Lavender Oil Characterization Using Agilent J&W DB-1ms Ultra Inert Capillary GC Columns

Application Note

Flavors and Fragrances

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Abstract

The essential oil extracted from lavender blossoms is often used in hair and skin care products and is a frequent component in the bouquet of fragrances found in perfumes. Characterization of these complex oils has been conducted using sophisticated gas phase analytical techniques including GC/MS, GC x GC, and GC/TOF. In this application note an Agilent J&W DB-1ms Ultra Inert column is used to resolve the main components in lavender oil samples from several suppliers. The well-resolved components are then identified using a single quad GC/MS versus NIST Library 5.01. The identified peaks then serve as the fingerprint for the analysis. The same separation using GC-FID can then be used to evaluate subsequent samples quickly and cost effectively. The well-resolved, sharp peaks observed on the Agilent J&W DB-1ms Ultra Inert column make this approach possible.

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Introduction

The essential oil extracted from lavender is often used in hair and skin care products and is a frequent component in the bouquet of fragrances found in perfumes. Lavender oil is also seeing increasing application as an aroma and topical therapy agent with a wide array of claimed medicinal benefits [1]. One of the main lavender cultivars for essential oil production is *Lavandula officinalis.* This particular cultivar is also known as *Lavandula angustifolia*, and also as English, common, or true lavender. The oil from this species is prized for the sweet overtones of its fragrance. Other lavender oil cultivars contain higher levels of terpenes, including camphor, which add a less desirable overtone to the fragrance.

Characterization of these complex oils has been conducted using a variety of gas phase analytical techniques, including GC-FID, GC/MS, GC x GC, and GC/TOF [2]. David and Klee used a two-column Deans' switch approach with separate temperature control of a low thermal mass (LTM) module containing a chiral column for the analysis of theses samples [3]. This application note highlights the use of Agilent J&W DB-1ms Ultra Inert columns to characterize lavender oil samples using GC-FID detection and single quadrupole GC/MS identification to establish a sample profile.

Every Agilent J&W Ultra Inert column is rigorously tested with demanding probes for activity. Propionic acid, 4-picoline, and tri-methyl phosphate are included in the test probe mixture to uncover any potential active sites. The goal is to provide the gas chromatographer with the most inert column available for demanding application with active analytes [4–6]. The high inertness of these columns, in combination with exceptional low bleed, translates to improved peak shapes and better resolution of critical pairs such as eucalyptol and d-limonene found in complex lavender oil samples.

Experimental

Sample Prep

A NOW Foods lavender oil sample (sample 1) was obtained at a local GNC retail store. The NOW food sample was labeled as 100% natural aroma therapeutic grade *Lavandula officinalis* as verified by GC/IR. A second sample (sample 2) of *Lavandula Officinalis* was purchased though Sigma Aldrich (Milwaukee, WI). The sample purchased through Sigma Aldrich was labeled as lavender oil 40/42 fleurs and designated as a kosher product. Additional information found on Sigma Aldrich's Web site indicates that the material does not meet either EU or U.S. requirements for natural certification and lists the country of origin as Russia. Each of these samples was diluted 1:20 in Ultra-Resi-Analyzed grade acetone from J.T. Baker (Phillipsburg, NJ) and analyzed using both GC-FID and GC/MS detection.

The chromatographic conditions and supplies used on both the GC-FID and GC/MS systems are highlighted in Tables 1 and 2.

