A Survey of Retail Lavender Essential Oils to Detect Adulterations

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Abstract

Currently there is little regulation of essential oils for fragrance, aromatherapy or homeopathic use. Good Manufacturing Practices (GMP) regulations that went into effect in 2010 require only that the oils be pesticide free, but do not address oil composition, formulation, the Lavender species that make the feedstock, or the claims made by distributors or retailers who re-package these products for individual sale.i

Identifying nomenclature is ambiguous. Package labels of most aromatherapy products claim to be 100% natural oil but the amounts of specific ingredients are not listed. Both Lavender angustifolia and Lavender officinalis are used for aroma therapy and labeled as 100% "Natural" Lavender oil. Some references claim that Lavender angustifolia and Lavender officinalis are just different names for the same thing. However, they are two different species, Lavender officinalis lacks the therapeutic qualities needed for aromatherapy.ii

The need for product uniformity is understandable, as plants, even those grown side-by-side, can be completely different, and plants grown in different geographic locations adapt to their local environment. This effect, is known as "Agronomic Accommodation". Plant feedstock inconsistency, man-made synthetic oils and formulations result in product ambiguity for both the retailers who buy bulk oils and repackage their own products, and for consumers who rely on labeling and advice for therapeutic efficacy.

This report presents a new analytical method that provides the ability to identify synthetic and natural lavender oils and differentiate between feed stocks from the different lavender species alone and in mixtures.

Introduction

Essential oils come from plants, usually harvested through distillation or pressing. Many of these oils are touted to have medicinal properties used for homeopathic medicine, while others are used as fragrances in air fresheners and toiletries. Lavender is a common aromatherapy and fragrance staple in both the wholesale and retail markets.

Different species of Lavender have their own advertized uses. Spike lavender is used for burns but claims to have better medicinal properties than the angustifolia speciesiii. Lavandin, a botanical hybrid of spike lavender and angustifolia, are usually used only for their aromatic properties.iv The spike lavender and lavandin may be used as natural replacement or adjuncts in angustifolia lavender formulations. There may also be synthetic adulterations to lavender oil as well.

Formulations include pure undiluted (neat) oils, mixtures of lavender oils of different species, mixtures of lavender with non-lavender oil and dilutions of the above. Such a variety with lax regulations governing quality control or content labeling leads to consumer and sometime wholesaler confusion. This method provides the analyst the ability to identify synthetic and natural lavender oils and differentiate between feed stocks from different lavender species. Manufacturers, wholesalers, and retailers may also find this analytical method useful to monitor feedstock, oil extraction, purity, and product formulations.

Materials and Methods

Lavender aromatherapy products with labels indicating 100 % natural oil were purchased from various companies. Analytical standard Linalool R and S enantiomers (R/S CAS# 126-91-0) was obtained from ACROS (New Jersey, USA 1-800-ACROS-01) and Lavender (CAS# 8000-28-0), Synthetic lavender (W512850), Spike lavender (CAS# 84837-04-7) were obtained from Sigma-Aldrich (1-800-325-3010). Lavandin, used as a standard, was purchased from Frontier and retail samples were purchased from local retailers. The oils were diluted to 0.001% in methanol for chiral analysis on an Agilent 6890 GC and Agilent 5975 N MSD (Agilent, Cupertino, CA) with a Restek β-DEXse 30 meter, 0.25 mmID, 0.25 μm capillary column. The following analytical conditions were used. Oven temp: 70°C (hold 1 min.) to 175°C @ 2°C/min. (hold 1 min.) to 230°C @ 100°C/min. (hold 2 min.), flow rate of 1.3 mL/min., and split ratio of 40:1, with helium carrier gas.

Results

Experiment 1: Identification of Natural and Synthetic Lavender Feedstocks by R and S Linalool Enantiomeric Ratios.

