‘Passing the Sniff Test’

Analysis of Liquid Contents of MONQ Personal Aromatherapy Vaporisers via Gas Chromatography Mass Spectrometry (GC-MS)

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Abstract

There is evidence of essential oils derived from plants being used by humans since Neanderthal times, with applications ranging from medicinal to food preparation. Traditional application methods for these highly concentrated oils include dermal application and inhalation via vaporisation in large quantities of water via steam diffusion. The increase in popularity of e-cigarettes and vaping in the past several years has led to the development of a novel way of delivering essential oils, which involves suspending them in a carrier liquid (in this case vegetable glycerin) and placing them inside a disposable e-cigarette system for inhalation. MONQ is a market leader in this type of product and to our knowledge no independent laboratory has examined the contents of these pens, or the safety of inhaling aerosolised volatiles such as essential oils. In this study, I analysed liquid from these personal aromatherapy vapes using GC-MS. These samples were compared against pure Essential Oils and the NIST17 database to confirm the presence of terpenes within the liquid. The MONQ company claims their pens contain 80:20 VG: EO blend with each 'puff' delivering 0.003 ml of essential oils directly to the user. Based on preliminary liquid analysis of the contents, this appears to be false, with essential oils appearing in the samples in very small amounts and their presence in the vapour unconfirmed. This study suggests that further quantitative analysis of the vapour and liquid is necessary to determine whether there are any potential negative health consequences associated with these products, which remain unregulated.

1. Introduction

Evidence of humans preparing and utilising essential and fragrant oils can be traced back to prehistoric times, with Neanderthal skeletons (c. 2800 BC) found buried with vials of aromatic oils at sites in what is now modern-day France and Iraq.[1, 2] Ancient Greek, Chinese, Indian and Egyptian civilisations traded, studied, wrote and developed many uses of Essential Oils (EOs) – a significant number of which still hold true and prove effective today.[2-4] When Christianity in the west condemned herbal and aromatherapies as paganism, all ancient texts and writings on the subject were transported to Syria, where the practice continued to grow and thrive. By the third century, Alexandria was considered the mecca of aromatic herbalism. By the ninth century apothecary shops were common and herbal and aromatic remedies were sold in pill, tincture, suppository, poultice, and inhalable forms.[2] The invention of the first modern distillation device, the Alembic, was developed by Mary the Jewess during this period.[5] Aromatherapy continues to be used widely today. The advent of social media, emergence of alternative wellness and influencer culture and multi-level marketing EO companies such as doTERRA® (Figure 1)[6] have led to something of a renaissance for the ancient art of aromatherapy.

Figure 1 A range of oils sold by Essential Oil company doTERRA®

EOs are concentrated, volatile, aromatic liquids extracted from botanical material. The main active components of EOs are secondary metabolites, mainly terpenes or terpenoids which give plants their individual scent profile.[2, 3, 7] Terpenoids (or isoprenoids) make up a significant number (up to 60% of known natural products) of lipids, comprising of up to five isoprene units arranged in thousands of different ways.[8] For the purposes of nomenclature, a terpene consists of hydrocarbons only while a terpenoid compound contains at least one oxygen.

Monoterpenes, a subcategory of terpenes of formula $C_{10}H_{16}$, make up most of the common terpenes in EOs, and are simple molecules based on different combinations of two isoprene units (Figure 2).[1] These secondary metabolites are often specific to the plant, unique in chemical composition and are responsible for a variety of non-essential physiological functions within the plant.[2, 3, 7] Some of these have also been shown to possess antifungal or anti-insecticidal properties, others attract pollinators to ensure ongoing species survival and some have healing properties for the plant itself.[2, 3, 7] The practice of using these oils in a therapeutic sense is known as aromatherapy and the scientific evidence of their efficacy varies for the treatment of various pathologies.[9]

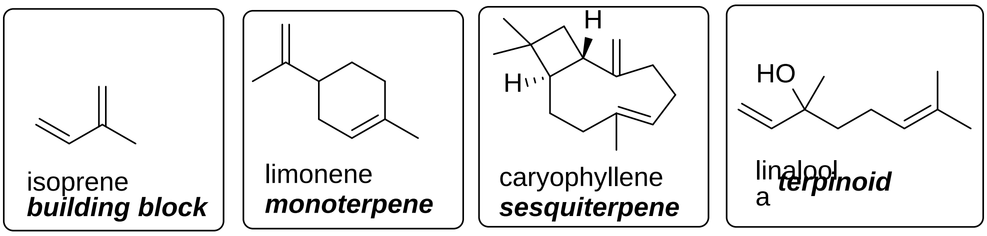


Figure 2 Structures of isoprene, the building blocks of terpenes and some common terpenes and terpenoids seen in essential oils

It is proposed that these compounds are formed in the plant cytoplasm and exist as tiny droplets between cells.[1, 3] Methods of extracting EOs depend on which part of the plant is used. Flowers, roots, bark, wood and whole plants are often extracted by steam distillation while peel, fruit and seed oils are more frequently cold pressed to extract the EOs.[1, 3] Different varieties of oils are named according to their botanical source.[3] The terpene content, chemical composition and purity of the EO will vary depending on the geographical location where the plant material was grown, the method of extraction, and the treatment of the oil post extraction (i.e. any adulterations or dilutions made).[1, 2, 10]

The most common application method of EOs, both historically and currently, is topical where the volatiles are readily absorbed through the skin due to the lipophilic nature of the monoterpenes.[1, 7] In massage therapy for instance, pure oils, such as lavender or ylang ylang are diluted with a carrier oil (fractionated coconut oil is popular) and applied, where both the scent experienced, and absorbance of oils through the skin are considered to have concurrent therapeutic effects.[2, 11] Some oils, such as Tea Tree (*Melaleuca Alternifolia*) and Eucalyptus (*Myrtaceae*) have proven antibacterial properties [12] and are often used in cleaning products, balsams and antiseptic treatments. A blend of Lavender oil, patented under the name Silexan®[13] has been identified as being potentially beneficial in the management of mild anxiety and depression via ingestion and is now sold therapeutically for this purpose. Humidifiers are another popular way of practicing aromatherapy. When used in humidifiers a few drops of EOs are typically added to a large volume of water and vaporised, usually ultrasonically and without heat (though historically through heating a coil to produce steam) (Figure 3). The water vapour produced is scented with the oil and has demonstrated relief in some circumstances from dryness and congestion caused by the common cold, in the case of Eucalyptus,[14] as well as providing a pleasant aroma.

Figure 3 Example of essential oil cold vapour diffuser, where a few drops of EOs are placed into a large tank of water and aerosolised via ultrasonic vibration https://bit.ly/34Be4YD

Electronic cigarettes have become increasingly popular in recent years,[15] either as a healthier, alternative nicotine delivery method to traditional combustible cigarettes, due to the reduced number and concentration of toxic chemicals the users are exposed to, or as a delivery method for other drugs, including cannabis for medicinal purposes.[16] Recently, several companies have brought to market vaping devices, engineered in a similar way to electronic cigarettes (e-cigarettes) containing essential oils and marketing them as 'portable aromatherapy' devices (Figure 4). These portable aromatherapy vape pens are engineered to work in the same way as their e-cigarette counterparts which work by heating a coil, resulting on the vaporisation of the e-liquid they contain, followed by inhaling of the aerosol by the users into the lungs where it is delivered to the blood stream. Although e-cig liquids usually consist of carrier fluids (mainly vegetable glycerine (VG) and/or propylene glycol (PG)), flavouring with or without nicotine, there is only limited information about the content of the vaporisation liquid contained in these aromatherapy vaping devices. There has not been, to the best of our knowledge, any independent scientific research studies to date conducted on the content of these novel aromatherapy vape pens.

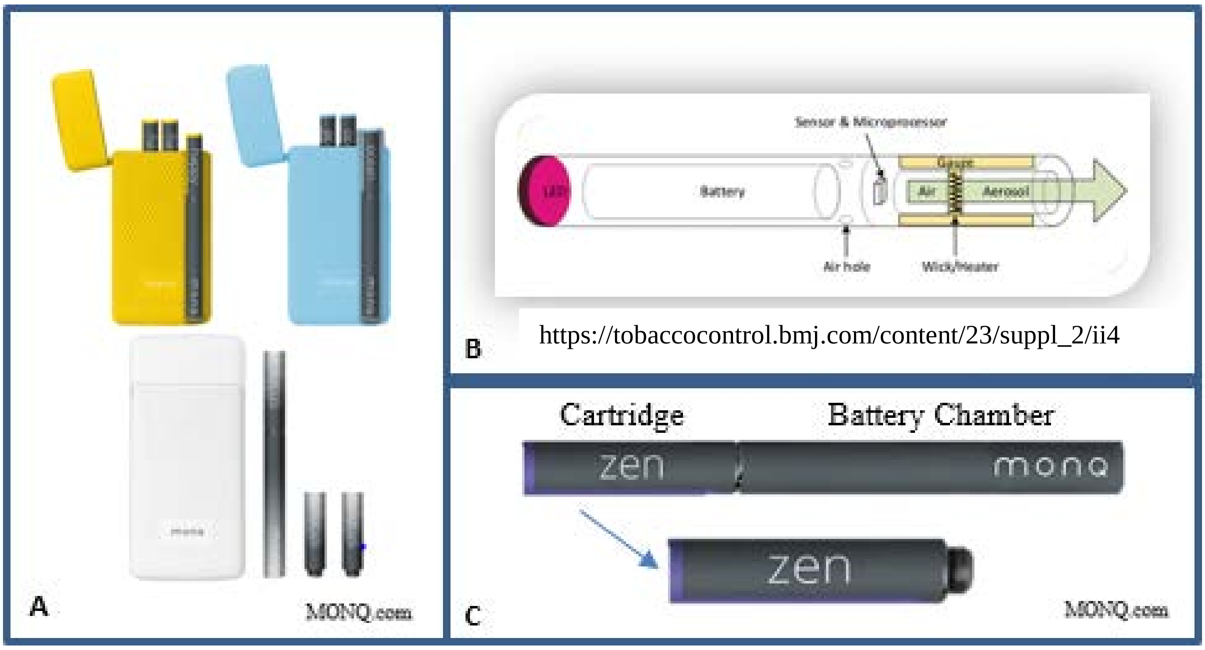


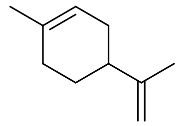
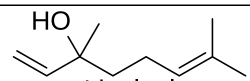
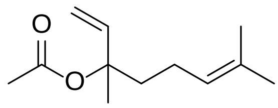
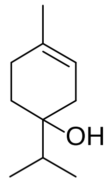
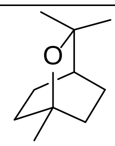
Figure 4 (A) Series of marketing photos of the MONQ R series aromatherapy vape pens; (B) Schematic of a ciga-like vape pen, the design used by MONQ pens; (C) Example of MONQ R assembled device and an individual cartridge.

A market leader in this niche area of portable aromatherapy pens, is MONQ, a Floridian based American company founded by Dr Eric Fishman MD a former orthopaedic surgeon turned aromatherapist and alternative health guru. MONQ is an acronym for “Modifying One’s Natural Qualia”,[17] with Qualia being noted to mean what it is like to be in a mental state caused by external stimuli.[17] Dr Fishman has identified a condition he calls ‘*Terpene Deficiency Syndrome*’ (TDS) which he defines as a cluster of symptoms brought on by the body not receiving the terpenes it needs to maintain proper homeostasis. Dr Fishman promotes the portable aromatherapy devices as delivering ***Therapeutic Air*** ®; the ‘*paleo air breathed by our ancestors*’ and that this ability to ‘*breathe nature anywhere*’ can correct the homeostatic imbalance in the body.[17, 18] The recommended method of inhalation using these devices is that of ‘retro sinus breathing’ or “MONQ breath” which is described as breathing without inhaling into the lungs and exhaling through the nose (Figure 5).[19] The MONQ company claims that the inhalation of terpenes through the nose, activates olfactory receptors which then use neurotransmission to affect human physiology.[19] While there is some evidence suggesting that this way of breathing physiologically prevents volatiles being inhaled directly into the lungs themselves,[20] this is, of course, dependent on the user correctly performing this breathing technique when using these devices.

Figure 5 *Instructions from the MONQ website on how to practice olfactory breathing (i.e. not inhaling directly into the lungs) when using the personal aromatherapy devices; source: https://bit.ly/3cbv382*

Peer reviewed evidence does exist to support particular EOs exerting observable therapeutic properties (Table 1), however there are also contraindications and safety warnings associated with their use.[9] The toxicity/safety data profile of most concentrated pure EOs is at least moderate, with most significant risks associated with skin irritation, phytotoxicity and, relevantly, respiratory toxicity.[7, 9, 21] Eucalyptus oil in doses as small as 2-3 ml has proven fatal to children when ingested and there have been noted cases of chemical pneumonitis, a serious lung infection, upon aspiration of concentrated oils.[22] Oils can often be adulterated with synthetic additives, such as artificial scents or solvents in order to produce greater yields at lower cost.[23, 24] There is little regulation within the industry with respect to purity or standardisation of essential oils, except those which purport to treat a medical illness or exert a medical property.[1, 25] This is problematic in many respects due to the prominence of EO’s for use in food, beverage, cosmetics industries [1, 23] and now vaping devices.