Table 1. Chromatographic Conditions

GCs:	Agilent 7890A/5975B MSD and a 6890N FID equipped
Sampler:	Agilent 7683B, 5.0 µL syringe (Agilent p/n 5188-5246), 1.0 µL injection
Carrier:	Helium 40 cm/s, constant flow MSD system, 35 cm/s FID system
Inlet:	200:1 split
Inlet liner:	MS Certified Deactivated Split Single Taper (Agilent p/n 5188-6576)
Column:	Agilent J&W DB-1ms Ultra Inert 30 m × 0.25 mm × 0.25 µm (Agilent p/n 122-0132UI)
Oven:	62 °C 12.5 min hold, 3 °C/min to 92 °C, then 5 °C/min to 165 °C, then 100 °C/min to 310 °C, 2.5 minute hold
Detection:	MSD source at 300 °C, quadrupole at 180 °C, transfer line at 280 °C, scan range 45–450 amu

Table 2. Flow Path Supplies

Vials:	Amber screw cap (Agilent p/n 5182-0716)
Vial caps:	Blue screw cap (Agilent p/n 5282-0723)
Vial inserts:	100 µL glass/polymer feet (Agilent p/n 5181-1270)
Syringe:	5 µL (Agilent p/n 5181-1273)
Septum:	Advanced Green (Agilent p/n 5183-4759)
Inlet liners:	MS Certified Deactivated Split Single Taper (Agilent p/n 5188-6576)
Ferrules:	0.4 mm id short; 85/15 Vespel/graphite (Agilent p/n 5181-6576)
	0.4 mm id long, 85/15 Vespel/graphite (Agilent p/n 5181-3323)
20x magnifier :	20x Magnifier loupe (Agilent p/n 430–1020)

Results and Discussion

Lavender oil samples were evaluated first by GC-FID area percent with integration parameters set such that the acetone solvent blank was ignored and that all sample components were integrated to provide as accurate an area percent profile as possible. Under the above conditions none of the major peaks was overloaded, enabling an accurate area percent picture for these samples. Peaks of interest greater than 0.25% by area were then targeted as major components and identified using GC/MS spectral data versus the NIST 5.01 Spectral Library. Thirty-five major components were identified in this

manner. These 35 peaks accounted for 96% of the area observed in the GC-FID chromatograms. The remaining 4% comprised more than 100 minor components that were beyond the scope of this study. A list of indentified components, their relative retention times, total ion chromatograms' percentages, and FID area percentages is found in Table 3. GC-FID chromatograms for lavender oil samples one and two are shown in Figures 1 and 3. GC/MS total ion chromatograms for lavender oil samples 1 and 2 are shown in Figures 2 and 4.

Thirty-five peaks at or near a TIC area percent of 0.25% were identified by GC/MS in sample 1. Spectral comparison and identification was done using the NIST 5.01 Mass Spectral Library (Agilent P/N G1033-60034). The spectral profile of sample 2 was quite similar to that of sample 1. Both samples contained less β-*cis*-ocimene than the range of between 4 and 10% called for by ISO Standard 3515.[7] Sample 1 also contained higher levels of champhor than called for in the specification.

The peaks identified by GC/MS account for 96% by area of all of the components in each of these samples. This suggests that the FID profile should suffice as a preliminary evaluation tool for lavender oils once compounds of interest have been identified by GC/MS and a sample profile has been established. This approach has the potential to resolve and characterize lavender oil samples cost-effectively through the use of GC-FID coupled with reliable and reproducible capillary GC separations achievable with Agilent J&W DB-1ms Ultra Inert columns.