Chiral molecules, called enantiomers, are different from each other because they are not super-imposable on their mirror-images. In plants enzymes produce R and S linalool enantiomers in a ratio of 85%:15% R respectively, while man-made linalool is synthesized in a R to S ratio that is closer to 55%:45%.v

Because enantiomers have the same structure and physical properties, they cannot be separated by conventional chromatography. Chiral separations are possible using special stationary phases based on beta-dextran polymers. Restek's β-DEXse column was specifically chosen for it's ability to separate linalool R and S enantiomers. By determining the linalool R and S ratios natural linalool from plant and synthetic linalool can be identified. Figure 1 presents the baseline separation of R and S enantiomers from the analytical standard only R and S linalool. Figure 2 presents the same chiral separation of natural and synthetic lavender oils. Figure 3 is a concentration curve showing how mixtures of natural and synthetic oils can be identified and quantitated.

Figure 1. Separation of Linalool R and S Entomers. The leading peak is the R enantiomer followed by the S enantiomer and Mass Spectra

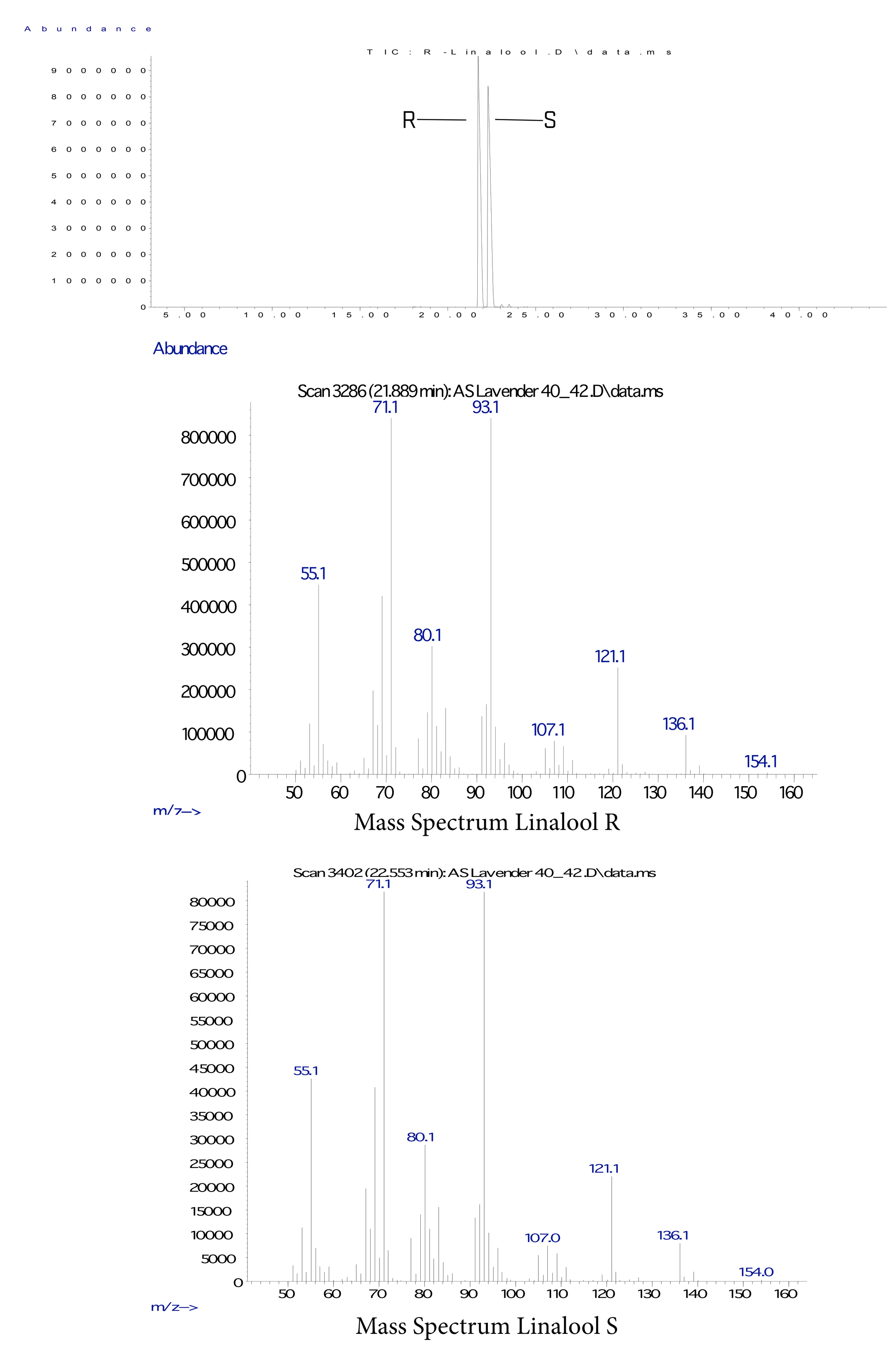
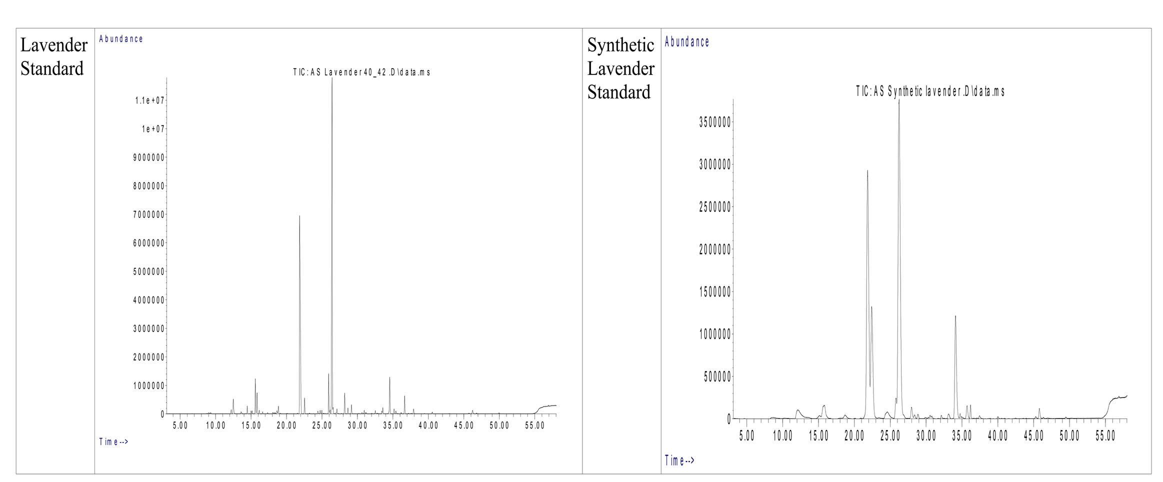