Table 1: Confirmed therapeutic effects induced by some active terpenes in essential oils adapted from the literature[3]

Essential Oil	**Primary Active Terpene**	**Therapeutic Effect**	**Structure**
Lemon (*Citrus limonium)*	Limonene (*Hydrocarbon*)[26]	Immune boosting, metabolic regulation, antimicrobial, antiviral, digestive tonic, purgative.[27-29]	 D-Limonene
Lavender *(Lavandula officinalis)*	Linalool (*Alcohol)* [11] Linalyl Acetate (*Ester*)[22, 23]	Anti-anxiolytic, sleep aid, calmative, can improve symptoms of mild anxiety and depression. [13, 28]	 Linalool  Linalyl Acetate
Tea Tree (*Melaleuca alternifolia)*	Terpinene-4-ol	Antimicrobial, antiviral, neurotonic, radioprotective, antispasmodic, antiasthenic, lymphatic, decongestant.[28, 29] [13]	 Terpinen-4-ol
Eucalyptus *(Eucalyptus globulus)*	1,8-cineole (Eucalyptol)	Anti-catarrhal, expectorant, antiviral and mucolytic antimicrobial.[14, 30, 31]	 1,8-Cineole

While the use of EO’s can be traced back to ancient time, no popular modern method of application has involved the inhalation of heated, aerosolised EOs potentially directly into the lungs, where based on their known lipophilicity would rapidly and extensively exchanged across the pulmonary capillaries and into the blood stream.[6] Thus, there is a public health imperative to undertake an analysis into the content and composition of the material within these portable aromatherapy devices, as well as their potential safety and suitability for inhalation via vaporisation.

This research project will qualitatively analyse the content of these novel vaping devices for the delivery of essential oils with the following aims:

- Confirm the identity and number of the terpenes and terpenoids present in selected MONQ blends (N=8) using Gas Chromatography Mass Spectrometry (GC-MS) and compare to the NIST17 database.
- Compare the identified terpenes/terpenoids to a set of pure unadulterated essential oils (N= 16) against literature and MONQ data to confirm terpene profiles of EOs listed in the ingredients.
- Qualify the terpenes in each sample and determine the potential toxicity risk profile associated with the inhalation of volatile EOs into the lungs from existing literature.
- Identify the composition of the carrier fluid in MONQ blends and compare to the limited information provided by the company.

2. Methods and Materials

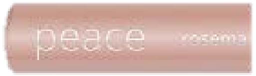
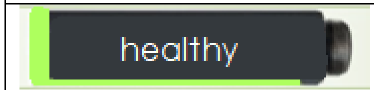
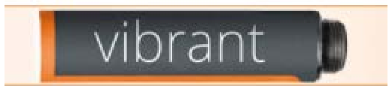
2.1. Materials

A selection of donated essential oils were obtained for comparative purposes and were either of the *doTERRA*®, *In Essence* brand (which is distributed by Heritage Brands, Australia), *Oil Garden Aromatherapy* or *Thursday Plantation* brands (See Table 2). MONQ Aromatherapy pens were purchased from the MONQ Australia online store [32] in February 2021. The blends selected for this study were 'Vibrant', 'Relieve', 'Forest', 'Peace', 'Healthy', 'Mountain', 'Focus' and 'Zen'. The contents of each blend as stated on the product/website are detailed in Table 3. The company has since removed 'Healthy' from its product range and reformulated 'Forest' but this study uses the 'Forest' formulation produced in 2020. LC-MS grade methanol, hexane and dichloromethane were sourced from Sigma Aldrich, Australia (Castle Hill), as was the quinoline. All water used in experiments was MilliQ.

Table 2 Essential Oils analysed including the extraction method and ingredients according to the manufacturer's specifications

Brand	**Oil**	**Extraction Method**	**Blend Ingredients as per Manufacturer**
doTERRA®	Cypress	Steam Distillation	Pure
doTERRA®	Grapefruit	Cold Press	Pure
OilGarden Aromatherapy	Basil	Steam Distilled	Pure
doTERRA®	Lemon	Cold Press	Pure
doTERRA®	Spearmint	Steam Distillation	Pure
doTERRA®	Lemon Eucalyptus	Steam Distillation	Pure
Thursday Plantation	Tea Tree	Steam Distilled	Pure
doTERRA®	Eucalyptus	Steam Distillation	Pure
doTERRA®	Lavender Peace	Various - Blend	Lavandula Augustrifolia (Lavender Oil), Juniperus Virginiana (Cedarwood) Wood Oil, Cinnamomum Camphora Linalooliferum (Ho Wood) Leaf Oil, Capanga Odorata (Ylang Ylang, Origanum Majorana (Marjoram) Leaf Oil, Anthemis Nobilis (Roman Chamomile) Flower Oil, Vetiveria Zizanoides (Vetiver) Root Oil, Vanilla Planifolia (Vanilla Bean) Extract, Santalum Paniculatum (Hawaiian Sandalwood) Wood Oil
In Essence	Sleep	Various - Blend	Lavender Angustifolia (Lavender) Herb Top Flowering (697 uL), Citrus Reticulata (Mandarin) Fruit Peel (227 uL), Chamaemelum Nobile (Roman Chamomile)Flower (30 uL), Valerinana Officialis (Valerian) Root (1 uL)
In Essence	Stress	Various - Blend	Citrus sinensis (Orange) Fruit Peel (300mg); Citrus Reticulata (Mandarin) Fruit Peel (200 mg); Pelargonium Graveolens (Geranium) Leaf 200mg, Pogostemon Cablin (Patchouli) Leaf (200mg); Citrus Aurantium (Bergamot) Fruit Peel (100 mg) [Per Ml of EO]
In Essence	Balance	Various – Blend	Citrus Sinesnsis (Orange) Fruit Peel (796.5 mg), Lavandula Angustifolia (Lavender) Herb Top Flowering (115 mg), Pelargonium Graveolens (Geranium) Leaf (70 mg), Jasminium Officialnale (Jasmine) Flower 1.8 mg), Rosa X Damascena (Rose) Flower (0.7mg)
doTERRA®	Citrus Bliss	Various - Blend	Citrus Aurantium Dulcis (Wild Orange) Peel Oil, Citrus Paradisi (Grapefruit Peel) oil, Lavandula Angustifolia (Lavender) Flower Oil, Michela Alba (Magnolia) Flower Oil, Anthernis Nobils (Roman Chamomile) Flower Oil.

Table 3 Names and listed ingredients of MONQ blends analysed as part of the study

MONQ BLEND	Main Oils Listed in Blend	Other Ingredients Listed
	Rosemary Frankincense Yellow Mandarin	Clary Sage, Frankincense Carterii, Lemon, Mandarin Petitgrain, Narrow Leaf Eucalyptus, Orange, Peppermint, Spearmint, Tea Tree & Organic USP Glycerin
	Ginger Helichrysum Spikenard	Bergamot, Black Pepper, Copaiba, Frankincense, Geranium, Jasmine, Lavender, Nerolina, Ravensara, Roman Chamomile & Organic USP Glycerin.
	Cinnamon Leaf Marjoram Turmeric	Basil, caraway, copaiba, dill seed, green mandarin and lemon (This blend is no longer sold by the company.)
	Juniper Leaf Peppermint Scotch Pine	No additional ingredients listed by the company
	Frankincense Sweet Orange Ylang Ylang	Black Pepper, Clary Sage, Dill Seed, Eucalyptus, Lemon, Rosemary, Sage, Spearmint Tea Tree & Organic USP Glycerin.
	Lemon Ginger Mandarin	Basil, Bergamot, Bitter Orange, Black Pepper, Caraway, Clary Sage, Copaiba, Dill Seed, Fennel, Geranium, Hyssop, Oregano, Parsley Seed, Peppermint, Rosemary, Sage, Sweet Orange & Organic USP Glycerin.
	Marketed as nootropic with no primary oils listed on ingredients.	Bergamot, Black Pepper, Caraway. Cardamom, Cinnamon Leaf, Clary Sage, Coffee, Frankincense, Ginger, Lemon Eucalyptus, Nutmeg, Rosemary, Spearmint & organic USP Glycerin.
	Frankincense Yellow Mandarin Rosemary	Basil, Bergamot, Bitter Orange, Black Pepper, Caraway, Clary Sage, Copaiba, Dill Seed, Fennel, Geranium, Hyssop, Oregano, Parsley Seed, Peppermint, Rosemary, Sage, Sweet Orange & Organic USP Glycerin.

2.2. Methods

2.2.1. Preparation of Essential Oils for Analysis by GC-MS

Samples were prepared from pure or blended concentrated essential oils (Table 2). Miscibility tests and existing literature indicated hexane to be the most suitable solvent for both the dilution of these oils and their subsequent analysis by GC-MS.[33] Initial dilutions (100 fold) were prepared in hexane in 2 mL microcentrifuge tubes by adding 5µL of the essential oil to 995 µL of hexane and vortexed for one minute. For GC-MS analysis, the oils were further diluted 100 fold (10,000 fold total dilution) in 2 mL GC-MS vials by adding 10 µL of the 100 fold diluted solution and 5 µL of quinoline (internal standard, 1 mg/ml in hexane) to 985 µL of hexane, and vortexed for one minute. The oils were analysed by GC-MS, and compound identities were carried out by matching to the NIST17 GC-MS database and compared against the ingredient information provided by the manufacturer. A threshold of 75% similarity was used to tabulate results, however, only matches above 90% can be considered as providing a confident match.

2.2.2. Preparation and analysis of MONQ aromatherapy vape pen vapour

The proposed vapour analysis method was adapted from a previous honours project within our group, who was analysing the flavour compounds in e-cigarettes.[34] Adaptations and optimisations of the method to fit these new devices included: the number of puffs, type of impinger used and volume of solvent used.[34] Initial testing involved two separate Dreschel bottles connected by inert vinyl tubing being connected to a pump at one end and the MONQ pen at the other (See Figure 6 and 7). Each bottle was filled with 100 ml of Dichloromethane (DCM).

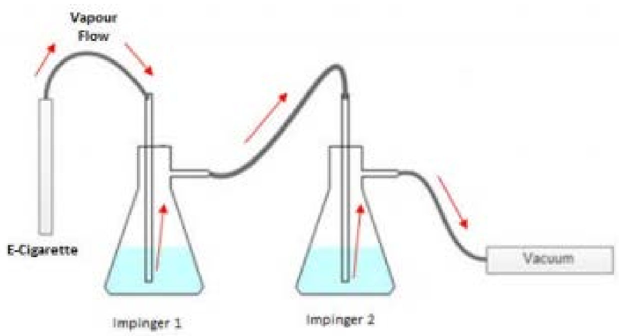


Figure 6 Vapour capture set up designed by former honours student within the research group.[34]

Figure 7 Unsuccessful vapour analysis set ups. Dual impingers (right) were replaced with a direct connection to the auto pump (left) to see if this provided any results.

The pump was controlled by an Arduino computer which was set to 1.1 L/min for 4 seconds per puff with a 25 second break in between puffs in order to allow the coil time to cool.[34] The MONQ Focus pen was used for analysis and the solvent liquid from the primary impinger was collected at both 30 puffs and 50 puffs and analysed for terpenes. None were observed. The method was repeated again using hexane as the impinger solvent, however this did not provide any observable terpenes on analysis.

2.2.3. Preparation of MONQ extracted oil/carrier fluid mix for analysis by GC-MS

Before dilution, all liquids collected from the vape pens were vortexed to ensure complete suspension of oil in carrier fluid for homogeneous sampling as separation of the oil and carrier fluid after extraction and storage could be observed in some instances (see Figure 8). Initial proof of concept testing involved combining pure VG in three different solvents (methanol, hexane and dichloromethane) to assess miscibility (see Figure 9).

Figure 8 Photo showing separation between carrier fluid and essential oil in MONQ Relieve after standing on the bench at room temperature for several days

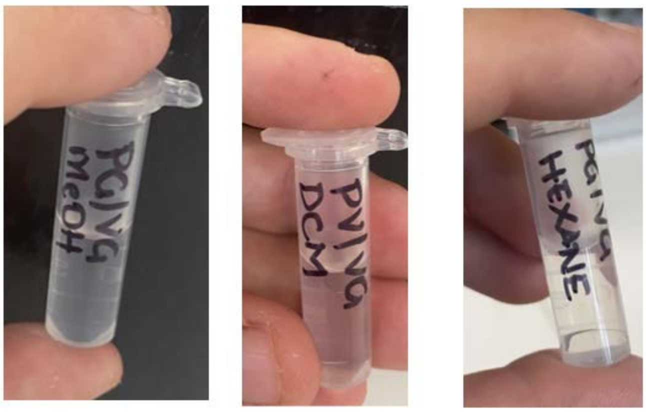


Figure 9 Miscibility tests showing that PG/VG is miscible in DCM and MeOH but not in Hexane.

