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Authentication of Lavender Essential Oil: Commercial Essential Oil Samples and Validity of Standard Specifications



Lavender samples
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Introduction

Lavandula angustifolia Mill., also known as Lavender, is comprised of more than 39 known species. It is one of the most popular essential oils in Aromatherapy and is part of the Lamiaceae family. The flowering plant of Lavender (*Lavandula angustifolia* or *Lavandula officinalis*) is used to produce Lavender essential oils. It is a sweet, camphoraceous, aromatic and pale yellow to colorless essential oil. It is widely used in Aromatherapy and in the fragrance industry because of its biological activities and aromatic potentials. It is also considered one of the most widely-used essential oils in Aromatherapy and the perfume industry. Lavender is grown worldwide with most of the industrial production coming from France, Bulgaria, China, India, New Zealand, Australia, Russia, and USA. Global production of Lavender is approximately 500 metric tons. Commercial essential oils are obtained by steam distillation of freshly cut flowering tops and stalks, with an essential oil yield in the range of 0.6 to 1 percent (Fenarali, 1971).

The wholesale price of Lavender essential oil starts from \$50 US to \$500 US per kg, depending upon factors such as origin and availability. Traditionally, certified organic Lavender essential oils are the highest priced variety. Typical retail pricing of Lavender essential oil can range from \$5 US to \$50 US per 15ml bottle. Because of its economic importance and popularity worldwide, Lavender essential oil is also one of the most commonly adulterated essential oils. In this article, the authors' intent is to present current trends in Lavender adulteration and techniques to detect those adulterations. We have randomly analyzed 15 different market samples so as to observe ongoing adulteration in the market. The samples were collected from a variety of sellers worldwide.

Most were obtained from online essential oil suppliers in the US, a few were directly from the distiller, a few were from retail stores, and one was prepackaged with a diffuser item. In addition, some of the oils were labeled as ECOCERT, organic, wild-crafted, and "certified natural therapeutic grade."

Trends in Lavender adulteration and detection

Common ways Lavender essential oil is adulterated:

1. Addition of nonvolatile component(s).
2. Addition of volatile components naturally found in Lavender essential oil. There are several components of Lavender that can be commercially obtained as pure volatile components.
3. Addition of fractions obtained from other essential oils to Lavender essential oil.
4. Intermixing of two similar oils to mimic a Lavender essential oil profile.

These adulteration trends are further outlined and explained in Table 1.

Lavender adulteration can also be determined by measurement of physical constants such as relative density (0.878 to 0.892), refractive index (1.455 to 1.466), optical rotation (-12.5 degrees to -6.0 degrees), acid value (max 1), and ester value (102.5–165). If a Lavender sample falls out of those ranges, it is considered adulterated in some form. In addition to GC/MS and physical constant measurement, Lavender adulteration can also be determined by enantioselective GC/MS. Stoyanova and Grozeva (2008) have performed chiral GC/MS on Lavender essential oil and have found authentic Lavender should have the following enantiomeric distribution: (+)-linalyl acetate (0%), (3R)-(-)-linalool (95.0–96.6%), (3S)- (+)-linalool (3.4–5.0%), (3R)-(+)-camphor (27.4–52.2%), and (3S)- (-)-camphor (47.8–78.6%).