Figure 2. Chiral Separation of Linalool R and S in Natural and Synthetic Feedstocks. In plants the S enantiomer ratio is 15% or less, while the S enantiomer in synthetic feedstock is greater that 15%.



Experiment 2:

Figure 3. Mixtures of Natural and Synthetic Feedstocks. The R to S linalool ratio is the basis for determining the relative abundance of synthetic oils mixed with natural oils. As the amount of synthetic lavender oil increases the peak of S linalool increases.

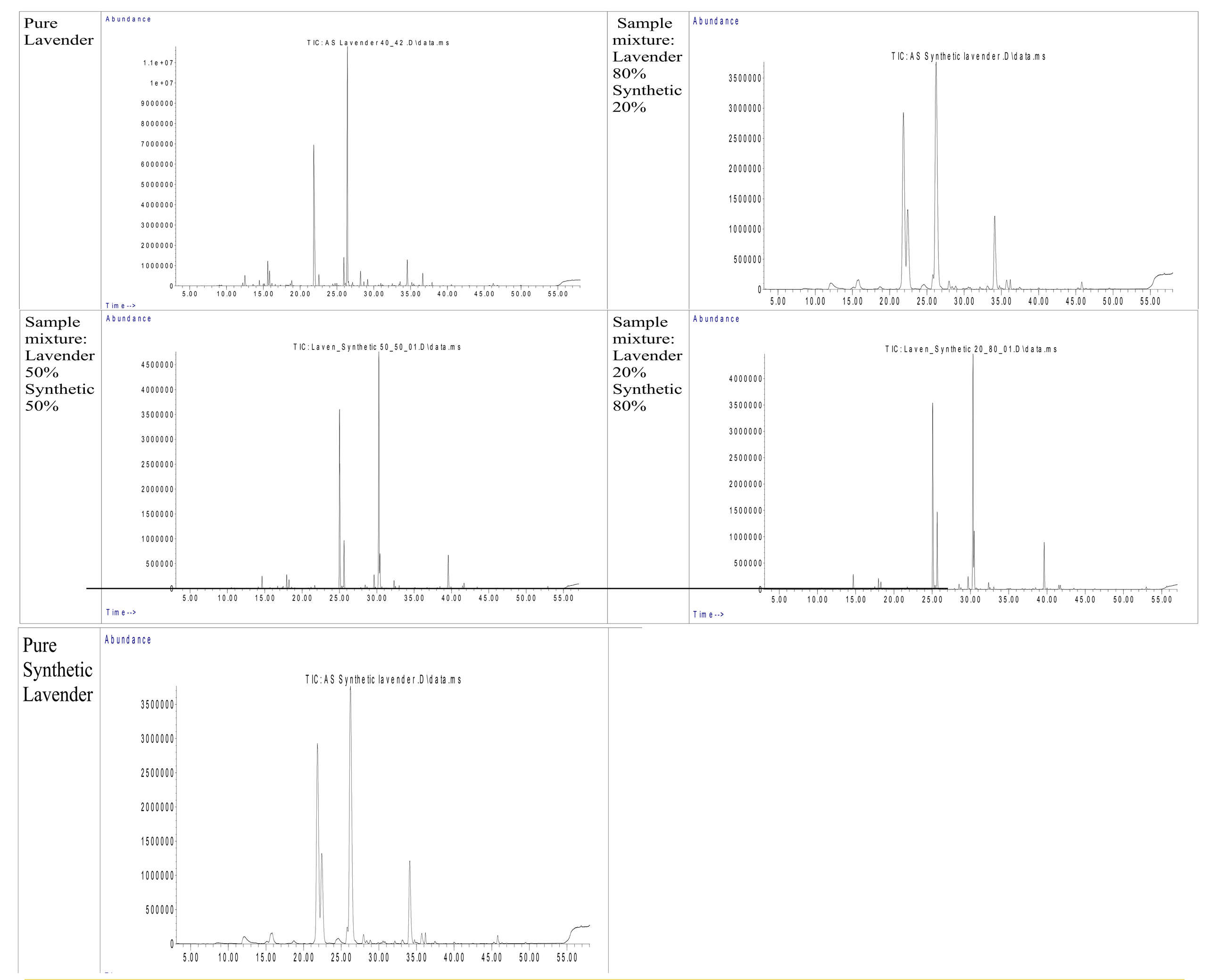
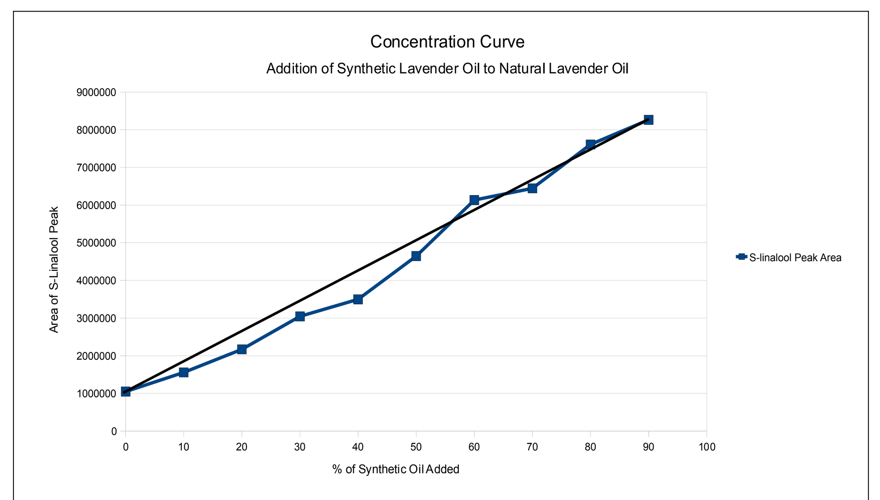


Figure 4. Concentration Curve to Determine the Amount of S Linalool in a Mixture Containing Synthetic Lavender oil



Amount (%) of Synthetic Lavender = {The peak area of S linalool in sample – 6.48×10^5 } / 8.43×10^4

Table 1. Lavender Feedstocks Are Identified by Their Prominent Chemicals.

