Rapid Analysis of Lavender Oil by GC-TOFMS: Automated Location of Major and Minor Components

LECO Corporation; Saint Joseph, Michigan USA

Key Words: GC-TOFMS

1. Introduction

Lavender oil is collected from the steam distillation of the lavendula officinalis flower and it is used a classic scent in perfume as well as hair and skin care products. The careful characterization of lavender oil components is important to ensure that commercial products containing the oil are manufactured consistently over time.

Gas chromatography (GC) with flame ionization detection (FID) is commonly used to characterize essentail oils such as lavender oil. However, these analyses are lengthy requiring 45 minutes or more to allow complete chromatographic resolution of the individual components for identification.

Analyses of lavender oil with fast GC techniques and the LECO Pegasus® II Time-of-Flight Mass Spectrometer (TOFMS) are complete in less than 3 minutes. Fast spectral acquisition rates (up to 500 spectra/second) allow for unique Deconvolution of overlapping chromatographic peaks. The fast data acquisition, automated data processing, and automated reporting allow for at least a ten-fold decrease in sample analysis and processing time.

2. Experimental Conditions

General GC and 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 lavender oil. The total acquisition time for the analysis was 2.7 minutes. Automated data processing using unique and automated peak finding and deconvolution algorithms with library searching against both the National Institute of Standards and Technology (NIST) Mass Spectral Database and the Terpene Essential Oil MS Library resulted in 51 identified components.[1] Analyte identifications are based upon both spectral similarity and DB-5 retention indices.

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 400u
GC:	Hewlett Packard® 6890

Column:
DB-5 4 m x 0.1 mm ID, 0.1 μ phase film

Oven:
40°C for 0.5 minute, then to 280°C at 75°C/minute, hold for 1 minute

Injector: 290°C

Carrier Gas:
Helium, 2.0 ml/minute constant flow

Sample:
No preparation required. 0.2 μL split (200:1) injection.

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

3. Results

Automated data processing includes 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 lavender oil. Figure 2 below shows mass chromatograms for two peaks on the tailing edge of the isopropyl myristate peak. The peaks are located and their spectra are automatically deconvoluted. The resulting deconvoluted spectra match the library spectra with similarities greater than 700 for even the most minor component.

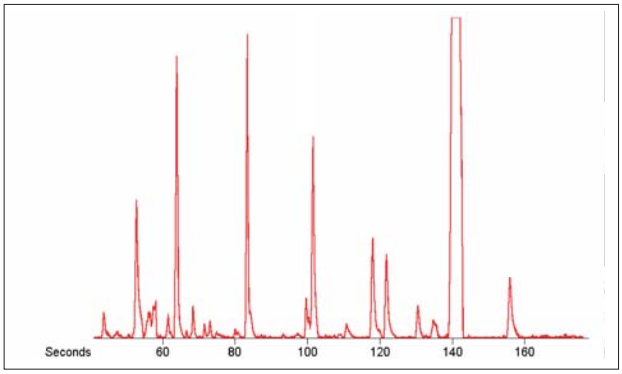


Figure 1. Total Ion Chromatogram (TIC) for 2.7 Minute Analysis of Lavender Oil.

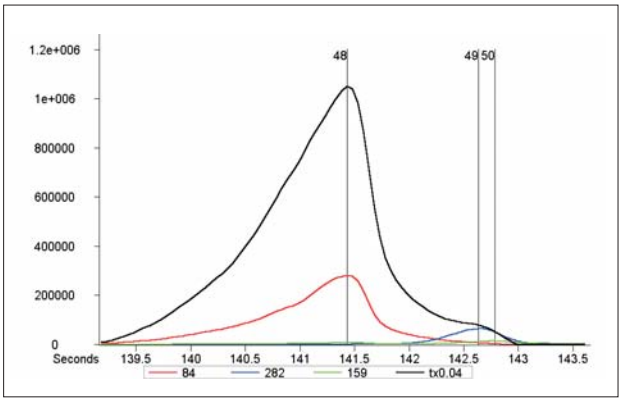


Figure 2. Automated Peak Finding Locates the Minor Components Musk Xylene and Versalide on Tailing Edge of Primary Component Isopropyl Myristate in Lavender Oil. The Total Ion Chromatogram (TIC) is shown in black (scaled by 4%).