Adulteration type	Means of adulteration	Means of adulteration detection	Adulteration level of sophistication
Unrelated, economically motivated addition of 1 or more non-volatile components	Simple addition of dipropylene glycol (DPG), tripropylene glycol (TPG), DPG diacetate, kerosene, herculene, diethyl phthalate, benzyl alcohol, benzyl benzoate, diethylene glycol monoethyl ether (DGME), diacetone alcohol, Isopar (odorless kerosene fraction), isopropyl myristate, triacetin, nonvolatile antioxidant butylated hydroxytoluene (BHT), butylated hydroxy anisole (BHA), Vitamin E, carrier oils such as coconut, cooking oils etc. Those components are odorless and can easily pass an organoleptic test. A more experienced nose can potentially detect an odor variance, but this is also not certain.	The paper absorption method can be performed by putting a drop of Lavender on a piece of blotting paper. If everything from the drop evaporates out in 1–2 hours, it is considered adulteration free from carrier oil. If a halo of grease remains, it's considered adulterated with carrier or some other non-volatile oil (Lis-Balchin, 2002). Thin layer chromatography (TLC), organoleptic evaluation, or running a high temperature gas chromatography/mass spectrometry (GC/MS) test (which can elute high-boiling compounds) are also means of detecting this variety of adulteration.	This is considered a simple and unsophisticated form of adulteration.
Addition of Lavender-related synthetic volatile component	Several components that naturally occur in Lavender can be chemically synthesized such as terpinen-4-ol, linalool, linalyl acetate, <i>cis</i> -ocimene, <i>trans</i> -ocimene, 3-octanone, 3-octanol, camphor, and borneol to name a few. These synthetic components can be added to Lavender to gain an economic advantage and/or create a more favorable aroma or chemical profile.	Synthetic markers (trace components produced during synthesis of aroma chemicals which do not exist in nature) and biomarkers of Lavender components (those components that are not economically feasible or are not possible to synthesise are called biomarkers). Biomarkers include cryptone (>0.1%), lavandulyl acetate (>3%), santalene (>0.01%) (Burfield, 2003) for French Lavender. Detecting synthetic markers in components such as terpinen-4-ol (trihydroxy terpineol), linalool (α -linalool, dihydrolinalool, tetrahydro-linalool, dehydrolinalool, plinol <i>cis</i> - or <i>trans</i> -), linalyl acetate (dihydrolinalyl acetate, dehydrolinalyl acetate, α -linalyl acetate, plinyl acetate <i>cis</i> - or <i>trans</i> -), 3-octanone and 3-octanol (shows homologous pair such as 3-nonanone, 3-heptanone or 2-heptaone or 3-heptanol or 3-nonanol marker) indicates synthetic component adulteration. Thermally rearranged markers can also be observed in thermally produced chemicals such as myrcene, ocimene, camphor etc. But most of the time they are undetectable in ordinary GC/MS (Satyal and Pappas, 2016).	This is considered a more sophisticated type of adulteration. In some cases, GC/MS and gas chromatography isotope ratio mass spectrometry (GC-IR-MS) fail to recognize this type of addition.
Addition of fractions obtained from other essential oils	Chemicals such as β -caryophyllene, linalool from Ho wood or Ho leaf (<i>Cinnamomum camphora</i>), acetylated Ho wood or Ho leaf, α pinene, α -thujene, limonene, citral, caryophyllene fraction obtained from Copaiba Balsam (<i>Copaifera officinalis</i>) and Gurjun Balsam (<i>Dipterocarpus turbinatus</i>) or Clove (<i>Syzygium aromaticum</i>) oil can be added as a natural (non-synthesized) adulteration.	Using GC/MS and looking over biomarkers and unexpected acetylated components found in Lavender essential oils such as linalool oxide acetate <i>cis</i> and <i>trans</i> . Balsam can be identified by higher α -copaene, aromadendrene, allo aromadendrene biomarkers.	Can be a very advanced means of adulteration. Often C14 and chiral GC/MS tests fail to detect such types of adulteration.

Table 1. Types of adulteration and how they are detected.

Addition of a similar essential oil	A similar, less expensive essential oil added to Lavender is common. The most common adulterants are Lavandin (<i>Lavandula x intermedia</i>) oil, Spike Lavender (<i>Lavandula latifolia</i>), Eucalyptus (<i>Eucalyptus globulus</i>) oil, Clary Sage (<i>Salvia sclarea</i>) oil (expensive, so not used often), Petitgrain (<i>Citrus aurantium</i> var. <i>amara</i> fol.) oil (expensive), Rosewood (<i>Aniba rosaeodora</i>) oil or fractions of these oils.	Lavandin oil has 1,8-cineole, camphor, borneol in concentrations of more than 1%; by looking over biomarker molecules, adulteration can be predicted. Presence of rosewood oxide confirms the presence of Rosewood EO in Lavender EO.	Chiral analysis of EO can also be useful in detecting this type of adulteration. Typically, this type of adulteration is challenging to detect.
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Table 1. Types of adulteration and how they are detected.