Lavender species/ Prominent diagnostic peaks	SIM Window Retention Time (min)	Duration (minutes)	Major Ions	Molecular Ion
Lavender				
Linalool, R enantiomer	25	0.4	71, 93, 55	155
Linalool, S enantiomer	25.6	0.4	71, 93, 55	155
Linalool acetate	26.2	0.4	93, 80, 121	197
Lavender, Synthetic				
Eucalytol	12	1	81, 108, 71	155
Linalool, R enantiomer	25	0.4	71, 93, 55	155
Linalool, S enantiomer	25.6	0.4	71, 93, 55	155
Linalool Acetate	26.2	0.4	93, 69, 121	197
Caryophyllene	34	1	93, 133, 69	205
Lavender, Spike				
Eucalytol	12	1	81, 108, 71	155
Linalool, R enantiomer	25	.04	71, 93, 55	155
Linalool, S enantiomer	25.6	0.4	71, 93, 55	155
Caryophyllene	34	1	93, 133, 69	205
Lavandin				
Eucalytol	12	1	81, 108, 71	155
Borneol	24.6	0.4	95, 110, 69	155
Linalool, R enantiomer	25	.04	71, 93, 55	155
Linalool, S enantiomer	25.6	0.4	71, 93, 55	155
Caryophyllene	34	1	93, 133, 69	205

Experiment 3: Survey of Retail Products and detecting mixtures.

