Lavender Oil Analysis Using Agilent J&W DB-WAX Ultra Inert Capillary GC Columns

Application Note

Flavors and Fragrances

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Abstract

Lavender essential oil samples were analyzed using the Agilent J&W DB-Wax Ultra Inert GC column and DB-Wax GC column by GC/FID and GC/MSD. Thirty-six major components were identified. The DB-Wax Ultra Inert GC column showed the same selectivity as the DB-Wax GC column. The DB-Wax Ultra Inert GC column however, generated better peak shape and improved sensitivity for active compounds for complex essential oil analysis.

Introduction

Lavender essential oil is one of the most versatile essential oils. The oil is an often used addition in hair and skin care products, and is a frequent component in the bouquet of fragrances found in perfumes. Lavender oil is also widely used in aromatherapy, and is thought to have calming, antiflatulence, and anticolic properties [1]. The oil typically contains more than 100 individual components with many of the minor constituents often unidentified or not quantitated. It normally includes high levels of linalool, and linalyl acetate, moderate levels of lavandulyl acetate, terpinen-4-ol, and lavandulol. The amount of 1,8-cineole and camphor often varies between low to moderate [2]. Lavender essential oil of the same variety can vary strongly in composition from supplier to supplier. Its composition depends on the species, location, ground, weather conditions, and the level of expertise of farmers and distillers. Because of its high market value, lavender oils are frequently adulterated to extend the volumetric quantity, or less valuable oils are counterfeited as lavender oil for economic reasons. Characterization of the oils through chemical analysis is a mandatory step in the production chain, carried out both by researchers and quality control labs.

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Characterization of these complex oils has been conducted using various gas phase analytical techniques, including GC-FID, GC/MS, GCxGC, and GC/TOF [3]. These analyses are often carried out using two different stationary phase columns, a low-polarity column, and a polar column to acquire more reliable qualitative and quantitative data. Relevant analysis and fingerprinting of lavender oil using nonpolar Agilent J&W DB-1ms Ultra Inert columns has been successfully performed by Lynam and Smith [4]. This study highlights the use of polar, Agilent J&W DB-Wax Ultra Inert columns to resolve the main components in lavender oil samples with both GC/FID and GC/MSD. It also compares the selectivity with the DB-Wax column, a column that has been used extensively in the analysis of essential oils. The retention indexes (RI) on the DB-Wax column have long been established, for example, see the Sadtler Standard Gas Chromatography Retention index Library (Sadtler Research Laboratories, Philadelphia, 1984).

Experimental

Samples

- **Sample 1**: Lavender oil was provided by Shanghai Oasis Flavor & Fragrance Co. Ltd (Shanghai, China).
- **Sample 2**: ChromaDex Lavender oil (RG, CAS No. 8000-28-0) was purchased from ANPEL Scientific Instrument Co. Ltd (Shanghai, China). It was noted in its specification that this product line was developed for research and qualitative purposes only.

Each of these samples was diluted 1:20 in ethyl acetate (>99.9%) from J&K Scientific (Beijing, China) and analyzed using both GC-FID and GC/MS detection.

Instrumentation

Table 1 shows the instruments and conditions, and Table 2 lists the consumable supplies flow path.

Table 1. Conditions.

Parameter	Value
GC system	Agilent 7890B/5977A MSD and an Agilent 7890B FID equipped
Column	Agilent J&W DB-Wax UI, 30 m × 0.25 mm, 0.25 µm (p/n 122-7032UI) Agilent J&W DB-Wax , 30 m × 0.25 mm, 0.25 µm (p/n 122-7032)
Autosampler	Agilent 7683B autosampler and sample tray, 5 µL syringe (p/n G4513-80213), 1 µL injection volume
Carrier gas	Helium, constant flow mode, RT locking: D-limonene locked to 8.450 min
Inlet	Split/splitless, 250 °C, split ratio 200:1
Oven	52 °C (2 min), 5 °C/min to 80 °C (4 min), 4 °C/min to 250 °C (1 min)
MSD	Agilent 5977A MSD
Solvent delay	3.4 min
MS temp	230 °C (source), 150 °C (quad)
Transfer line	250 °C
MS	EI, Scan 40–400 amu

Table 2. Flow path supplies.