After miscibility tests, to identify the composition of the carrier fluid, 20 µL of the liquid extracted from the MONQ Focus pen was combined with 200 µL of hexane, and run through the GC-MS instrument using the parameters set out at Table 4. Methanol was used to extract the carrier VG solution as it is miscible with it. The terpenes being lipophilic in character, drawn to the hexane phase, where VG is not miscible, leaving it behind in the methanol and water and allowing isolated extraction of terpenes from the hexane.

The solution required an additional extraction step before GC-MS analysis was performed in order to remove the large and obstructive VG peak, so terpenes present could be analysed and qualified. This involved 10 µL of the MONQ samples (oil/carrier fluid homogenised mix) being diluted into 2 ml microcentrifuge tubes with 200 µL of water, 200 µL of methanol, and 200 µL of hexane. Samples were vortexed for one minute then centrifuged for one minute (13.2k rpm), until a distinct phase separation could be observed. The top hexane layer was removed using a manual micropipette. The hexane extraction process was repeated once more and both hexane layers combined. From this combined hexane extraction, 100 µL was transferred to a 0.1 ml glass insert inside a 2 mL GC-MS vial for analysis. All extractions for each sample were carried out in duplicate. An extraction method blank was also prepared. To extract the liquid from the soaked fibre and wick extracted from the vape pen, these were

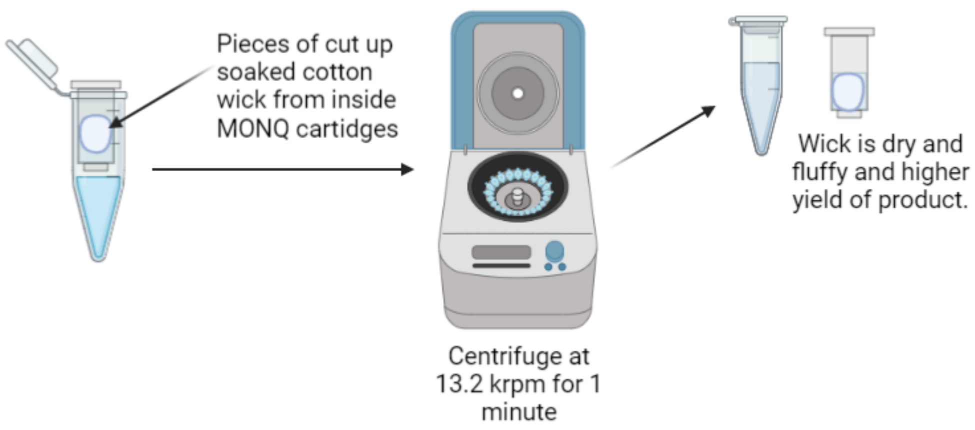


Figure 10 In house microcentrifuge tube setup to extract liquid from vape pen cotton and wick. The soaked fibre is placed inside a small tube from which the bottom has been cut off with a scalpel. These two elements are placed inside a larger collecting Eppendorf flask and centrifuged for one minute at 13.2krpm for one minute

transferred to an in house apparatus as described in Figure 10 and centrifuged (Eppendorf 5415 D model) for one minute at 13.2k rpm.

2.2.4. GC-MS Analysis method

The following method was employed for all analysis performed on both the EOs and the MONQ liquids solutions as prepared above. The instrument used for all analysis was a Shimandzu QP2020 GCMS. This method was developed by another student in our research group for analysing terpenes found in various types of cannabis. The parameters for the GC-MS instrument are listed below in Table 4.

Table 4 The Parameters of the Shimadzu QP2020 mass spectrometer used in analysing the terpene content of essential oils and MONQ vape pen liquid samples

Parameters	Method
Column type	SH-Rxi-5sil-MS
Column Dimensions	0.25µm x 30.0m x 0.25mm
Helium Flow Rate	1ml / min
Oven Program	60°C, 2 min hold + 10°C min to 110°C, + 40°C min to 300°C, hold for 5 min
Total Runtime	16.75
Split Ratio Mode	10:1
Injection Volume	1 µL
Solvent Delay	3 min
Total Ion Scan	40 – 500 m/z, 3-16.75 min
Ion Source Temp	270°C
Interface	300°C

3. Results and Discussion

3.1. Original vapour analysis method preparation

The original aim of this study was to analyse the aerosolised product, produced by drawing on the MONQ vape pens. The initial method was based on a vapour capture method developed by this research group.[34] However, trials conducted involving capture in a variety of solvents (including hexane and dichloromethane) showed no evidence of the terpenes being extracted into the solvent in their aerosolised form (See Section 2.2.2 and Figure 6 and 7 above). Time constraints and a limited amount of sample product forced this method to be abandoned.

3.2. Extraction of the liquid content from of MONQ portable aromatherapy pens

As no independent study has ever been conducted on these vape pens an analysis of the liquids they contained was carried out. MONQ cartridges are disposable vape pens, as such these are not meant to be refilled, and so access to the liquid they contained implied that the device had to be forcefully opened. Figure 11 shows an unopened and opened cartridge and its contents. When opened, it was observed that all of the liquid in the cartridge was soaked into a cotton wick, rather than exposing the e-liquid directly to the coil. This design has been identified by our research group as being the primary method used by newer disposable-style e-cigarette devices which have exploded in popularity within the last twelve months. To extract the liquid from the soaked fibre and wick extracted from the vape pen, these were transferred to an in house apparatus as described in Figure 11 and centrifuged (Eppendorf 5415 D model) for one minute at 13.2k rpm. The oils/carrier fluid mixes obtained from the vape pens were stored in the microcentrifuge tube in which they were collected at room temperature on the bench.

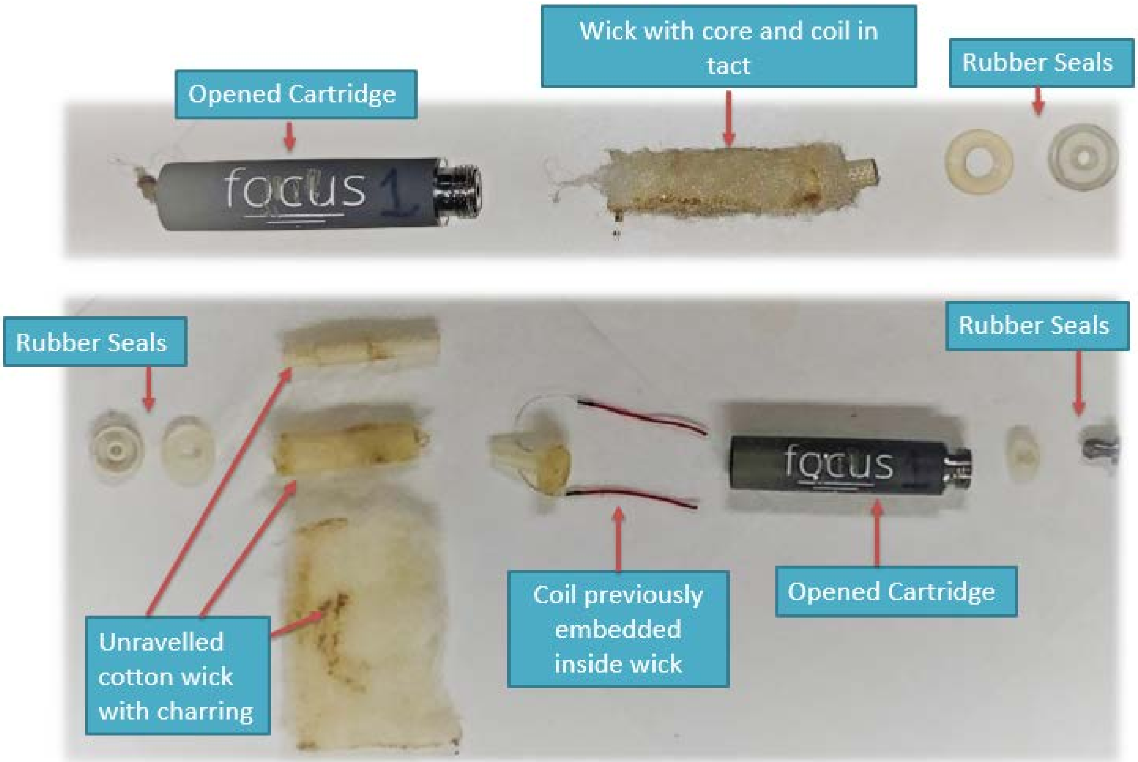


Figure 11 Picture of closed and opened MONQ R cartridge after it had been used in formative vapour analysis but before extraction of the remaining liquid, note the discolouration and charring of the cotton wick.

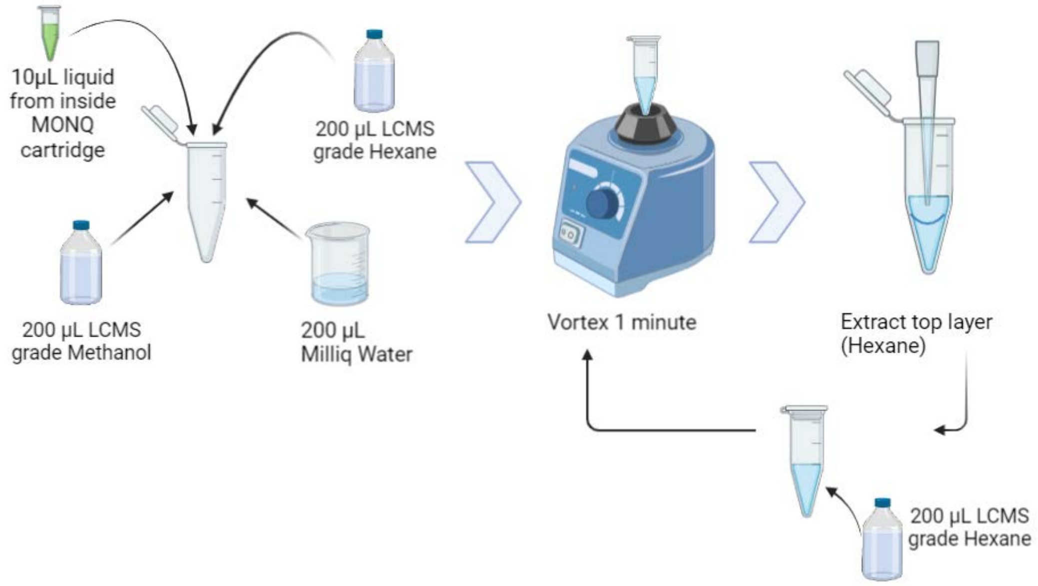


Figure 12 Steps in preparing the two step hexane extraction to remove the VG from the sample and analyse terpenes in MONQ liquids

3.3. Analysis of Carrier fluid using GC-MS

In order to determine the composition of the carrier fluid in the MONQ vape pens, 20 µL of the liquid extracted from the MONQ Focus pen was combined with 200 µL of Methanol and run through the GC-MS (using the same parameters as those set out in Table 4), where a distinct strong VG peak was detected at an elution time of around 6-8 minutes, but no other common carrier fluid peak such as PG (elution time of ~ 3 minutes under these conditions) or additives like nicotine, (estimated elution time of approximately 8.5 minutes) were observed. [35] This VG peak is consistent with the elution times of a VG standard using this method, thus it can be concluded that the MONQ companies' claims that their products only contain VG as a carrier fluid are accurate.

3.4. Analysis of Pure Essential Oils by GC-MS

Full tabulated analysis and the chromatograms for each EO analysed by GC-MS can be found in Appendix 1 (N=13). This analysis was based solely on similarity matches in the NIST17 library with all potential peaks with more than 80% correlation tabulated but only matches >90% can be reported with certainty. NIST17 matches are reported in brackets throughout as a % match (xx % match) for the remainder of this paper. The full list of ingredients according to the manufacturer can be found in Table 3. All EOs analysed (both pure and blended) were compared to relevant literature in order to confirm the integrity of the method and provide a comparative basis upon which to analyse the contents of the MONQ vapour pens. Where possible, literature profiling of the geographically specific species of the botanical has been used for comparative purposes. Where a geographic location was not provided comparison was made based on species alone. The specific part of the botanical used to obtain the oil was consistent in comparison between the literature and the samples analysed.

3.4.1. Cypress

According to the doTERRA website[36] the oil for this product is obtained from the *cupressus sempervirens* and *lusitanica* varieties of the plant, this is relevant as different strains of Cypress trees can produce different terpenes based on their geographical location.[37] The literature indicates the high levels of α-pinene and δ-3-carene are to be expected here,[38] however not only does the analysis not show any δ-3-carene present, it shows a significant peak with a high level of similarity to *cis*-β-ocimene (95% match). A number of terpenes (including terpinolene, β-myrcene and β-pinene) in smaller abundance were detected all of which correspond with the literature.[38] Also present, but not mentioned in the literature, were trace amounts of cedrol, caryophyllene, terpin-4-ol and sabienen.