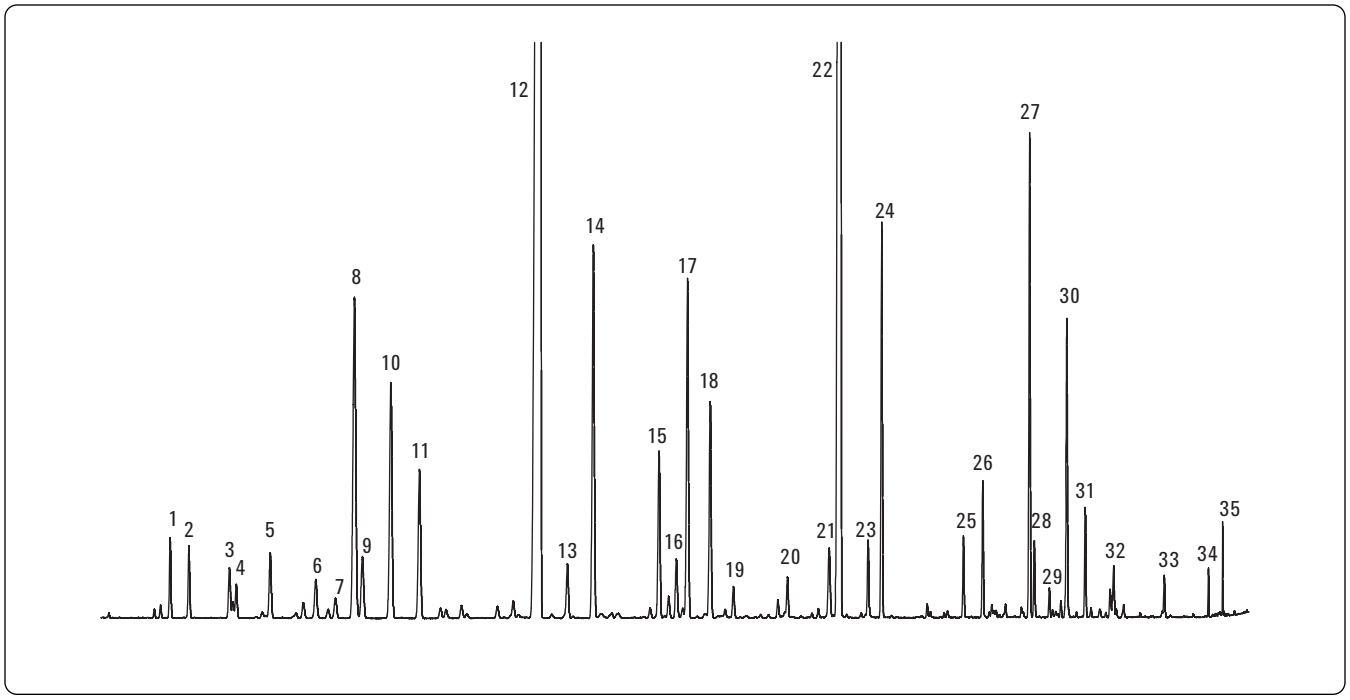

Figure 1. GC-FID chromatogram of lavender oil sample 1 on an Agilent J&W DB-1ms Ultra Inert 30 m × 0.25 mm × 0.25 µm capillary GC column. Refer to Table 1 for chromatographic conditions and to Table 3 for a peak number key.