Parameter	Value
Vials	Amber, write-on spot, certified, 2 mL, screw top vial packs (p/n 5182–0554)
Septa	Nonstick BTO septa (p/n 5183–4757)
Column nut	Self-tightening, inlet/detector (p/n 5190–6194) Self-tightening, for MS interface (p/n 5190–5233)
Ferrules	15% graphite: 85% Vespel, short, 0.4 mm id, for 0.1 to 0.25 mm columns (10/pk, p/n 5181–3323)
Liner	Agilent Ultra Inert split liner with glass wool (p/n 5190–2295)
Inlet seal	Ultra Inert, gold-plated, with washer (p/n 5190–6144)

Results and Discussion

The purpose of the tests was to evaluate the performance of Agilent J&W DB-Wax Ultra Inert columns, and compare the selectivity of Agilent J&W DB-Wax Ultra Inert columns with Agilent J&W DB-Wax columns for essential oil analysis.

Lavender oil samples were evaluated first by GC-FID, then identified by GC/MS. Figures 1 and 2 show GC-FID chromatograms for lavender oil samples 1 and 2. Figures 3 and 4 show GC/MS total ion chromatograms for lavender oil samples 1 and 2.

As shown in Figure 1, the J&W DB-Wax Ultra Inert GC column can separate major components well, with excellent peak shape. Figures 2, 3, and 4 demonstrate the identical selectivity between the DB-Wax and DB-Wax UI GC columns. That means virtually no method development or revalidation is needed when upgrading from a DB-Wax to a DB-Wax UI column.

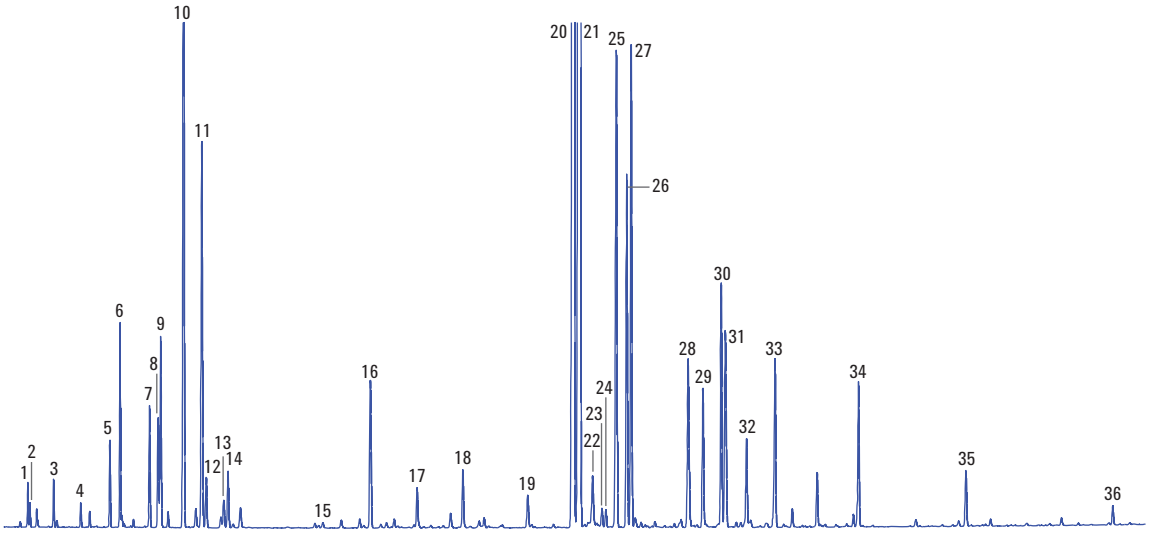

Figure 1. GC/FID chromatogram of lavender oil sample 1 on an Agilent J&W DB-Wax Ultra Inert, 30 m column. See Table 1 for chromatographic conditions, and Table 3 for peak identification.