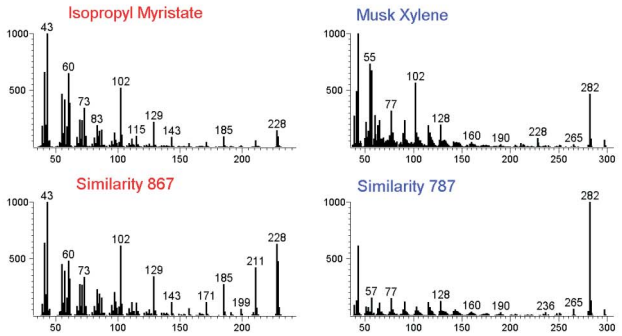
Life Science and Chemical Analysis Solutions

Delivering the Right Results

Table 1. Compound Name, Retention Time (RT), Library Hit Number and Forward and Reverse Spectral Similarity Index for Lavender Oil Analysis.

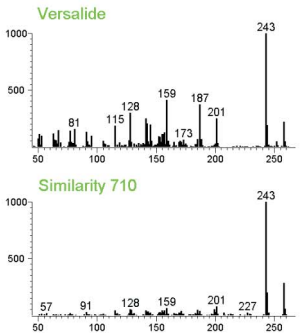
Peak	Name	R.T. (sec.)	Hit Number	Similarity	Reverse
1	1R-à-Pinene	36.13	2	871	888
2	Camphene	38.68	1	833	907
3	á-Pinene	43.78	1	896	897
4	á-Myrcene	47.43	1	745	810
5	à-Terpinene	50.83	4	596	695
6	o-Cymene	51.98	1	818	827
7	Limonene	52.78	1	884	895
8	1,8-Cineole	52.93	1	785	786
9	2-Propanol, 1,1'-oxybis-	54.08	1	820	859
10	γ-Terpinene	57.43	1	800	859
11	Dipropylene glycol	58.13	1	894	918
12	1-Propanol, 2-(2-hydroxypropoxy)-	61.63	2	790	822
13	Plinol A	62.18	1	813	833
14	Linalool	63.93	1	888	891
15	Plinol C	66.63	1	811	835
16	Camphor	68.48	1	917	944
17	Menthone	69.98	1	609	677
18	Isoborneol	70.38	4	718	792
19	Borneol	71.58	1	883	897
20	Terpinen-4-ol	73.13	1	865	895
21	α-Terpineol	74.93	1	794	851
22	Tetrahydroionol	80.03	1	886	935
23	Linalool acetate	83.38	2	899	899
24	Geranyl alcohol	84.43	2	705	729
25	Isobornyl acetate	86.28	1	773	798
26	Lavandulyl acetate*	87.33	1	727	826
27	2,4-Decadienal	89.58	1	704	831
28	α-Terpinyl acetate	93.38	1	724	813
29	Neryl acetate	95.23	1	806	833
30	Geranyl acetate*	97.33	2	610	898
31	α-Cedrene	99.73	1	867	867
32	cis-β-Farnesene	100.43	1	853	860
33	Coumarin*	101.58	1	763	898
34	trans-β-Farnesene*	104.98	5	654	785
35	6-Methyl-γ-trans-Ionone*	106.83	2	697	784
36	trans-β-Ionone	107.43	1	693	735
37	Cuparene*	109.23	1	777	874
38	6-Methyl-α-trans-Ionone*	110.83	1	802	889
39	Lilial	111.33	1	696	785
40	Undecanoic acid, 10-methyl-, methyl ester	111.78	1	650	744
41	Diethyl Phthalate	117.98	1	885	886
42	Epicedrol	118.23	1	793	804
43	Ethyl Dodecanoate*	118.58	1	665	795
44	Dodecanoic acid, 1-methylethyl ester	121.88	1	905	907
45	Methyl tetradecanoate	130.53	1	870	870
46	Benzyl Benzoate*	132.83	1	737	911
47	Luciferin aldehyde	138.58	3	676	697
48	Isopropyl Myristate	141.43	1	867	875
49	Musk xylene	142.63	1	787	809
50	Versalide	142.78	1	710	759
51	Isopropyl Palmitate	156.03	1	903	925

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



Peak 48; Isopropyl Myristate; Similarity 867; Reverse Similarity 875.

Peak 49; Musk Xylene; Similarity 787; Reverse Similarity 809.



Peak 50; Versalide; Similarity 710; Reverse Similarity 759.

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. (Top: Pegasus II spectrum. Bottom: NIST Library spectrum.)

4. Conclusions

General conditions for rapid flavor and fragrance analyses were used to analyze a sample of lavender oil in just 2.7 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. 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.

LECO Corporation • 3000 Lakeview Avenue • St. Joseph, MI 49085 • Phone: 800-292-6141 • Fax: 269-982-8977
info@leco.com • www.leco.com • ISO-9001:2008 • No. FM 24045 • *LECO is a registered trademark of LECO Corporation.*

Form No. 203-821-089 1/09-REV2