Beyond chiral GC/MS and standard GC/MS, isotopic ratio GC/MS is also used to detect adulteration (Jung *et al.*, 2004). GC-IR-MS is only applicable if fossil fuel-derived synthetic compounds have been added to the essential oils. This equipment is considerably more expensive than standard GC/MS setups (Satyal and Pappas, 2016).

Not only is adulteration carried out by the above-described methods, sometimes it is also carried out using expired or oxidized Lavender essential oils. The major components of Lavender are linalool, linalyl acetate, and caryophyllene (Sköld *et al.*, 2007). Out of these components, trans-caryophyllene is most susceptible to oxidation to produce oxidized chemicals (caryophyllene oxide) which is the least sensitizing molecule (Sköld *et al.*, 2006). Highly oxidized Lavender produces hydroxide, epoxides and hydroperoxides of linalool, linalyl acetate, and caryophyllene (Nilsson *et al.*, 2008). Autoxidation happens in the allylic positions of double bonds in linalool and linalyl acetate (C6-C7) (Sköld *et al.*, 2007). Oxidized components of Lavender are found to possess sensitizing effects on skin (Nilsson *et al.*, 2008); however, few contact allergies have been reported (Letizia *et al.*, 2003). Proper handling and storage of volatile compounds is always good practice. Poorly handled or expired products can be added to pure fresh Lavender for economically motivated adulteration. An increase in oxidized products like linalool oxide and caryophyllene oxide indicates autoxidation of linalool and caryophyllene (Misharina *et al.*, 2003). Linalool readily undergoes autoxidation to form cyclized molecules: furanoids and pyranoids (Sköld *et al.*, 2002), hence the concentration of furanoids and pyranoids in expired Lavender essential oil is relatively high as compared to freshly distilled Lavender (Lawrence, 2012). Comparatively, due to the large bulky ace-

tate group, linalyl acetate is unable to form cyclized furanoid and pyranoid molecules. Sesquiterpene oxide is formed from the oxidation of sesquiterpenes (Hădărugă *et al.*, 2014). Ascaridole glycol *cis*- and *trans*- along with epoxide are observed as oxidized products of terpinen-4-ol (Tranchida *et al.*, 2010).

Market survey of commercially available essential oil in conjunction with standard ranges

The International Organization for Standardization (ISO) and the British Pharmacopoeia are helpful in adulteration detection; however, they have some limitations. For example, their ranges do not necessarily identify adulterated essential oils because of genetic variation and other factors, such as age, vegetative cycle stage, climatic condition, and soil composition, responsible for the considerable variation in essential oil compositions (Lawrence, 2007). This means sometimes a pure Lavender essential oil may not fit the established standard ranges. ISO and the British Pharmacopoeia only mention one biomarker (linalyl acetate) for consideration in adulteration identification. Ideally it is better to have more than one biomarker in the specification table to track multiple types of adulteration such as in the cases of cryptone and santalene. The British Pharmacopoeia is also missing one of the most active odorant molecules, ocimene (*cis*- and *trans*-), in its specification ranges. In this study of Lavender adulteration, other biomarkers are also observed so as to provide their abundance in authentic and adulterated Lavender essential oils.

Fifteen different and random commercial samples were tested specifically for indications of adulteration. It was found that most of the commercial samples were adulterated by some means. Those results and comments on those results are outlined below and in Table 2.

A. Unadulterated samples which follow standard specification ranges:

Samples J, L, R, S, V, X follow standard specification ranges, indicating that they are unadulterated standard samples. Even though there is not an indication of adulteration in these samples, there is some variation that could be a result of various origins. They do also have healthy levels of lavandulyl acetate, lavandulol, and cryptone.

B. Unadulterated samples which do not follow standard specification ranges:

Sample Q contained 4.41% lavandulyl acetate and 0.37% cryptone, indicating it was an authentic sample. In spite of this, it had high 1,8-cineole (1.68%), which is not within the standard specification range. This unusual chemical composition might be caused by various factors such as soil condition, harvesting time, and genetic features. While not certain, it may be possible that a very small amount of Lavandin was grown along with Lavender. This would not affect quantities of biomarkers (lavandulyl acetate and cryptone) in the Lavender, but could account for the small increase (0.68%) of 1,8-cineole.