3.4.2. Grapefruit – cold pressed peels

Comparative analysis of the Grapefruit oil was unremarkable, with the collected data corresponding with that in the literature.[39] D-limonene (95% match) and β-myrcene (87% match) both eluting as expected with a relatively high degree of certainty. The literature indicates there are a number of trace terpenes [39] that were not seen in this data set and this is possibly due to not having a low enough limit of detection on the instrument or variation in the extraction method employed. Alternatively, the dilution factor selected for analysis (10,000:1) may be too high to see the terpenes present in lower abundance.

3.4.3. Basil Oil – steam distilled flower

Analysis of the basil oil was clear and unambiguous, with two clear elution peaks with high levels of similarity. These are identified as linalool (94% match) and estragol (96% match) and are confirmed by the literature as being the primary components of basil EO derived from the flower of the plant [40]. P-cymene was identified in the literature as a secondary component but this was not observed in this data.[40]

3.4.4. Lemon Oil – cold pressed peels

The strongest elution peaks in this sample correlate with D-limonene (95% match) and γ-terpene (93% match), more moderate peaks were observed for β-pinene (95% match) and β-myrcene (85% match). Trace amounts of α-pinene (89% match) and sabinen (82% match) are also present, with a low match elution of caryophyllene (65% match). When compared with the literature, this correlates quite well, with only neral and geranial identified as being expected to be present but not observed in this analysis.[41] Figure 13 shows the chromatogram derived from a 1:10,000 of doTERRA® Lemon essential oil with the main elution peaks identified.

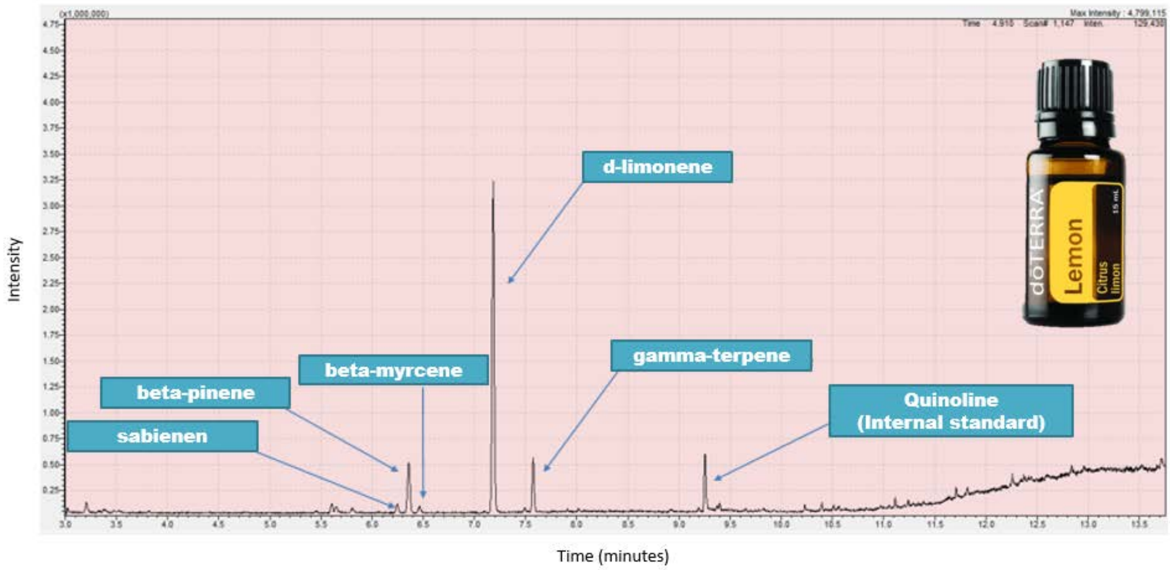


Figure 13 GC-MS chromatogram of doTERRA Lemon essential oil 1:10,000 dilution with observed eluted terpenes labelled according to similarity with NIST17 database

3.4.5. Spearmint Oil – steam distilled leaves, flowers and stems

Analysis of the Spearmint essential oil showed expected strong elution peaks for D-limonene and carvone. Evidence from the literature [42] identifies a number of other oxygenated monoterpenes found to be present, such as 1,8-cineole, with this data supporting that, showing elution of both Eucalyptol (86% match) and Cineole (86% match) peaks. With respect to the class of sesquiterpene hydrocarbons, there are low threshold matches for caryophyllene (73% match) in the data set only. The appearance of α-bourbonene (86% match) is a novel observation which does not appear in the literature.

3.4.6. Lemon Eucalyptus – steam distilled from leaves and flowers

Terpenes citronellal and citronellol are expected to be the most concentrated in this sample, in this respect the data in this instance corresponds with the literature.[43] Isopulegol and isocaryophyllene also show in low concentrations but with high levels of similarity, (92% and 87% matches respectively). The literature [43] indicates the presence of a number of additional terpenes (including α-terpineol, geraniol and aromandendrene) which were not seen in this analysis, possibly either due to the underdeveloped method or alternatively, they are not present in this oil.

3.4.7. Tea Tree – steam distilled from leaves

Tea tree is widely accepted to have antimicrobial and antifungal properties[44] and it has been the subject of much scientific research. The presence of a strong elution peak for terpinen-4-ol is in concurrence with the literature [44] which identifies this, along with γ-terpinene, α-terpinene, 1,8-cineole and α-pinene as being present in highest concentrations. This corresponds with the data set in this instance with very good matches for α-pinene (91% match), γ-terpinene (94% match), 1-8-cineole (93% match) and α–terpineol (95% match).

3.4.8. Eucalyptus – steam distilled leaves

Eucalyptus oil is one of the more widely used essential oils, it is also one of the most toxic, with doses of less than 2ml causing death in children when ingested.[22] There are many varieties of Eucalyptus tree, with each differing slightly in their terpene profile.[45] The doTERRA brand used in this study is from the *Eucalyptus radiata* species,[46] which has been found to contain over 72 identifiable terpenes,[45] however the GC-MS method used by that study involved a total run time of over 60 minutes. Relevantly, the data collected in this study shows the four most common terpenes eluted for this species, limonene (94% match), α–terpineol (96% match), α-terpinyl acetate (84% match) and α-pinene (90% match). Trace levels of a variety of other terpenes correlating with the literature were also detected, however a significant number do not appear in this study. This is likely due to their presence in low abundance, which makes them undetectable either due to the Level of Detection (LOD) used in this method being too high to see them or they have co-eluted with more abundant terpenes which has caused them to become obscured in the chromatogram (see Appendix 1).

3.4.9. Lavender Peace – blend; various

Main correlative peaks observed include linalool (95% match), linalyl acetate (95% match), terpinen-4-ol (91% match), cis-thujopsene (94% match), cedrol (91% match) – all of which are known terpene compounds of the ingredients listed. A variety of smaller peaks appear which correlate with additional expected terpenes, but with lower NIST correlations (below 80% match) and can be found at Appendix 1. Despite the inclusion of vanilla bean in the ingredients, there is no observable peak for vanillin nor is there a discernible carvone peak which would be expected given the inclusion of vetiver oil. However the literature indicates [47] that elution of the primarily sequiterpenes in vetiver oil via GC-MS requires a different flow rate and temperature than the method used to elute all of the terpenes contained within.[47]

3.4.10. Sleep Blend – various

The three main peaks identified in this instance are D-limonene (95% match), citronellol (90% match) and linalyl acetate (93% match). The D-limonene and linalyl acetate are the main expected terpenes found in mandarin oil [48] and lavender oil [49] respectively. Despite the presence of roman chamomile in the ingredients list, no chamazulene or bisabolol were detected in this sample.[50] Also absent from this data set are patchouli alcohol and α-bulnesene which the literature indicates should be present given the inclusion of valerian root.[51]

3.4.11. Stress Blend – various

The strongest elution peak corresponds to d-limonene (95% match) which is consistent with the primary ingredients being as orange and mandarin EO as well as bergamot. Citronellol (85% match), citronellyl formate (formic acid) (85% match) could be attributed to the geranium oil, but there is a conspicuous lack of geraniol, which according to the literature should be present in large quantities.[52] The presence of linalyl acetate (92% match) and linalool (83% match) can be attributed to the bergamot, as can the caryophyllene (86% match).[53] Patchouli accounts for the alpha-guaiene (91% match) and the patchouli alcohol (90% match).[54] The presence of azulene (93% match) is novel, not being seen in the literature and its origin is unclear. Small peaks corresponding to β-myrcene (86% match) were also observed and accounted for in the literature.[52, 55, 56]

3.4.12. Balance Blend – various

The strongest elution peak is once again D-limonene (95% match), as orange oil is the primary ingredient this is to be expected.[56] The linalool and linalyl acetate peaks showed relatively high correlation with the library and can be attributed to the inclusion of lavender oil [11] and jasmine.[57] Geranium is again listed as an ingredient but there is not an elution peak for geraniol, however the citronellol peak (90% match) can likely be attributed to the geranium and the inclusion of rose oil. The literature indicates that due to the inclusion of jasmine EO an 1 H indole [57] should be seen but is not.[57] The inclusion of rose oil accounts for the α-pinene (88% match), β-myrcene (85% match) and β-ocimene (83% match) elution peaks, however a number of other expected terpenes are not seen in this study, including neral, geranyl acetate and farnesol.[58]

3.4.13. Citrus Bliss – various

This sample had a very intense peak for D-limonene (95% match), which is conducive with the main citrus blends included here (orange and grapefruit) [56] and is consistent with previously observed elution times for D-limonene using this method. Limonene is also one of the primary terpenes found in magnolia flower oil, which is also an ingredient here.[59] The linalool and linalyl acetate present can be attributed to the inclusion of lavender, though the library matches are on the low side (81% and 86% match respectively) they are high enough for us to make an assumption this is likely correct based on ingredients.[49] The inclusion of roman chamomile would indicate that there should be visible elution peaks for α-bisabolol, azulene and chamazulene [50] however none of these were observed in this dataset.

4. GC-MS analysis of MONQ Aromatherapy Vape Pens

The MONQ website claims that their pens contain a ratio of carrier fluid (VG) to essential oil of 80:20 and that each 'therapeutic breath' contains approximately 0.003 ml of Essential Oils.[60] Analysis of the liquid inside the pens aimed to capture the terpenes in the hexane solvent and extract these for analysis, leaving the VG carrier fluid, and its obtrusive peak, (retention time covering an elution period also corresponding to the elution of some terpenes) in the methanol: water layer.

4.1. Peace

The first elution of a terpene in this sample was isoborneol (92% match), the literature indicates this is a terpene present in rosemary, however identifies, 1,8-cineole, camphor and caryophyllene being also present in high concentrations and none of those are seen in this data set.[61] The absence of any peak for d-limonene is curious as analysis of oils containing citrus usually feature this elution prominently, according to the literature, [56, 62] and the analysis of the pure oils discussed above. However, all other terpenes eluted in this data set (See Appendix 2, Table 1) have been observed in varying trace amounts in literature analysing the yellow mandarin.[63] The primary terpenes expected to be observed from the inclusion of frankincense include α-pinene, α-thujene, β-pinene, myrcene, p-cymene, sabienen, limonene and β-caryophyllene.[64] None of these were observed in this data set. The terpenes which were observed, could be attributable to any of the ingredients listed, as most contain at least trace amounts of these terpenes in their overall composition.[21]

4.2. Relieve

The three primary EO ingredients listed for the Relieve Blend on the MONQ website are ginger, helichrysum and spikenard. Based on the literature observable amounts of zingiberene, ar-cucumene, β-bisabolene and β-sesquiphellandrene should be present due to the ginger,[65] these are not observed in the dataset. The inclusion of helichrysum as a primary EO should show peaks correlating with α-pinene, 1,8-cineole, p-cymene, though these are not observed in this dataset, however elutions of caryophyllene (93% match) and carophyllene oxide (89 % match) could be attributable to this ingredient.[66] Spikenard is a unique ingredient, not analysed in any of the previous blends or pure essential oils. The literature indicates its composition is mainly calarene, β-maaliene, valerena-4,7(11)diene, aristolene, γ-terpinene and seychellene.[67] None of these are observed in this dataset.

Interestingly, it is the secondary ingredients which account for the vast majority of the observable peaks in this sample. Linalool is a primary terpene in lavender EO,[49] and bergamot [53], though limonene is not observable, so it is unlikely bergamot is present in any high amounts here.[53] The presence of observable elutions for citronellol (89% match), and geraniol (90% match) indicate the presence of geranium oil in the blend.[52] No α-bisabolol elution was observed despite the blend listing roman chamomile as being present.[50] The presence of caryophyllene (93% match) in the sample could be attributable to the inclusion of copaiba, which the literature indicates is its primary terpene component.[68] The inclusion of nerolina accounts for the nerolidol (87% match) and nerolidyl acetate (80% match) [69] and also potentially for a portion of the linalool (74% match), and α-terpineol (85% match) observed. The inclusion of black pepper EO could also account for the caryophyllene seen in this sample, as per the literature, however, as in previous instances, other expected terpenes such as sabienen, β-myrcene and p-ocimene should have been observed and they were not.[70] Overall, this data indicates that the essential oils listed in the ingredients are not present in detectable amounts in the samples tested here, or they are not adequately captured by this extraction method.