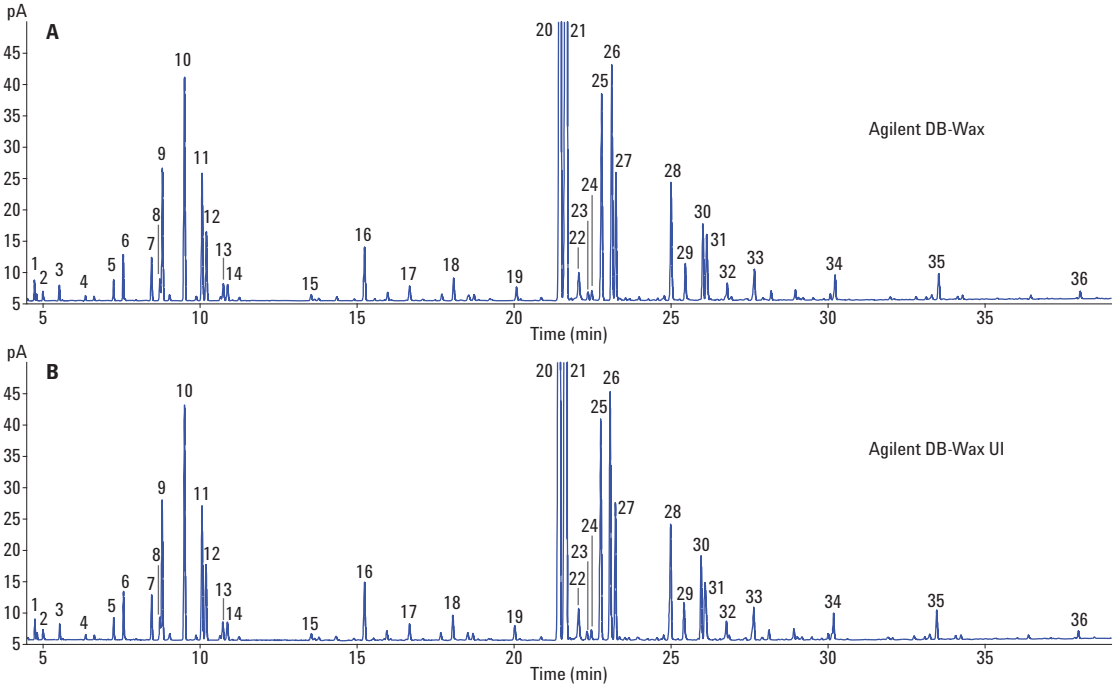

Figure 2. GC/FID chromatogram of lavender oil sample 2 on an Agilent DB-Wax and an Agilent DB-Wax Ultra Inert GC column. For peak identification, see Table 3.