Sample I is a lab-distilled Chinese Lavender containing a low level of linalyl acetate (12.73%) and high linalool content (53.91%) compared to standard specification. Linalyl acetate will decompose and become linalool in a slightly acidic environment, such as during hydrodistillation where the pH of the water is lower than 7. This is an example where a pure oil does not follow the standard, expected component ranges.

C. Adulterated samples which follow standard specification ranges:

Sample P meets the standard specifications, but contains synthetic markers such as cis- and trans-plinyl acetate, which are indicators of synthetic linalyl acetate. Similarly, synthetic linalool has been detected in this sample which can be confirmed by the presence of plinol as the synthetic marker.

Sample W met the standard specifications, but contained trace quantities of synthetic markers such as a-linalool and dihydrolinalyl acetate, as well as the presence of synthetic linalool and synthetic linalyl acetate. It appears that the concentration of linalool

Compounds	BP	F/S	F/M	Bu	Ru	Au	Ot	I	J	K	L	M	N	O
3-octanone	0.1-5.0	tr-2	1-2.5	0.2-1.6	0-0.6	2-5	0-3	0.14	0.1	ND	0.37	0.29	ND	0.81
limonene	<1	0-0.5	0-0.3	0-0.6	0-1	0-0.5	0-1	0.2	0.39	4.92	0.36	0.92	2.42	0.95
β-phellandrene		tr-0.5	0-0.2	0-0.6	0-1	0-0.5	0-1	0.02	0.17	0.07	tr	ND	0.31	0.61
1,8- cineole	<2.5	0-1	0-0.5	0-2	0-2.5	0-1	0-3	0.32	0.54	3.58	1.39	6.70	2.38	8.38
cis-β-ocimene		4-10	0-2.5	3-9	3-8	3-9	1-10	2.48	3.67	ND	2.07	6.70	ND	2.73
trans-β-ocimene		1.5-6	0-2	2-5	2-5	0.5-1	0.5-6	1.55	1.91	ND	1.23	0.83	ND	1.96
linalool	20-45	25-38	30-45	22-34	20-35	25-38	20-43	53.91	33.56	9.45	32.62	36.4	22.36	43.11
camphor	<1.2	tr-0.5	0-1.2	0-0.6	0-0.6	0-0.5	0-1.5	0.12	0.21	3.04	0.60	6.92	0.41	5.74
lavandulol		>0.3	0-0.5	>0.3	>0.1	>0.3	0-3	4.70	1.95	ND	0.43	ND	ND	0.04
terpenin-4-ol	0.1-8.0	2-6	0-1.5	1.5-2	1-2.5	1.5-6	1-8	1.57	0.82	1.58	4.54	1.84	1.25	0.27
cryptone								0.08	0.23	ND	0.08	ND	ND	0.23
α-terpineol	<2.0	0-1	0.5-1.5	0.8-2	0.5-2	0-1	0-2	2.39	1.41	3.55	0.76	1.14	3.16	0.91
linalyl acetate	25-47	25-45	33-46	30-42	29-44	25-45	25-47	18.57	31.59	12.73	40.36	30.45	22.40	19.03
lavandulyl acetate	>0.2	>2	0-1.3	2-5	1-3.5	>1	0-8	4.92	9.92	ND	1.53	1.29	ND	0.24
α-santalene								0.01	0.39	ND	0.31	ND	ND	ND

Table 2. Comparison of market-surveyed Lavender essential oils with standard specification ranges (ISO and British Pharmacopoeia).

Note that “ND” refers to not detected component, “tr” refers to component less than 0.01%, items outside of specification are indicated in red. All the numbers presented in the table are in percentages. Components in bold letters are biomarkers of Lavender essential oils. These are not economically feasible to adulterate. The samples analyzed are categorized in the following categories 1. Lab distilled (sample I); 2. Online supplier (samples S, K, M, T); 3. Mid-size online supplier (samples U, V, W); 4. Large US distributor (not MLM) (sample P); 5. Direct from distiller; origin is known (samples Q, C, J, L, R, X); 6. Large US shopping center (samples N, O).

and linalyl acetate was increased to 28% and 50% (respectively), but in doing so has lowered its key components of cryptone and lavandulyl acetate to 0.09% and 0.68%. So from this example it can be concluded that minor biomarkers are also important in addition to synthetic markers in detecting adulteration in Lavender essential oil (EO).