4.3. Healthy

The primary EO ingredients listed in the MONQ Healthy blend are cinnamon leaf, marjoram and turmeric. Eugenol is one of the primary terpenes seen in both cinnamon leaf and marjoram, [71, 72] thus its absence here is notable. Cinnamon leaf is also known to contain detectable levels of benzyl benzoate (>3%),[72] which is seen eluted here (78% match), though with a low match threshold. Cinnamon leaf is also a source of alloaromadendrene,[73] whose derivative aromandendrene (78% match) is seen eluted here, though with relatively low similarity. Marjoram EO could account for the linalyl acetate (84% match) and terpinene-4-ol (90% match),[71] with linalool (80% match) also being attributable to the cinnamon leaf.[72] Turmeric is a pungent and coloured essential oil, with its main terpene component being aR-tumerone, which appears distinctly in this dataset with a high level of similarity (90% match). Turmeric is also likely responsible for the presence of curlone (88% match).[74] All three main EOs contain varying amounts of β-caryophyllene according to the literature,[71, 72, 75] and this could account for the two distinct elutions in this data set (at 10.187 and 10.217 minutes) both with high degrees of similarity to the library (95% and 93% match, respectively). The most likely source of the carvone (91% match) and aceteugenol (90% match) is the green mandarin in the secondary ingredients list.[56, 63] Caraway EO also contains high levels of carvone however the absence of any observable limonene elution makes the presence of caraway EO impossible to confirm.[76] This could also account for the presence of linalool (80% match), α-terpineol (87% match) and terpinen-4-ol (90% match).[63] The data doesn't show any peaks for estragol or p-cymene as would be expected with the inclusion of basil EO, as confirmed in the EO analysis discussed above and in the literature.[40] The elution of copaene (79% match), despite being a low threshold match suggests the presence of copaiba EO as does the caryophyllene (95% match) and caryophyllene oxide (90% match).[68].

4.4. Mountain

The MONQ Mountain blend diverges most significantly from expectations of the pure oils based on listed ingredients. The three main ingredients listed in the Mountain blend are; Juniper leaf, Scotch Pine and Peppermint,[77] and there are no secondary additional ingredients listed. The primary elutions expected to be seen from those three ingredients would include α-thujene, α-pinene, sabienen, myrcene and α-terpinene with respect to the juniper leaf.[78] With respect to the Scotch pine, strong peaks corresponding to α-pinene, carene and bornyl acid should be seen.[37] In the case of peppermint, strong elution peaks for menthol, menthone, 1,8 cineole, limonene and myrcene should have been observed.[79] The strong correlations in this sample with α-terpineol (94% match), eugenol (92% match) and d-carvone (94% match) may correspond to the inclusion of the peppermint, however again the absence of limonene draws its presence into question. Strong correlative library matches with α-curcumin (88% match), aR-tumerol (89% match), aR-tumerone (91% match) and curlone (92% match) indicate the presence of turmeric, if we are to be guided by the literature and what has so far been observed in this study.[75] Juniper leaf has been found to contain a number of the terpenes eluted, albeit in low concentrations.[78] However, it's possible that the terpinene-4-ol (NIST=94), linalyl acetate (NIST=90) and caryophyllene (NIST=96) could be accounted for by the Juniper leaf. It is possible that there is some degree of instrument / column contamination in this particular sample as there were a number of operational errors with the GC-MS around this time and the Mountain samples followed the Healthy blend samples sequentially in both repetitions of the run. A full character table for this sample can be found at Appendix 2, Table 4

4.5. Vibrant

The Vibrant blend, according to MONQ has three primary EO ingredients; Ginger, Lemon and Mandarin. Immediately, from previous samples analysed we can identify that limonene should be a prominent terpene given the presence of two citrus varieties. The inclusion of Ginger EO, as discussed above for the Relieve blend, should give strong peaks for zingiberene, ar-cucumene, β-bisabolene and β-sesquiphellandrene.[65] However, none of those appear in this data. A number of alkanes appeared in this sample, which are also observable at a lower intensity in the hexane method blank. A possible reason for this could be related to potential contamination caused by performing the hexane extractions in plastic lab-ware. Other members of the research group encountered the same problem, these alkane peaks disappeared when other students' samples were retested after being prepared in glass vials. Time prevented this process from being repeated here. The most prominent terpene peak identified in this instance, which also has the highest degree of library correlation is carvacrol (85% match). Other terpenes that could be identified here but with low correlations with the NIST17 library, include citronellol (77% match) and nerolidol (77% match). Given the potent nature of the primary essential oils reported as being a part of this mix and the extensive list of additional ingredients (See Table 3 in Section 2), it was expected that more terpenes would be observed.

4.6. Focus

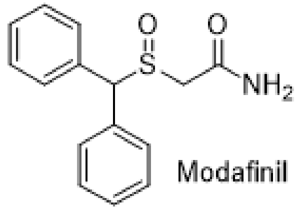


Figure 14 Chemical structure of pharmaceutical CNS 'Nootropic' drug Modafinil

The Focus blend is marketed differently from the other samples in the MONQ range. It is marketed as a nootropic. Nootropics, colloquially known as “smart drugs” consist of a wide range of chemical compounds with a variety of biological functions which purport to improve memory and cognitive function in users, via CNS modulation instead of traditional CNS stimulation methods.[80] Nootropic blends range from herbal supplements (containing wildly variable ingredients), with some blends containing ingredients such as cat’s claw, oat straw and amino acids such as L-thymine and L-tyrosine, to actual synthesised pharmaceutical drugs, the most well-known being Modafinil (Figure 14). The MONQ company conducted a study in their own private lab – assessing the ability of the Focus blend to improve cognitive skills and reaction times in users.[81, 82] The results of these tests were all overwhelmingly positive (See Figure 15). The control used was Adderall, a stimulant medication used to treat Attention Deficit Hyperactivity Disorder (ADHD). However, there has been no formal publication of this study in any journal, with the data being presented as a series of slides on the MONQ website, which form part of their patent application.[81] Based on this study, they filed and have a paper pending that seeks to patent the formula within the Focus pens as a therapeutic aid that may be used to treat neurodegeneration, potentially restoring cognitive function in those with neurodegenerative diseases such as Alzheimer’s.[82]

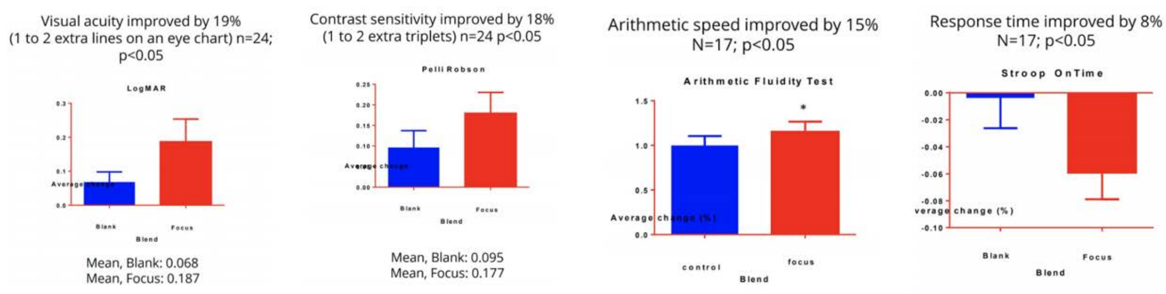


Figure 15 Examples of improved cognitive function results from MONQ's patent application presentation regarding FOCUS blend available at https://bit.ly/3fJaEsY

The GC-MS analysis of the Focus blend shows extremely low levels of essential oils being present, this is in concurrence with all of the samples analysed above. Certainly far less than the 80:20 ratio of carrier fluid to oil provided by the MONQ company. There are no primary ingredients identified in this sample, see Table 3 in Section 2 for a full list of ingredients. The strongest correlative match for this sample relates to d-carvone (91% match) which follows an initial (though slightly less well matched) elution (82% match) also matching to d-carvone. The presence of carvone is likely related to the bergamot, though once again no limonene is observed, which makes confirmation difficult. Almost any of these ingredients could have produced the trace amounts of dihydrocarveol (79% match), carvone (91% and 82% matches; two distinct elution peaks) and aceteugenol (83% match).

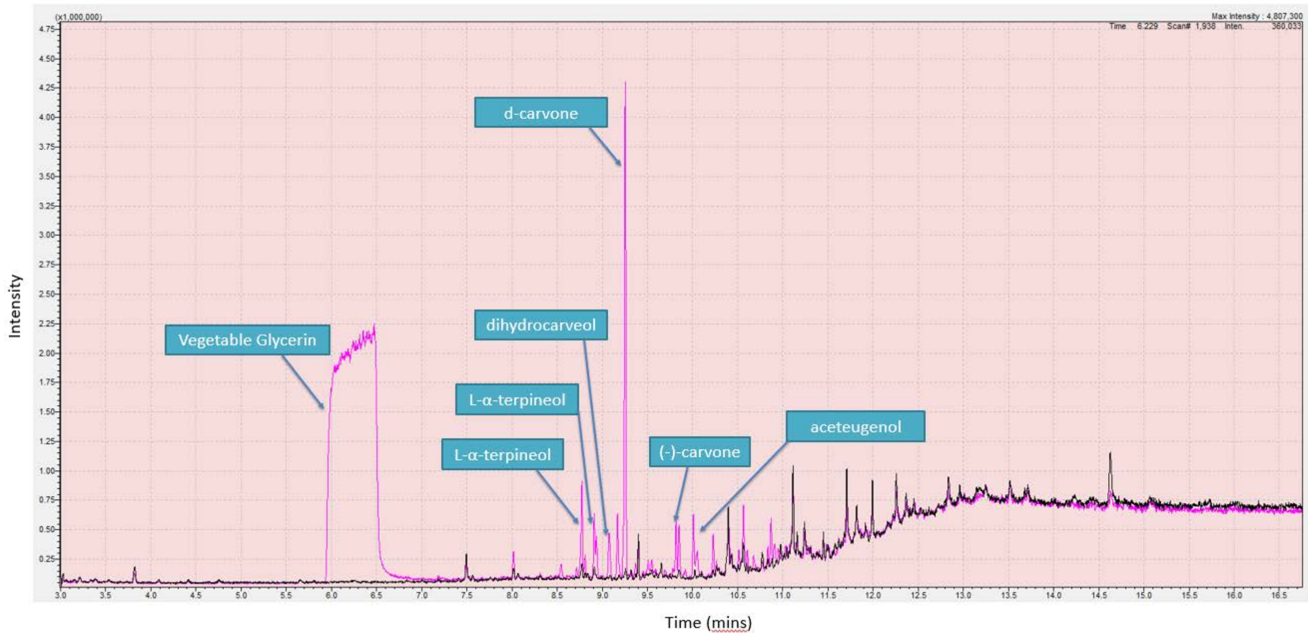


Figure 16 GC-MS chromatogram showing MONQ FOCUS liquid analysis. Pink line shows run without removal of VG via extraction with identified terpene elution peaks labelled

4.7. Forest

Despite several repetitions of analysis of the Forest blend sample by GC-MS, using varying levels of dilution, no terpenes were detected in the sample (See Appendix 2, Figure 7 for chromatogram). There could be several reasons for this, the contents of this particular cartridge sample having undetectably low concentrations of the listed essential oils suspended in the carrier fluid, though this seems unlikely given the pungent odour of the sample. It is a possible that for this particular sample, the extraction method used was not sufficient to extract the oils from the carrier fluid. Alternatively, when examining the wick of the dismantled forest cartridge discolouration can be observed (see, Figure 11 above), this indicates the possibility that the terpenes in the oils in this sample have remained embedded in the wick of the device were not extracted with the carrier fluid. This could also be a quality control issue with the product itself and it is unknown if the compounds would vaporise with the carrier fluid or remain embedded in the wick upon heating of the sample.

4.8. Zen

Similarly to the Forest blend, despite several repetitions, and halving the dilution factor the Zen pen liquid eluted no terpenes, only a series of alkanes. It is possible the contents of this sample were not evenly distributed throughout the carrier fluid, the terpenes were stuck in the wick of the product and not extracted, or there was a quality control issue with the product itself, being that the levels of essential oils in the sample were so low they fell below our level of detection for this method. Again the appearance of alkanes indicates some level of contamination from performing the extractions in plastic lab ware as discussed above in Section 4.5 relating to the Vibrant blend. A full character table for the Zen blend can be found in [Appendix 2, Figure 8.]

5. Are they safe to use?

None of the aerosolised products were able to be captured and analysed so we don't know what, if any adducts or pyrolytic by-products may be produced when heating these volatile oils to high temperatures. What can be ascertained from this very preliminary study though, is that there are essential oils in the MONQ liquid, however they are only present in very low quantities. Preliminary analysis suggests that this is far lower than the 80:20 VG to EO advertised by the company,[60] however, this would need to be confirmed by quantification of the terpenes and VG present. This means it is likely each inhalation would contain less than the advertised 0.003 ml of EO.[60] It can also be confirmed that the carrier liquid is pure vegetable glycerine (VG), due to the absence of any PEG or PG peaks being observed in the data. There are some studies that suggest the heating of VG into aerosolised form for inhalation can generate some volatile and potentially toxic aldehydes, [83] however it is generally considered non-toxic.