Figure 5: Chromatographic Survey of Retail Aromatherapy Lavender Oil Products

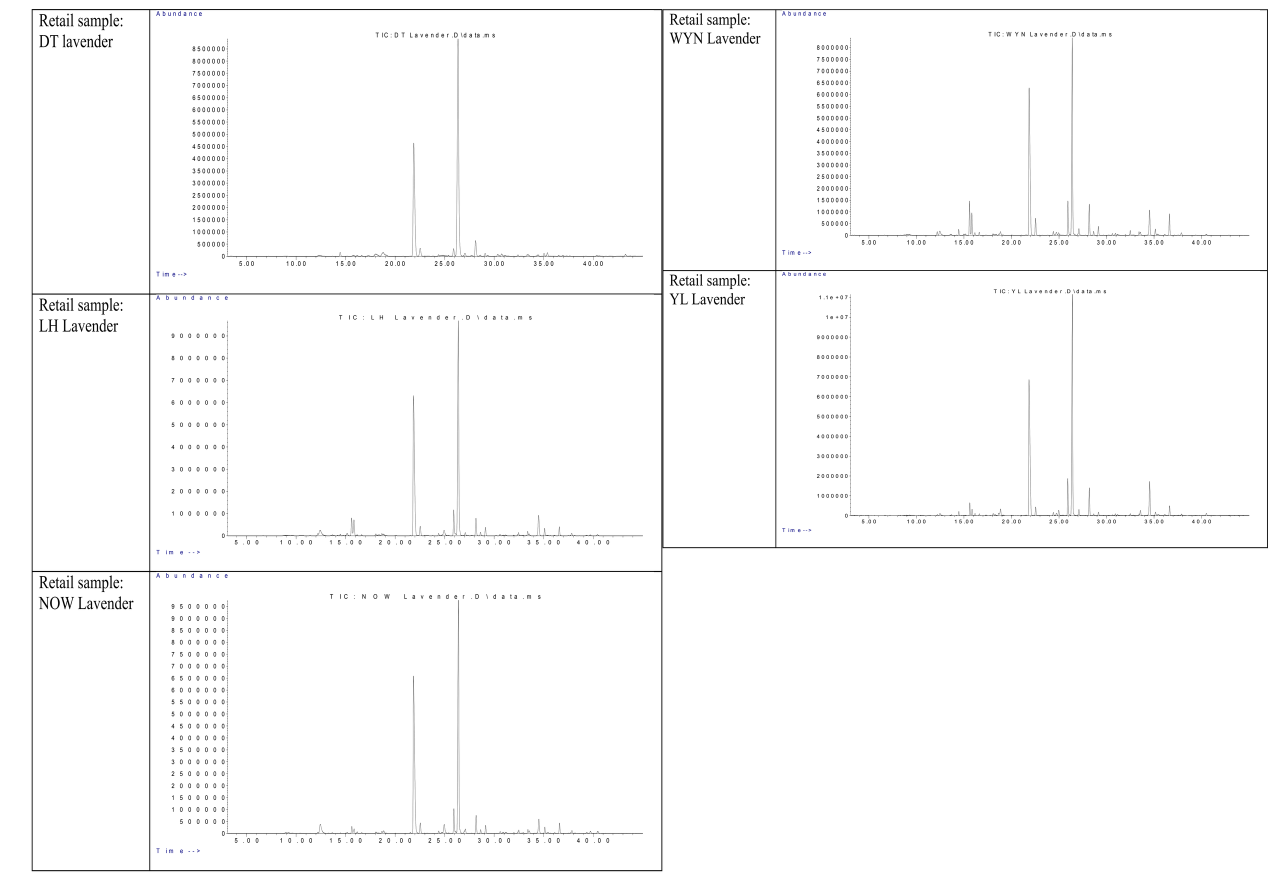
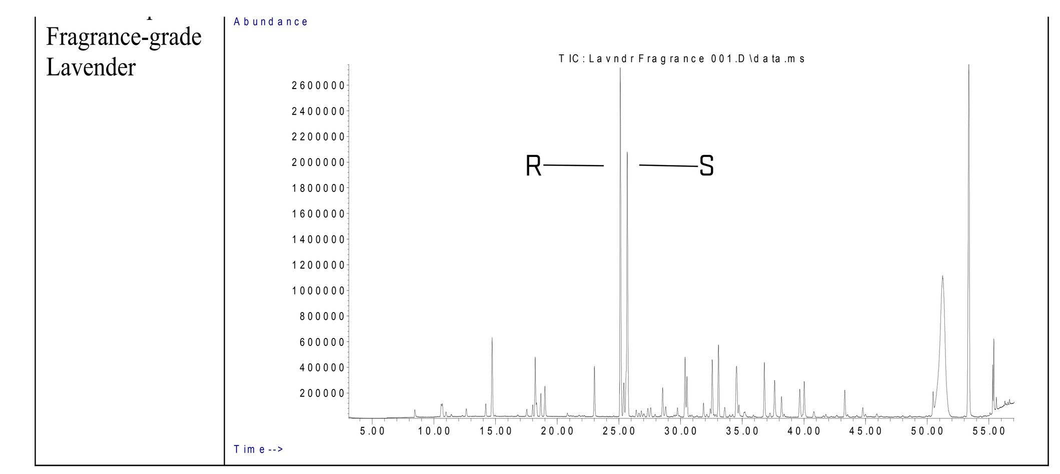


Figure 6: Chromatographic Survey of Retail Fragrance Lavender Oil Product



Discussion

The method resolved the R and S linalool in the retail samples. The integrated ratios for the R and S were close to 95%/5%, well within the accepted values. The sample mixtures also had sufficient peak separation to integrate the peak ratios which were larger than 85%/15%. The method proves useful in determining the enantiomeric ratios needed for detection and the sufficient separation of the peaks to determine the constituents. The chiral chromatograph is especially helpful in separating the specific enantiomers that may be useful in identify the product.

The method was also able to resolve other prominent constituents in the retail oils. The other major constituents in Spike and Lavandin, eucalyptol and borneol, did not appear in large amounts of the retail Lavender officinalis samples.

However, the fragrance grade lavender had a very large R/S linalool ratio indicates that the fragrance-grade oil was synthetic. There were also other constituents of the fragrance lavender that were not common to the aromatherapy grade lavender samples.

Disclaimer

A relationship with the manufacturer or retailers for product promotion, monetary gain or other support for this work did not exist.

http://comps.fotosearch.com/bigcomps/CSP/CSP008/k0088272.jpg

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