D. Adulterated samples which do not follow standard specification ranges:

Sample K contains levels of 1,8-cineole and camphor that are much too high to be true Lavender. The 1,8-cineole content should not be over 1% in true *Lavandula angustifolia* or *L. officinalis*. The level of α -pinene (10.98%) is too high. Furthermore, lavandulol or lavandulyl acetate are components that are always present in Lavender EO and this product did not contain either of these components. In addition, the percentages of dihydrolinalool (0.09%) and dihydrolinalyl acetate (0.11%) indicate that all the linalool and linalyl acetate are from synthetic sources, as these markers are by-products of the synthetic manufacturing process. β -ionone (0.19%) was also observed and nopyl acetate, which does not occur in

P	Q	R	S	T	U	V	W	X
2.08	0.6	1.87	1.62	0.18	0.14	0.66	0.81	1.79
0.26	0.42	0.84	0.55	0.44	0.20	0.28	0.14	0.40
0.24	0.12	0.45	0.27	0.03	tr	0.17	tr	0.32
0.61	1.68	1.14	0.94	4.76	6.55	0.82	2.05	0.51
1.03	1.83	0.55	0.51	0.19	ND	3.29	0.09	3.98
0.96	1.36	0.66	1.30	0.15	0.15	3.17	0.17	2.75
37.25	27.46	26.79	31.97	35.19	23.59	26.93	28.02	25.81
0.13	0.42	0.35	0.45	3.74	11.98	0.26	0.87	0.24
0.48	0.55	0.27	0.98	ND	1.21	1.05	0.30	0.81
1.38	6.94	0.70	0.57	1.91	0.54	4.56	1.38	5.57
0.14	0.37	0.49	0.47	0.02	ND	0.19	0.09	0.23
0.52	1.14	1.39	2.18	0.61	0.62	0.50	1.91	1.38
45.14	37.78	49.99	40.52	42.25	24.9	37.76	50.00	37.45
1.74	4.41	1.78	4.45	0.43	2.53	5.28	0.68	4.70
ND								

BP=British Pharmacopoea, F/S=France ISO (Spontaneous), F/M=France ISO Maillette, Bu=Bulgarian ISO, Ru=Russian ISO, Au=Australian ISO, Ot=Other ISO

nature, was found in the product at almost 12%.

Sample M was a mixture of synthetic linalool, linalyl acetate and Lavandin. Presence of Lavandin can be confirmed by the high 1,8-cineole (6.70%) content like in the case of sample K. Synthetic linalool is present in this sample which can be confirmed by the presence of plinol and plinyl acetate. This sample is considered a misrepresentation of Lavender and does not pass standard specification.

Sample N was a completely reconstructed Lavender due to the presence of DPG (10%) with other unrelated synthetic aroma chemicals such as nopyl acetate (0.98%), ricalidic acid lactone (0.19%), and cyclohexanol <4-tert-butyl acetate>. Related aroma chemicals such as synthetic linalool and linalyl acetate have been detected from its minor synthetic markers: dihydrolinalool and dihydrolinalyl acetate.

Sample T is similar to sample K in terms of chemical profile. Lavandulyl acetate (0.43%) and cryptone (0.02%) are the lowest of all 15 samples, confirming the misrepresentation of the botanical. It also has synthetic markers for linalool and linalyl acetate as dihydrolinalool (0.31%) and dihydrolinalyl acetate (0.57%).

Sample U is also a misrepresentation of botanical Lavender, similar to samples K and T.

Conclusion

There are various testing methods such as GC/MS, enantiomeric ratio comparison, biomarkers, trace synthetic markers and physical constant testing that can be used to detect Lavender oil adulteration. Economically motivated adulteration of Lavender essential oil is a common practice in the industry. Nearly half of the samples reviewed for this paper had some form of detectable adulteration. The means of adulteration are varied and meet the generally accepted specifications, as per ISO and the British Pharmacopoeia. There is no guarantee that a Lavender essential oil has not been adulterated. ☞

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