Corporation Pegasus II Time-of-Flight Mass Spectrometer
Transfer Line: 300°C
Source: 200°C
Acquisition Rate: 30 spectra/second
Stored Mass Range: 35 to 400 u
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 minute, then to 280°C at 75°C/minute, hold for 1 minute
Injector: Split/Splitless at 290°C
Carrier Gas:
Helium, 2.0 ml/minute constant flow
Sample:
No preparation required. 0.1 µL split (150:1) injection

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

3. Results

Based upon the Total Ion Chromatogram overlay (Figure 1), the samples appear quite similar in composition. The comparison algorithm is able to detect and identify components present in one oil and not in the other in addition to analytes present in both oils, but outside of a user-defined tolerance level for concentration variation. The results from these analyses are in Tables 1, 2 and 3. An extracted ion profile of m/z 83 is plotted for both oils in Figure 2 This profile highlights two analytes present in the Midwest oil and not the Scotch as well as analytes present in both oil, but outside of the concentration tolerance set for the analysis.

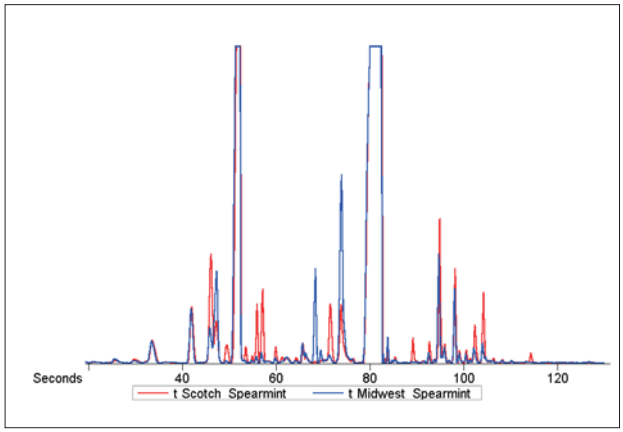

Figure 1. Total Ion Chromatogram (TIC) Overlay of Scotch and Midwest Spearmint Oils.

Table 1. Spearmint Oil Difference Peak Table. Analytes found in Scotch spearmint and not in Midwest spearmint.

Name	R.T.
7-Octen-4-ol	44.18
trans-Piperitol	78.13
2-Cyclohexen-1-one, 3-methyl-6-(1-methylethenyl)-	83.38
Globulol	114.33

Table 2. Spearmint Oil Difference Peak Table. Analytes found in Midwest spearmint and not in Scotch spearmint.

Name	R.T.
2-Methoxyethyl benzene	58.87
Benzene, 4-ethenyl-1,2-dimethyl-	60.15
Isomenthone	69.54
Bicyclo[3.1.1]heptan-3-one, 2,6,6-trimethyl-, (1α,2α,5α)-	70.45
8-α-Acetoxyelemol	110.19
cis-3-Hexenyl Phenyl Acetate	118.07

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Table 3. Spearmint 'Out of Tolerance' Peak Table. Analytes found in both spearmint oils, but at concentrations outside of a user-defined tolerance (150% used here). The Match Number indicates the spectral similarity between the spectra in the two spearmint oils. The Relative Concentration is the concentration of analytes in the Scotch sample relative to those in the Midwest sample. Concentrations in the Midwest sample are set to 100% by default. RT is the Retention Time.