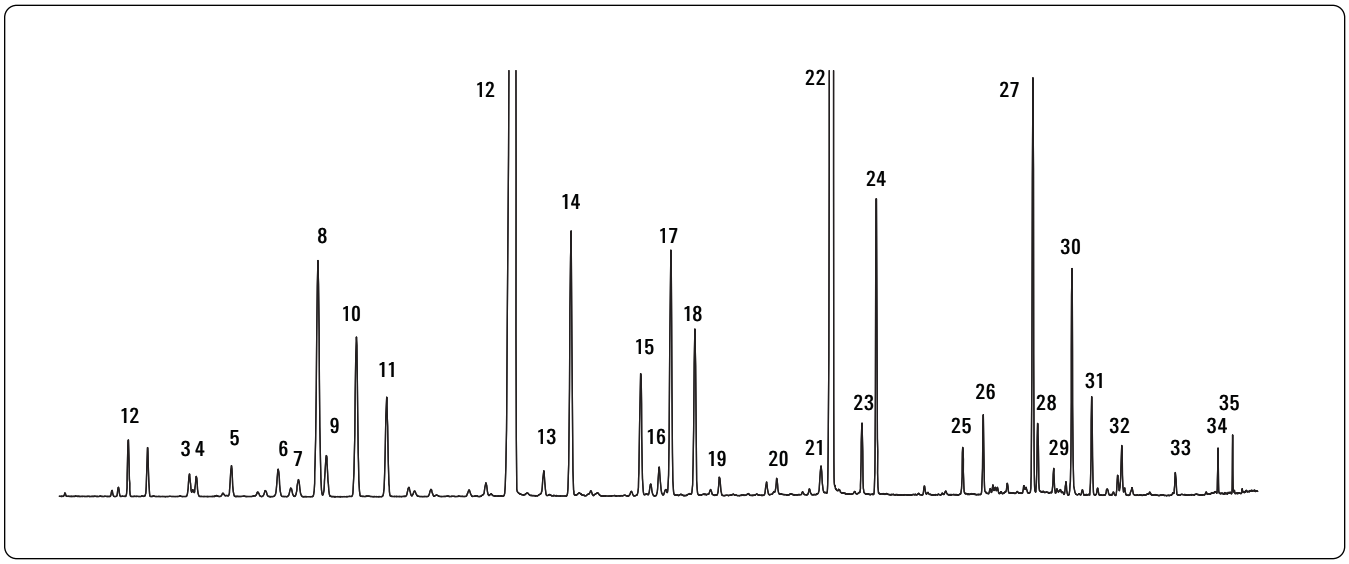

Figure 2. GC/MS total ion chromatogram of lavender oil sample 1 on an Agilent J&W DB-1ms Ultra Inert 30 m × 0.25 mm × 0.25 µm capillary GC column. Refer to Table 1 for chromatographic conditions and to Table 3 for a peak number key.