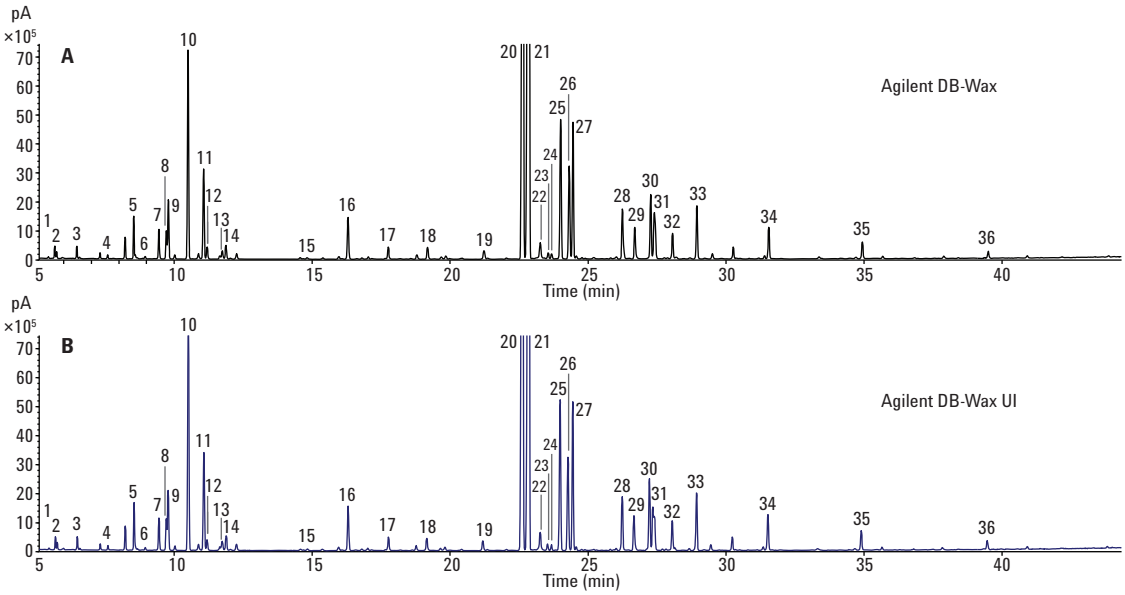

Figure 3. GC/MS total ion chromatogram of lavender oil sample 1. Using an Agilent J&W DB-Wax, 30 m × 0.25 mm, 0.25 µm GC column and an Agilent J&W DB-Wax Ultra Inert, 30 m × 0.25 mm, 0.25 µm GC column.

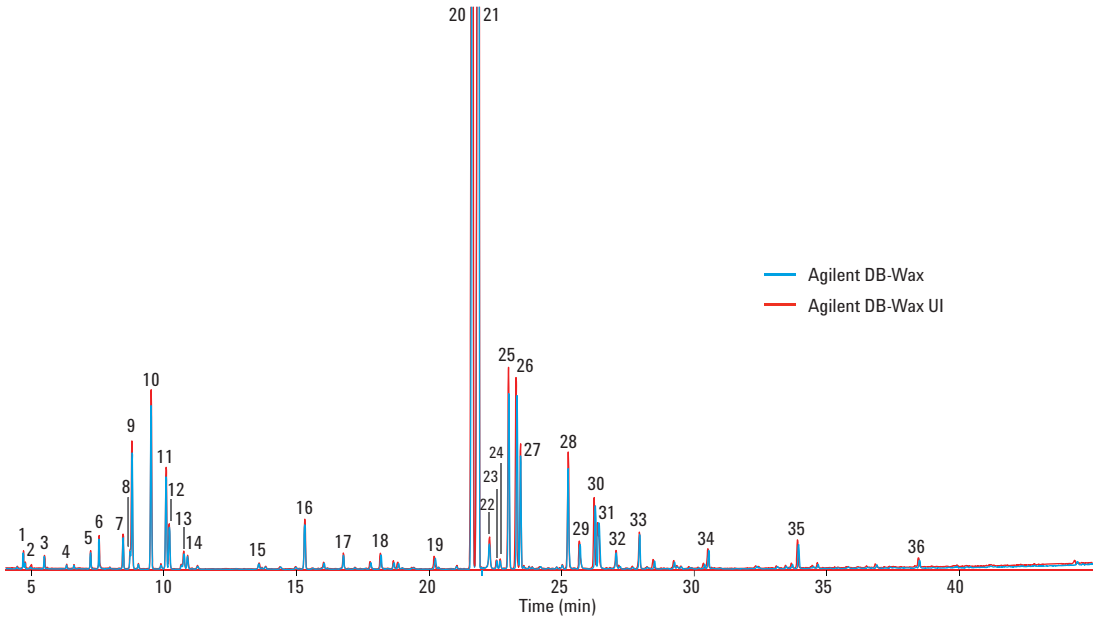

Figure 4. GC/MS total ion chromatogram of lavender oil sample 2. Using an Agilent J&W DB-Wax (blue) and an Agilent J&W DB-Wax Ultra Inert, 30 m × 0.25 mm, 0.25 µm GC column (red).

Table 3. Peak identification by GC/MS and GC/FID area percentages for lavender oil samples 1 and 2 using Agilent DB-Wax UI and Agilent DB-Wax columns.