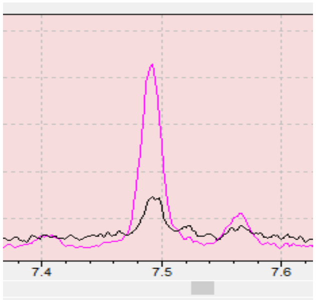


Figure 17 Excerpt from chromatogram showing the hexane method blank (black line) and the sample being run in hexane (pink line) showing a much higher elution peak for an alkane contamination.

Many of the GC-MS methods in the literature differed significantly from the one used in this study, especially with respect to time. Often lower temperatures and longer run times were used as well as much higher detection and quantification parameters than time allowed in this study. Variances in the literature and data obtained could also be attributed to the geographical source of the oil, as discussed above in Section 3. It is likely the oils reacted with the plastic microcentrifuge tubes they were stored in as there are correlations between the method blank alkane peaks and stronger peaks in the samples with similar NIST matches for each when the two are compared (see Figure 17). The highly volatile nature of the oils, combined with their storage at room temperature probably contributed to these unintended reactions/contaminations. Future storage method of samples should aim to store the volatile liquids in a dark place, in dark glassware, under 4°C to prevent unwanted reactions whilst in storage.

6. Conclusions and Future Directions

The range of terpenes expected to be seen in this blend of ingredients and the levels seen based on the company's 80:20 VG:EO ratio, is significantly less than what was seen in this data set. However, it appears from this data that VG is the only carrier fluid used in the fluids, due to the absence of PG or PEG peaks. All MONQ samples are also nicotine free, with no elution peak appearing anywhere in the results. It is unable to be confirmed by this study that the essential oils were actually transported into the vapour phase along with the carrier fluid, however liquid analysis confirms their presence.

Areas of further study for this project should include developing an optimised method for analysing the vapour produced by the MONQ pens. Cell toxicity studies could be conducted if identified compounds could be isolated or synthesised successfully. In future, due consideration should be given to storage, with glassware being preferred to plastic lab ware, especially with respect to storage of the volatile liquids. Future vapour analysis would eliminate this issue due to employing a direct capture method. Quantification of terpenes in the aromatherapy pens would be useful to determine the concentrations in which these terpenes and essential oils appear in the sample. Both of the aforementioned methods would need to be developed and optimised first in order to produce results. The composition of the carrier fluid is also another area that could be explored, with the MONQ company claiming it is sourced from organic coconuts only. Also, the ethical and environmental impact of the non-disposable lithium ion rechargeable batteries in these devices is an area of ongoing concern and research from an environmental perspective.

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Appendix 1

Table 1 Character table recording the elution times and NIST17 library matches for analysis of GC-MS analysis of doTERRA® Cypress pure essential oil

CYPRESS – doTERRA Pure Essential Oil			
Retention Time	***NIST Match***	***NIST ID***	***Comments and Intensity***
2.589; 2.665	93 96	Furan Tetrahydrofuran- 2-5-dimethyl	Known contaminant on the cylinder for this particular stock of hexane
3.152	82	oxetane, 2-methyl-4-propyl	Column contamination
3.035	86	oxetane, 2,2,4-trim,ethyl-	Column contamination
5.485	86	β-thujene	Low
5.594	97	alpha pinene	Very strong
6.273	84	sabinen	Low
6.390	89	beta pinene	Low
6.462	91	beta-myrcene	Low
6.843	95	Trans-beta-ocimene	Medium
7.168	91	limonene	Low
7.570	78	gamma-terpene	Low
7.902	91	terpinolene	Low
8.790	81	terpinen-4-ol	Low
9.232	91	quinaldinic acid	Low
9.853	88	alpha-terpinyl acetate	Low
10.243	84	caryophyllene	Low
10.990	84	cedrol	Low

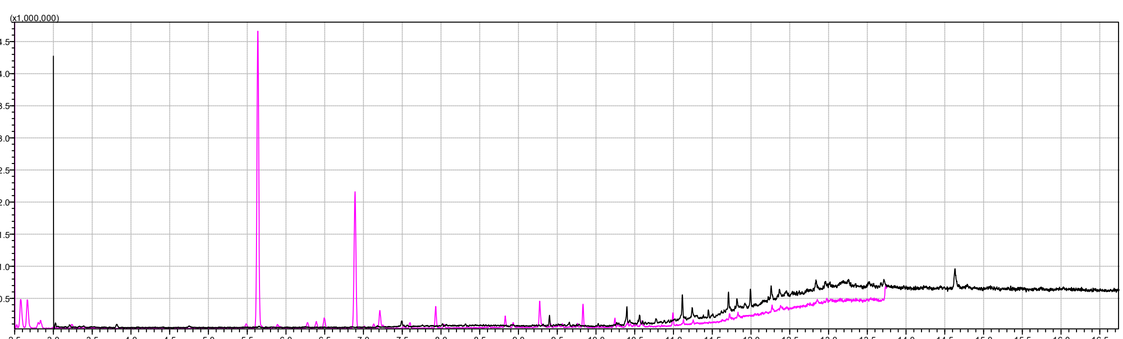

Figure 1 Chromatogram of doTERRA® Cypress pure essential oil.

Table 2 Character table recording the elution times and NIST17 library matches for analysis of GC-MS analysis of doTERRA® Grapefruit pure essential oil

GRAPEFRUIT – doTERRA Pure Essential Oil			
Retention Time	***NIST Match***	***NIST ID***	***Comments and Intensity***
2.589; 2.665	93 96	furan tetrahydrofuran- 2-5-dimethyl	Known contaminant on the cylinder for this particular stock of hexane
6.497	87	beta-myrcene	Low
7.248	95	d-limonene	Very strong

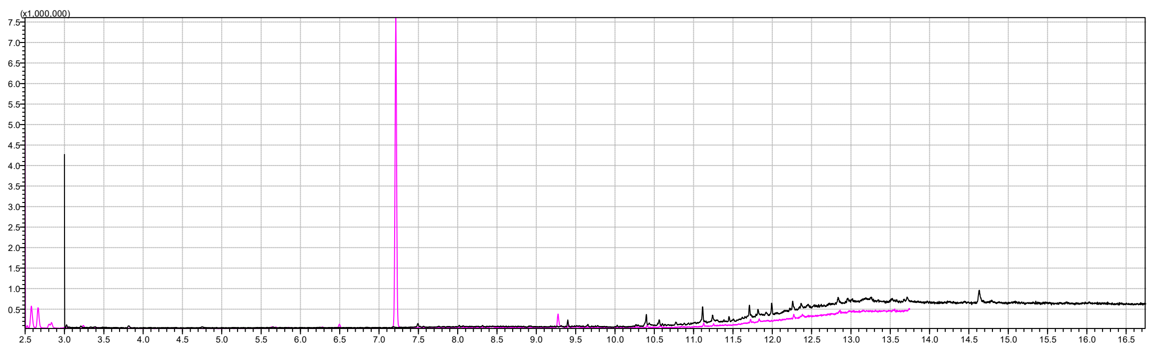

Figure 2 GC-MS chromatogram of doTERRA® Grapefruit essential oil

Table 3 Character table recording the elution times and NIST17 library matches for analysis of GC-MS analysis of Oil Garden Aromatherapy brand basil pure essential oil.

BASIL – Oil Garden Aromatherapy Pure Essential Oil			
Retention Time	***NIST Match***	***NIST ID***	***Comments and Intensity***
7.987	94	linalool	Medium
8.880	96	estragole	Strong
9.25		Quinolone	

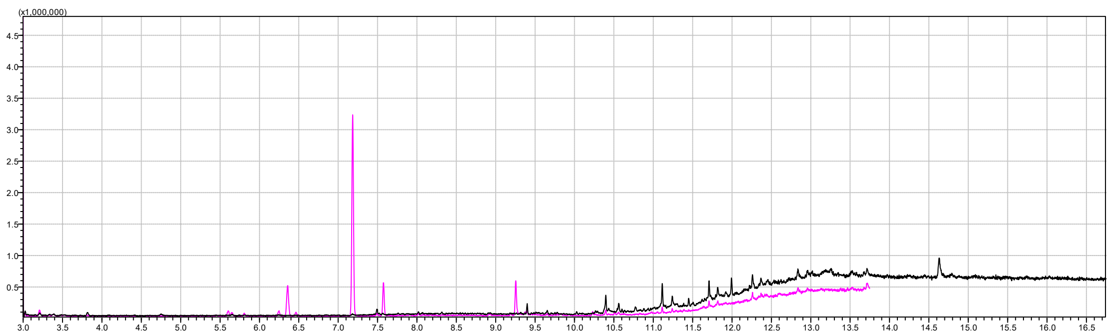

Figure 3 GC-MS chromatogram of Aromatherapy Garden Basil essential oil at a 1:10,000 dilution

Table 4 Character table recording the elution times and NIST17 library matches for analysis of GC-MS analysis of doTERRA® Lemon pure essential oil.

LEMON – doTERRA® Pure Essential Oil			
Retention Time	***NIST Match***	***NIST ID***	***Comments***
5.603	89	alpha pinene	Very low
6.238	82	sabinen	low
6.328	95	beta pinene	Medium
6.452	85	beta myrcene	Medium
7.137	95	d-limonene	Strong
7.547	93	gamma.terpene	Strong
10.227	65	caryophyllene	Low

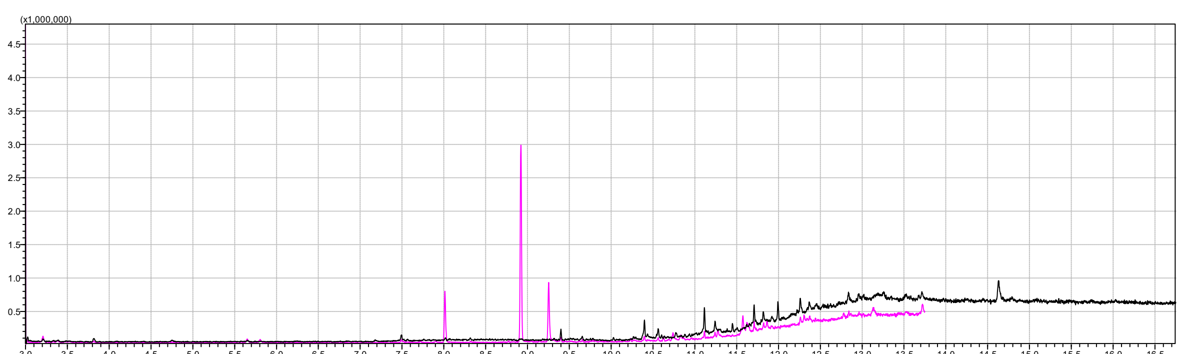

Figure 4 GC-MS chromatogram of doTERRA® pure essential oil at a 1:10,000 dilution

Table 5 Character table recording the elution times and NIST17 library matches for analysis of GC-MS analysis of doTERRA® Spearmint pure essential oil.

SPEARMINT – doTERRA® Pure Essential Oil			
Retention Time	***NIST Match***	***NIST ID***	***Comments***
2.589; 2.665	93 96	furan tetrahydrofuran- 2-5-dimethyl	Known contaminant on the cylinder for this particular stock of hexane
7.247	87	d-limonene	strong
7.273	86	eucalyptol / cineole	Low
8.958	86	dihydrocarvone	Low
9.272	95	(-)-carvone	Very strong
10.085	86	alpha-bourbonene	Low
10.258	73	caryophyllene	low

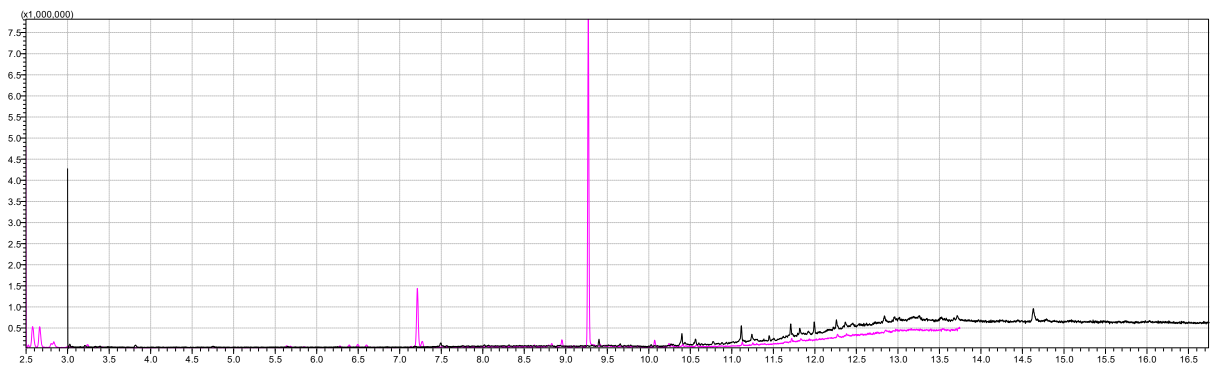

Figure 4 GC-MS chromatogram of doTERRA® Spearmint essential oil at a 1:10,000 dilution

Table 6 Character table recording the elution times and NIST17 library matches for analysis of GC-MS analysis of doTERRA® brand Lemon Eucalyptus pure essential oil.