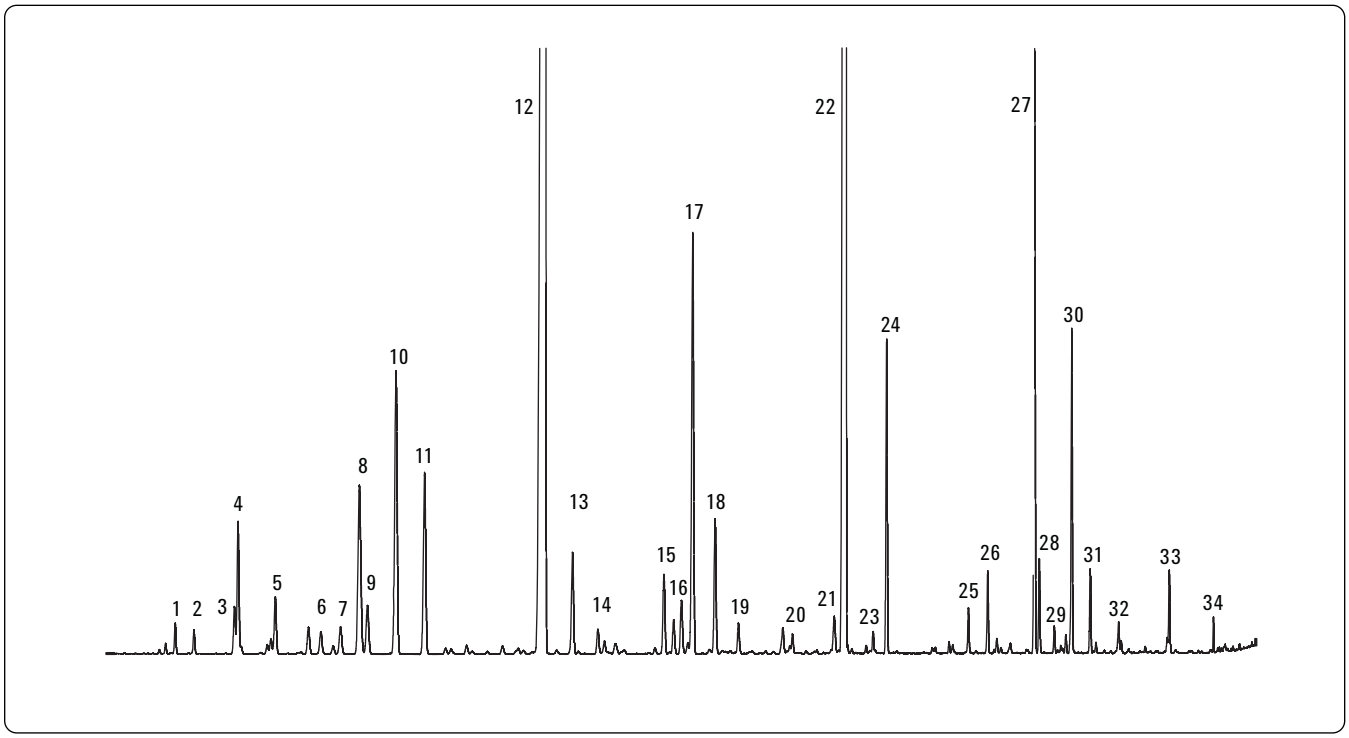

Figure 3. GC-FID chromatogram of lavender oil sample 2 on an Agilent J&W DB-1ms Ultra Inert 30 m × 0.25 mm × 0.25 µm capillary GC column. Refer to Table 1 for chromatographic conditions and to Table 3 for a peak number key. Peak 35 was below integration setpoints in this sample.

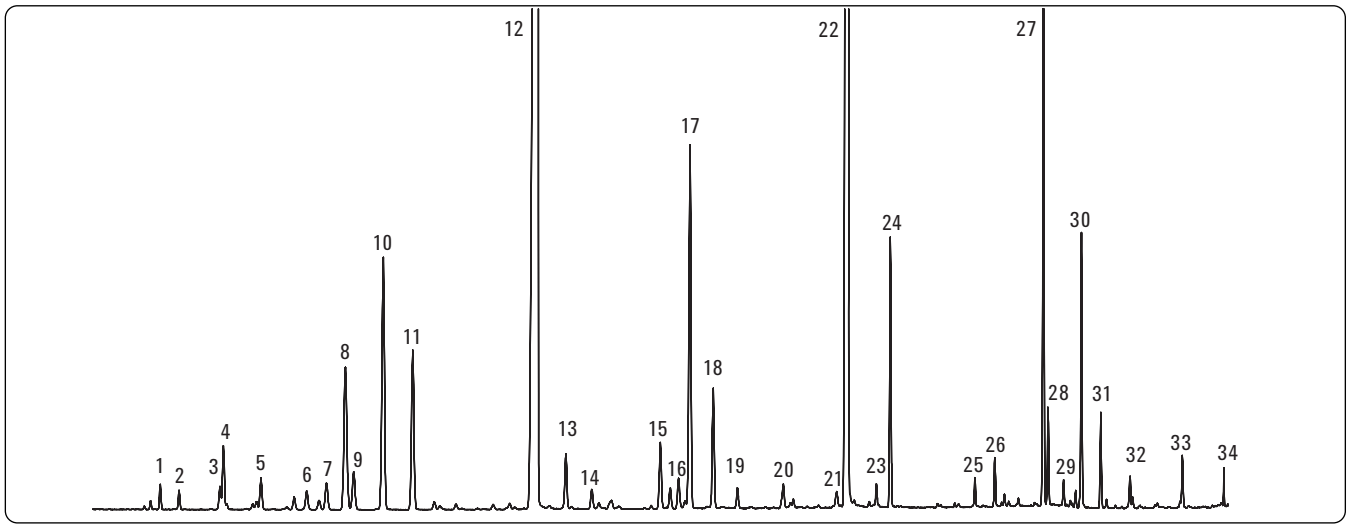

Figure 4. GC/MS total ion chromatogram of lavender oil sample 2 on an Agilent J&W DB-1ms Ultra Inert 30 m × 0.25 mm × 0.25 µm capillary GC column. Refer to Table 1 for chromatographic conditions and to Table 3 for a peak number key. Peak 35 was below integration setpoints in this sample.