Peak no.	Compound	Agilent DB-Wax UI RT Sample 2	Agilent DB-Wax RT Sample 2	Agilent DB-Wax UI Area% Sample 1	Agilent DB-Wax Area% Sample 1	Agilent DB-Wax UI Area% Sample 2	Agilent DB-Wax Area% Sample 2	ISO 3515 spec
1	α-Pinene	4.755	4.734	0.166	0.171	0.161	0.158	
2	α-Thujene	4.822	4.799	0.088	0.089	0.052	0.052	
3	Camphene	5.542	5.527	0.194	0.201	0.136	0.134	
4	β-Pinene	6.366	6.356	0.103	0.105	0.047	0.047	
5	3-carene	7.258	7.251	0.373	0.384	0.200	0.096	
6	Myrcene	7.573	7.561	0.846	0.852	0.424	0.416	
7	D-limonene	8.468	8.465	0.594	0.604	0.448	0.416	0–1%
8	β-Phellandrene	8.730	8.730	0.554	0.564	0.227	0.223	
9	Eucalyptol	8.803	8.809	1.029	1.039	1.555	1.524	0–3%
10	*cis*-β-Ocimene	9.518	9.510	5.079	5.105	2.589	2.538	1–10%
11	*trans*-β-Ocimene	10.078	10.069	2.275	2.280	1.580	1.549	0.5-6%
12	3-Octanone	10.199	10.204	0.294	0.296	0.885	0.867	0–3%
13	O-cymene	10.739	10.746	0.178	0.182	0.239	0.235	
14	Hexyl acetate	10.883	10.878	0.362	0.361	0.228	0.224	
15	Hexyl isobutanoate	13.550	13.540	0.035	0.034	0.093	0.091	
16	1-Octen-3-yl-acetate	15.243	15.241	0.980	0.974	0.750	0.735	
17	Hexyl butyrate	16.672	16.673	0.271	0.269	0.218	0.213	
18	1-Octen-3-ol	18.052	18.077	0.374	0.368	0.334	0.328	
19	Camphor	20.024	20.074	0.229	0.227	0.199	0.195	0–1.5%
20	β-Linalool	21.447	21.477	32.713	32.657	33.671	33.016	20–43%
21	Linalool acetate	21.665	21.675	32.506	32.651	38.258	37.513	25–47%
22	α-Santalene	22.049	22.059	0.460	0.454	0.554	0.543	
23	Bornyl acetate	22.321	22.348	0.118	0.133	0.135	0.132	
24	α-Bergamotene	22.458	22.469	0.107	0.113	0.135	0.133	
25	Caryophyllene	22.758	22.782	3.138	3.157	2.876	2.820	
26	Terpinen-4-ol	23.058	23.105	2.165	2.157	3.068	3.008	0–8%
27	Lavandulyl acetate	23.228	23.241	2.843	2.858	1.599	1.568	0–8%
28	β-Farnesene	24.985	24.991	1.204	1.196	1.639	1.607	
29	Lavandulol	25.409	25.443	0.868	0.837	0.501	0.491	0–3%
30	α-Terpineol	25.953	26.004	1.453	1.445	0.972	0.953	0–2%
31	Bornanol+ Germacrene D	26.076	26.128	1.460	1.443	1.032	1.012	
32	Nerol acetate	26.760	26.779	0.557	0.554	0.265	0.260	
33	Geranyl acetate	27.627	27.645	1.083	1.082	0.495	0.485	
34	Geraniol	30.127	30.213	0.870	0.864	0.367	0.361	
35	Caryophyllene oxide	33.445	33.501	0.372	0.364	0.368	0.361	
36	*tau*-Cadinol	37.949	38.004	0.119	0.119	0.096	0.094	

Thirty-six major components in lavender oil samples, from International Standard ISO 3515 [5], were identified, and the area percent was integrated and calculated in this work. These 36 compounds accounted for about 96% of the area observed in the GC-FID chromatograms. Table 3 has a list of identified components, their relative retention times, and FID area percentages. International Standard ISO 3515 specifies certain characteristics of the lavender oils from various origins to facilitate assessment of their quality. The supplier could not provide species or location for sample 2. Lavender for sample 1 was cultivated in Xinjiang, China. The content of key compounds in sample 1 and sample 2 are different, but both of them can meet ISO requirements (Table 3).