LEMON EUCALYPTUS – doTERRA® Pure Essential Oil			
Retention Time	***NIST Match***	***NIST ID***	***Comments***
2.589; 2.665	93 96	furan tetrahydrofuran- 2-5-dimethyl	Known contaminant on the cylinder for this particular stock of hexane
8.593	97	citronellal	Very strong
8.653	92	isopulegol	Medium
9.142	86	citronellal	Low
10.242	87	isocaryophyllene	Low
11.803	68	napthalene	low

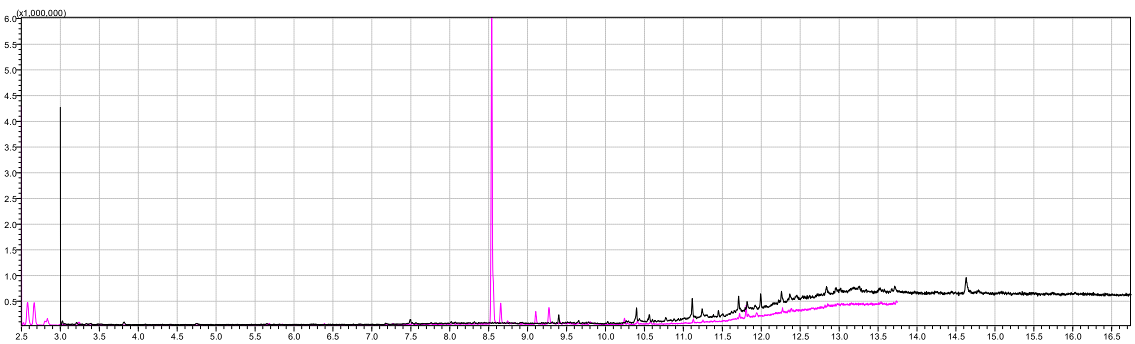

Figure 5 GC-MS chromatogram of doTERRA® Lemon Eucalyptus essential oil at a 1:10,000 dilution

Table 7 Character table recording the elution times and NIST17 library matches for analysis of GC-MS analysis of Thursday Plantation brand Tea Tree pure essential oil

TEA TREE – Living Plantation Brand Pure Essential Oil			
Retention Time	***NIST Match***	***NIST ID***	***Comments***
2.589; 2.665	93 96	furan tetrahydrofuran- 2-5-dimethyl	Known contaminant on the cylinder for this particular stock of hexane
5.487	76	alpha.phellandrene	Low
5.635	91	alpha.pinene	Low
6.393	76	beta-pinene	Low
7.012	88	(+)-4-carene	Low
7.133	95	p-cymene	Medium
7.210	79	limonene	Low
7.720	93	eucalyptol (cineole)	Medium
7.598	94	gamma terpinene	Medium
7.932	86	terpinolene	Low
8.828	97	terpinen-4-ol	Very strong
9.928	95	alpha-terpineol	Medium
10.323	88	aromandendrene	Low
10.523	86	(+)-lendene	Low
10.593	85	napthalene	Low
10.887	71	globulol	low

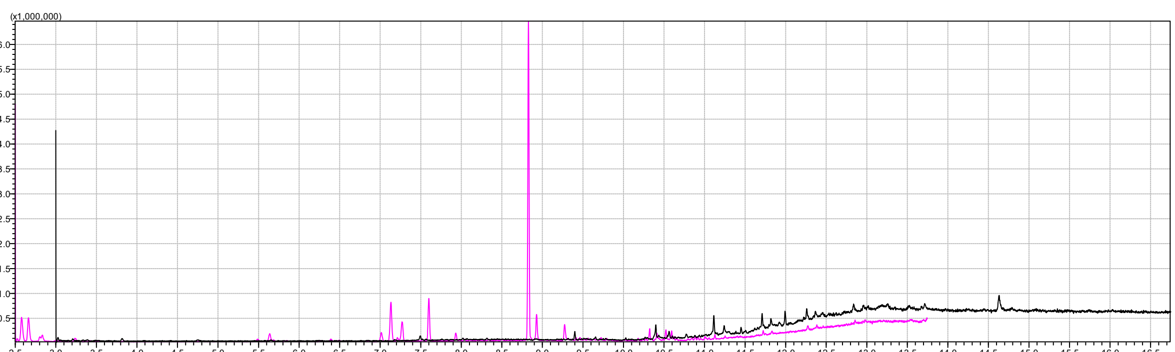

Figure 6 GC-MS chromatogram of Thursday Plantation brand Tee Tree essential oil at a 1:10,000 dilution

Table 8 Character table recording the elution times and NIST17 library matches for analysis of GC-MS analysis of doTERRA® brand Eucalyptus pure essential oil

EUCALYPTUS – doTERRA® Pure Essential Oil			
Retention Time	***NIST Match***	***NIST ID***	***Comments***
2.589; 2.665	93 96	furan tetrahydrofuran- 2-5-dimethyl	Known contaminant on the cylinder for this particular stock of hexane
2.833	84	butanol	Low
3.242	88	2-furanmethanol	Low
5.638	90	alpha-pinene	Low
6.278	79	sabinen	Low
6.388	76	beta pinene	Low
6.503	80	beta myrcene	Low
7.138	87	beta cymene	Low
7.212	94	d-limonene	Low
7.270	93	eucalyptol / cineole	Very strong
8.738	64	trans-linalool oxide	Low
8.828	77	terpinen-4-ol	Low
8.907	96	alpha.terpineol	Medium
9.833	84	alpha.terpinyl acetate	Low
10.242	75	caryophyllene	low

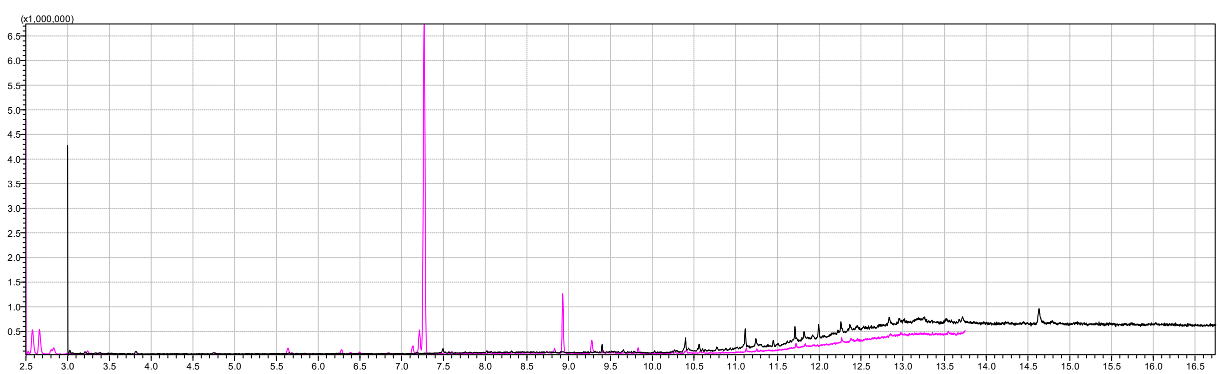

Figure 7 GC-MS chromatogram of doTERRA® Eucalyptus essential oil at a 1:10,000 dilution

Table 9 Character table recording the elution times and NIST17 library matches for analysis of GC-MS analysis of doTERRA® Lavender Peace blended essential oil

LAVENDER PEACE – doTERRA® Blend Essential Oil			
Retention Time	***NIST Match***	***NIST ID***	***Comments***
2.589; 2.665	93 96	furan tetrahydrofuran- 2-5-dimethyl	Known contaminant on the cylinder for this particular stock of hexane
5.638	76	alpha pinene	Low
6.275	74	sabinen	Low
6.495	81	beta-myrcene	Low
7.010	75	(+)-carene	Low
7.208	70	fragranyl acetate	Low
7.248	86	alpha-pinene	Low
7.602	73	gamma-terpinene	Low
8.007	95	linalool	Very Strong
8.090	80	p-menth-8-en-1-ol	Low
8.805	91	terpinen-4-ol	Medium
8.937	77	alpha.terpineol	Low
9.252	95	linalyl acetate	Strong
9.445	89	lavendulyl acetate	Low
10.242	92	di-epi-alpha-cedrene	Medium
10.277	85	(e)-beta. famesene	Medium
10.320	94	cis-thujopsene	Medium
10.480	84	aromandendrene	Low
10.523	73	andrographolide	Low
10.597	75	napthalene	Low
10.998	91	cedrol	Medium

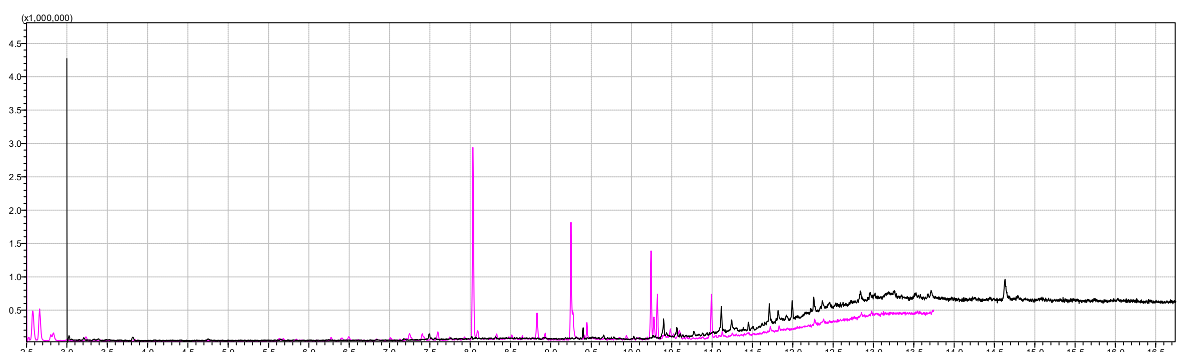

Figure 8 GC-MS chromatogram of doTERRA® Lavender Peace essential oil blend at a 1:10,000 dilution

Table 10 Character table recording the elution times and NIST17 library matches for analysis of GC-MS analysis of doTERRA® Sleep blended essential oil

SLEEP BLEND – In Essence Essential Oil Blend			
Retention Time	***NIST Match***	***NIST ID***	***Comments***
5.638	76	alpha.pinene	Low
5.678	79	hydroperoxide	Low
7.212	94	limonene	Very Strong
7.243	86	alpha.pinene	Low
7.497	74	gamma.terpinene	Low
8.010	95	linalool	Very Strong
8.095	80	p-menth-8-en-1-ol	Low
8.772	60	isoborneol	Low
8.827	93	terpinen-4-ol	Medium
8.934	79	L.alpha.terpineol	Low
9.257	92	linalyl acetate	Very Strong
9.438	79	linalyl isobutyrate	Medium
10.258	92	beta cucumine	Medium
10.278	85	cis.beta.farnesene	Low
10.323	95	cis.thujopsene	Low
10.990	91	cedrol	Low

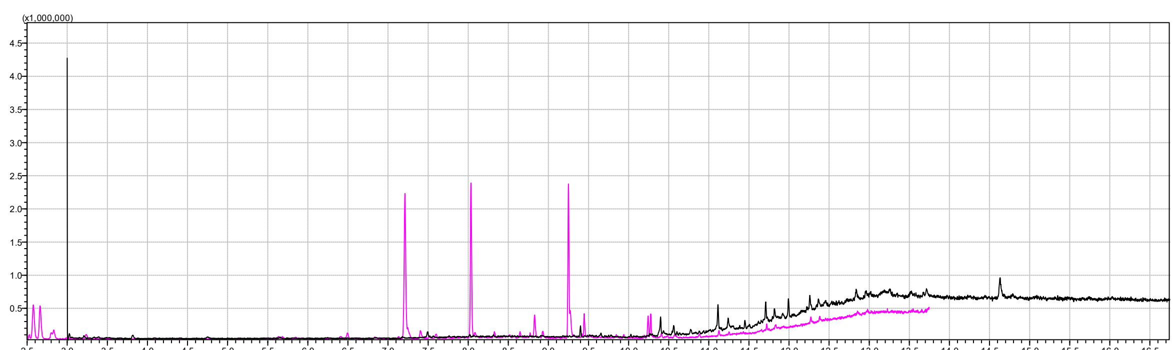

Figure 9 GC-MS chromatogram of doTERRA® Sleep essential oil blend at a 1:10,000 dilution

Table 11 Character table recording the elution times and NIST17 library matches for analysis of GC-MS analysis of doTERRA® Stress blended essential oil