Name	R.T.	Match	Relative Concentration
Butanal, 3-methyl-	5.78	773	157.2 %
Butanal, 2-methyl-	5.93	851	19.52 %
Furan, 2-ethyl-	7.08	783	150.02 %
1-Pentanol	8.58	673	236.9 %
Thuja-2,4(10)-diene	37.78	720	40.7 %
Benzaldehyde	38.33	686	39.9 %
3-Octanone	44.98	666	13.09 %
β-Myrcene	46.13	961	304.99 %
3-Octanol	47.23	918	42.19 %
α-Terpinene	49.48	917	810.4 %
p-Cymene	50.48	712	273.9 %
cis-β-Ocimene	53.53	920	828.3 %
trans-β-Ocimene	54.93	925	263.5 %
γ-Terpinene	55.93	954	1089.8 %
2-Cyclohexen-1-ol, 1-methyl-4-(1-methylethyl)-, cis-	57.13	967	797.6 %
1-Octanol	58.58	726	206.4 %
Terpinolene	59.98	924	313.4 %
trans-Sabinene hydrate	61.28	849	500.54 %
3-Nonanol	61.88	633	49.5 %
Nonanal	62.48	825	44.4 %
7-Hydroxy-6-methyl-oct-3-enoic acid	64.13	585	459.0 %
Cyclohexanone, 5-methyl-2-(1-methylethyl)-	68.38	848	1.7 %
Terpinen-4-ol	71.58	918	867.1 %
cis-Dihydro carvone	73.88	937	25.4 %
neo-iso-Dihydro Carveol	75.93	778	166.6 %
Carvone	82.43	956	4.1 %
cis-Carvone oxide	82.93	828	257.2 %
Edulan I, dihydro-	85.43	870	201.0 %
Dihydrocarvyl acetate	89.18	866	2607.0 %
Piperitenone	89.88	781	158.0 %
10,13-Octadecadiynoic acid, methyl ester	91.28	432	665.2 %
Piperitone oxide	92.58	398	14.4 %
cis-Carvyl Acetate	92.73	937	281.3 %
β-Elemene	95.63	850	173.1 %
1,6,10-Dodecatriene, 7,11-dimethyl-3-methylene-, (Z)-	99.78	715	158.5 %
α-Humulene	101.38	905	164.2 %
1,6,10-Dodecatriene, 7,11-dimethyl-3-methylene-, (E)-	102.38	961	268.7 %
γ-Muurolene	102.63	932	188.6 %
Germacrene D	104.23	972	364.0 %
Phenethyl isovalerate	104.83	876	22.8 %
Germacrene A	106.43	915	174.1 %

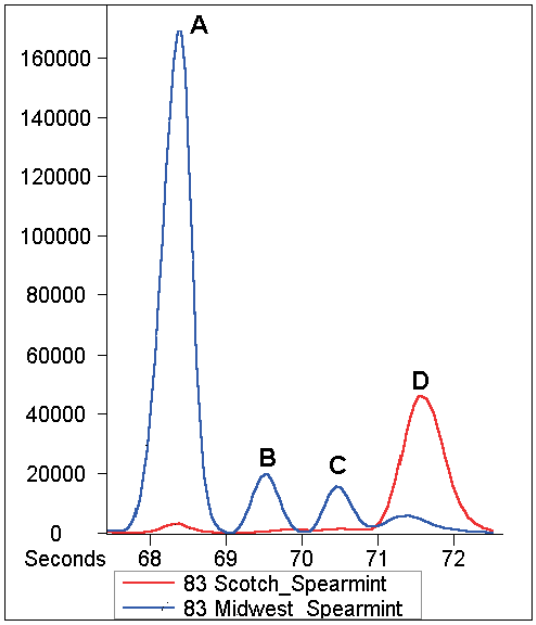

Figure 2. Extracted Ion Profile Chromatogram Overlay of m/z 83 in Scotch and Midwest Spearmint Oils.

Peak A: Cyclohexanone, 5-methyl-2-(1-methylethyl)-, Scotch oil contains 1.7% of that in Midwest oil. Peak B: Isomenthone, Not Found in Scotch oil. Peak C: Bicyclo[3.1.1]heptan-3-one, 2,6,6-trimethyl-, (1α,2α,5α), Not Found in Scotch oil. Peak D: Terpinen-4-ol, present in Scotch oil at 8.7 times greater than that in Midwest oil.

4. Conclusions

The combination of unique and automated peak find, spectral deconvolution, and comparison algorithms with the Pegasus II allow for the rapid identification of even minor differences between two spearmint oils taken from different regions.

5. References

[1]The Terpene Library contains mass spectra of essential oil components and DB-5 retention indices compiled by Robert P. Adams, Baylor University Plant Biotechnology Center.

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