DB-Wax and DB-Wax UI GC columns show the same selectivity for lavender oil analysis, and overall similar performance. However, the DB-Wax UI does provide better peak shape in some cases, and some sensitivity improvement because of its improved inertness. Figure 5 shows that some more active compounds such as terpinen-4-ol (peak 26) and α-terpineol (peak 30) exhibit excellent peak shape and better response on the DB-Wax UI GC column. This illustrates the good inertness performance of the column. Although Bornanol and Germacrene D (peak 31) could not be separated well on DB-Wax UI under the testing conditions in Table 1, these two compounds were coeluted on DB-Wax. Better resolution of Bornanol and Germacrene D, and acceptable resolutions of other main compounds could be achieved if the oven temperature program was from 50 °C (5 minutes), 5 °C/min to 250 °C (5 minutes). However, Bornanol and Germacrene D are not the compounds specified for quality evaluation of lavender oil. The testing conditions in Table 1 were used in this application note to get more reliable results.

Every J&W DB-Wax Ultra Inert column is rigorously QC tested with demanding probes to ensure the highest degree of inertness from column-to-column.

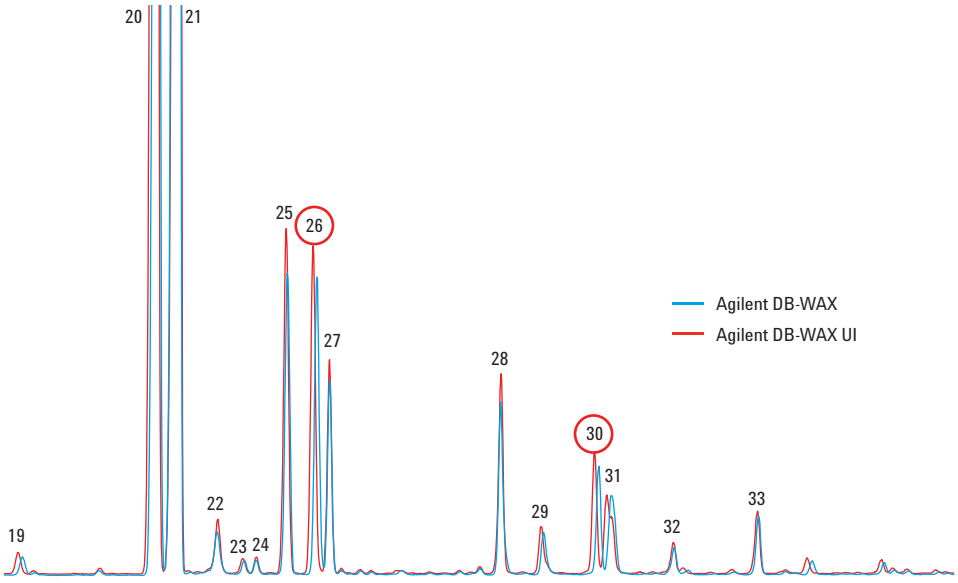

Figure 5. Enlarged section of GC/MS chromatogram of lavender oil sample 2 shown in Figure 4, using an Agilent J&W DB-Wax (blue) and an Agilent J&W DB-Wax Ultra Inert 30 m × 0.25 mm, 0.25 µm GC column (red).

Conclusions

An Agilent J&W DB-Wax Ultra inert GC column was evaluated by analyzing lavender oil samples using GC/FID and GC/MSD. In comparison with the DB-Wax GC column, the same selectivity was successfully demonstrated. This selectivity means that no additional method development or validation is needed when using a DB-Wax UI GC column to replace an Agilent DB-Wax column. Better peak shape and improved sensitivity for active compounds can be achieved because of the high inertness performance of the DB-Wax UI GC column. Characterization of complex lavender essential oil with the DB-Wax UI GC column, combined with the nonpolar Agilent J&W DB-1ms Ultra Inert column can acquire more reliable qualitative and quantitative data, ensuring product quality.

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