Table 3. Peak Identification Table Listing the Relative Total Ion and FID Percentages for Lavender Oil Samples 1 and 2 (The shaded boxes in the table indicate results that depart from ISO specifications.)

Retention time	Peak number	Compound name	TIC% Sam 1	Area % Sam 1	TIC % Sam 2	Area % Sam 2	ISO spec.
6.964	1	α-Pinene	0.492	0.419	0.200	0.173	
7.505	2	Camphene	0.462	0.412	0.160	0.148	
8.692	3	1-Octen-3-ol	0.207	0.357	0.175	0.336	
8.791	4	3-Octanone	0.062	0.103	0.475	0.914	0–2%
9.888	5	β-Myrcene	0.360	0.491	0.301	0.439	
11.216	6	3-Carene	0.387	0.343	0.241	0.232	
11.79	7	o-Cymene	0.244	0.186	0.344	0.252	
12.341	8	Eucalyptol	3.723	3.129	2.121	1.786	0–15%
12.581	9	D-Limonene	0.587	0.562	0.289	0.464	0–0.5%
13.434	10	β-*trans*-Ocimene	2.283	2.623	3.188	2.610	2–6%
14.291	11	β-*cis*-Ocimene	1.374	1.263	1.980	1.601	4–10%
17.926	12	β-Linalool	33.062	33.933	32.889	33.351	25–38%
18.735	13	Octen-1-ol acetate	0.280	0.430	0.595	0.792	
19.512	14	Camphor	3.307	2.777	0.222	0.203	0–0.5%
21.485	15	Borneol	1.445	1.183	0.703	0.591	
22.008	16	Lavandulol	0.331	0.415	0.209	0.407	
22.342	17	Terpine-4-ol	2.796	2.334	3.697	3.063	2–6%
23.025	18	α-Terpinol	1.845	1.488	1.190	0.966	0–1%
23.717	19	Hexyl butyrate	0.189	0.206	0.191	0.215	
25.059	20	Cumic aldehyde	0.125	0.115	0.269	0.116	
26.604	21	*cis*-Geraniol	0.336	0.516	0.159	0.289	
26.947	22	Linalool acetate	35.174	34.416	39.626	38.450	25–45%
27.771	23	Borneol acetate	0.573	0.440	0.169	0.132	
28.176	24	Lavandulyl acetate	2.255	1.919	1.868	1.593	0.3% min
30.63	25	Nerol acetate	0.331	0.385	0.193	0.240	
31.213	26	Geranyl Acetate	0.558	0.615	0.329	0.381	
32.621	27	Caryophyllene	3.172	2.278	4.210	2.937	
32.753	28	α-Santoloene	0.504	0.348	0.641	0.437	
33.21	29	α-Bergamotene	0.173	0.127	0.165	0.127	
33.728	30	β-Farnesene	1.632	1.399	1.734	1.483	
34.288	31	Germacrene D	0.722	0.496	0.613	0.397	
35.14	32	γ-Cardinene	0.357	0.251	0.197	0.188	
36.661	33	Caryophyllene oxide	0.153	0.189	0.305	0.391	
37.871	34	tau-Cardinol	0.166	0.150	0.137	0.096	
38.286	35	α -Bisabolol	0.161	0.155	0.037	0.016	

Conclusions

This application successfully demonstrated reliable analysis and fingerprinting of lavender oil using Agilent J&W DB-1ms Ultra Inert columns with both FID and GC/MSD detection. Peaks of interest at or above the 0.25% by area level were identified by GC/MS and labeled in a corresponding GC-FID chromatogram with the same separation. Once the fingerprinting and identification of the peaks of interest are established, characterization of subsequent samples by GC-FID is straightforward. This two-step approach helps to characterize these complex samples quickly and cost-effectively.

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