STRESS BLEND - In Essence Essential Oil Blend			
Retention Time	***NIST Match***	***NIST ID***	***Comments***
6.478	86	beta-myrcene	Low
7.152	95	d-limonene	Very Strong
7.577	75	gamma-terpinene	Low
8.015	83	linalool	Medium
9.087	95	citronellol	Medium
9.238	92	linalyl acetate	quinolone
9.382	85	citronellyl formate (formic acid)	Low
10.095	76	4,7-methanoazulene	Low
10.232	86	caryophyllene	Low
10.258	91	alpha.-guaiene	Medium
10.403	81	androsta-1,4-dien-3-one	Medium
10.448	83	1h-3a,7-methanoazulene	Medium
10.525	77	alloaromadendrene	Low
10.547	93	azulene	Medium
11.03	80	2-naphthalenemethanol	Low
11.112	73	nerolidyl propionate	Low
11.200	90	patchouli alcohol	Medium

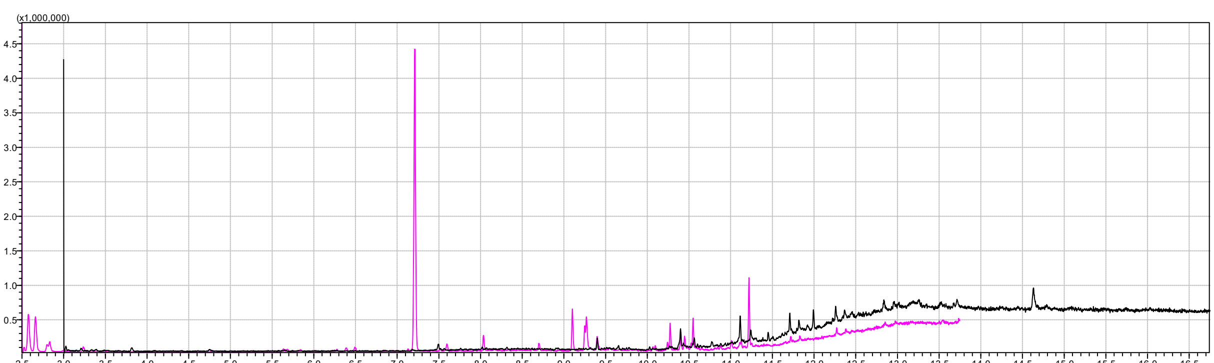

Figure 10 GC-MS chromatogram of doTERRA® Stress blended essential oil blend at a 1:10,000 dilution

Table 12 Character table recording the elution times and NIST17 library matches for analysis of GC-MS analysis of doTERRA® Balance blended essential oil

BALANCE BLEND –Pure Essential Oil Blend			
Retention Time	***NIST Match***	***NIST ID***	***Comments***
5.638	88	alpha pinene	Low
6.462	85	beta myrcene	Low
6.870	83	beta ocimene	Low
7.157	95	d-limonene	Very Strong
8.010	87	linalool	Low
9.092	90	citronellol	Low
9.235	88	linalyl acetate	Medium
10.230	81	caryophyllene	Low

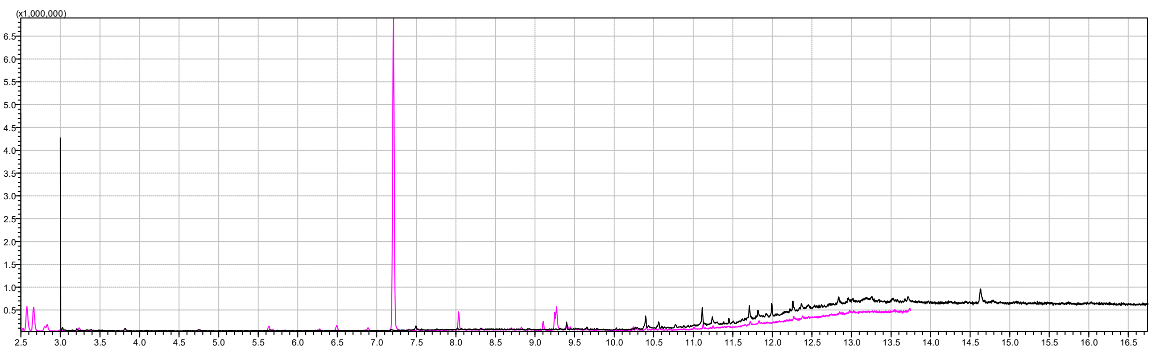

Figure 11 GC-MS chromatogram of In Essence Balance blended essential oil blend at a 1:10,000 dilution

Table 13 Character table recording the elution times and NIST17 library matches for analysis of GC-MS analysis of doTERRA® blended essential oil

CITRUS BLISS BLEND– doTERRA Pure Essential Oil Blend			
Retention Time	***NIST Match***	***NIST ID***	***Comments***
6.457	84	beta myrcene	Low
6.865	66	linalyl isobutyrate	Low
7.153	95	d-limonene	Very Strong
7.983	81	linalool	Medium
9.220	86	linalyl acetate	Eluting with Quinolone
10.253	78	caryophyllene	Low

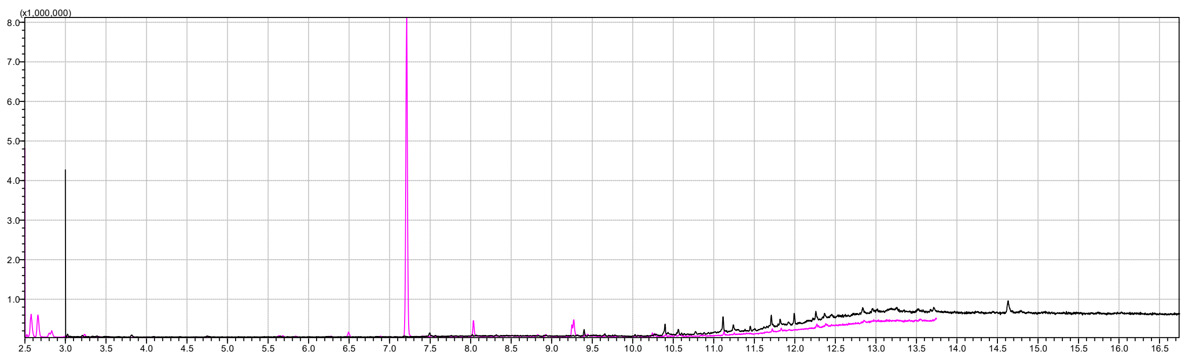

Figure 12 GC-MS chromatogram of doTERRA® Citrus Bliss blended essential oil blend at a 1:10,000 dilution

Appendix 2

Table 14 Character table recording the elution times and NIST17 library matches for analysis of GC-MS analysis of MONQ personal aromatherapy vape pen liquid – Peace Blend

PEACE BLEND – MONQ Aromatherapy Vape Pen			
Retention Time	***NIST Match***	***NIST ID***	***Comments***
7.488	94	decane	Low
7.567	81	1-iodo-2-methylnonane	Low
8.005	86	carbonic acid, non-vinyl ester	Low
8.757	92	isoborneol	Low
8.792	95	levomenthol	Low
8.808	94	terpinen-4-ol	Low
8.897	91	alpha-terpineol	Low
9.083	82	3-trifluoroacetoxypentadecane	Low
9.173	84	tridecane	Low
9.183	90	hexadecane	Low
9.185	84	decane	Low
9.223	90	linalyl acetate	Low
9.255	81	d-carvone	Low
9.307	89	carbonic acid	Low
9.372	85	decane	Low
9.375	86`	ether, 6-methylheptyl vinyl	Low
9.612	85	ether, octadecyl vinyl	Low
9.665	91	dodecane	Low
9.827	79	nerolidol	Low
9.917	77	geranyl propionate	Low
10.110	84	pentadecane	Low
10.147	89	methyl methanthranilate (methyl ester)	Low
10.230	91	2h-pyran	Low

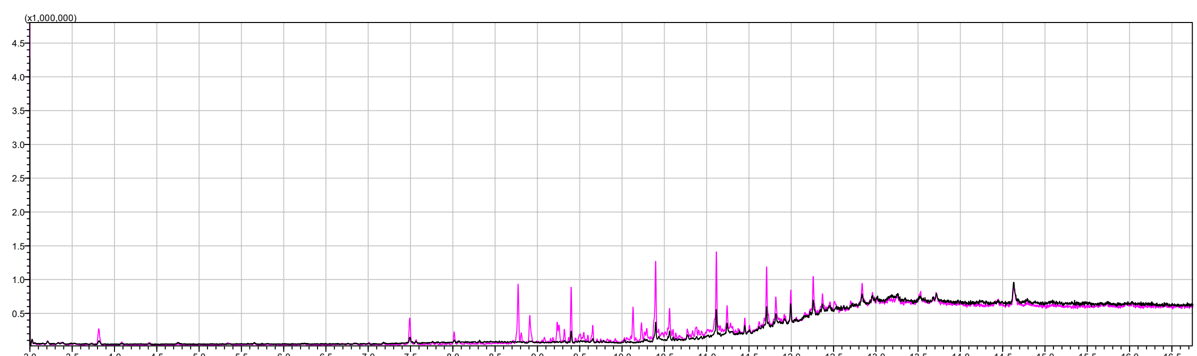

Figure 13 GC-MS chromatogram showing hexane method blank (black line) and the liquid from the MONQ Peace blend aromatherapy vape pen (pink line)

Table 2 Character table recording the elution times and NIST17 library matches for analysis of GC-MS analysis of MONQ personal aromatherapy vape pen liquid – Relieve Blend

RELIEVE BLEND –MONQ Aromatherapy Vape Pen			
Retention Time	***NIST Match***	***NIST ID***	***Comments***
3.215	81	pantolactone	Low
7.987	74	linalool	Low
8.760	78	isoborneol	Low
8.912	85	alpha terpineol	Low
9.058	89	citronellol	Low
9.262	90	geraniol	Low
9.397	92	pentadecane	Low
9.807	76	phenol	Low
10.005	76	cedrene	Medium
10.202	93	caryophyllene	Strong
10.363	82	1-octadecanesulphonyl chloride	Low
10.488	78	(1s,2r,5r)-2-methyl-5-((r)-6-methylhept-5-en-2-yl)bicyclo[3.1.0]hexan-2-ol	Low
10.540	80	nerolidyl acetate	
10.582	86	cedrene	Medium
10.665	87	nerolidol	Low
10.868	89	carophyllene oxide	Strong
10.920	84	linalool	Medium
10.940	80	pseudodiosgenin	Low
11.092	82	1-octadecanesulphonyl chloride	Low
11.173	83	1(2h)-napthalenone	Low

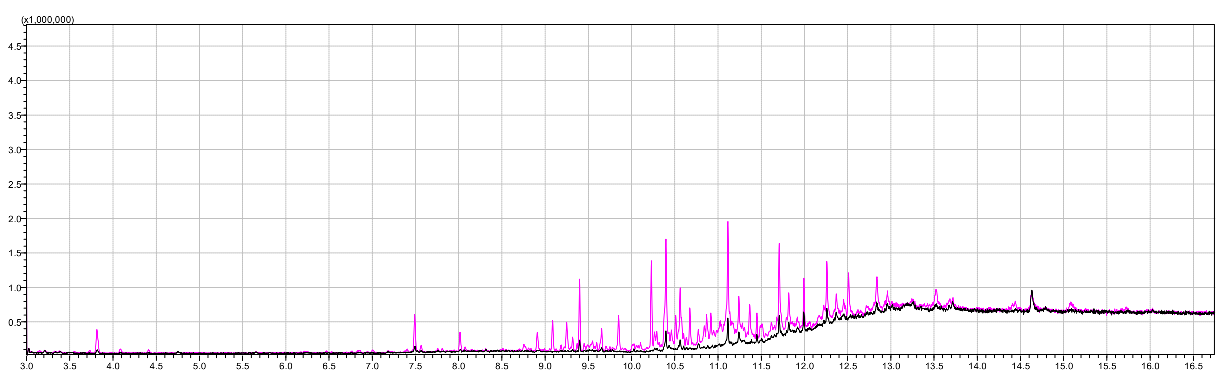

Figure 2 GC-MS chromatogram showing hexane method blank (black line) and the liquid from the MONQ Relieve blend aromatherapy vape pen (pink line)

Table 3 Character table recording the elution times and NIST17 library matches for analysis of GC-MS analysis of MONQ personal aromatherapy vape pen liquid – Healthy Blend

HEALTHY BLEND –MONQ Aromatherapy Vape Pen			
Retention Time	***NIST Match***	***NIST ID***	***Comments***
7.988	82	linalool	small
8.773	86	levomenthol	Medium
8.810	90	terpinen-4-ol	Medium
8.908	87	alpha.terpineol	Medium
9.235	84	linalyl acetate	Low
9.257	91	d-carvone	Medium
9.802	90	aceteugenol	Very Strong
10.01	79	copaene	Low Intensity
10.053	79	aromandendrene	V small
10.187	95	caryophyllene	Very strong
10.217	93	caryophyllene	Very strong
10.382	80	linalool	Low
10.743	76	Butyric acid	Strong
10.867	90	caryophyllene oxide	Low
10.920	77	farnesense epoxide	Medium
10.923	77	trans-Z-.alpha.-Bisabolene epoxide	Medium
11.027	90	ar-turmerone	Very Strong
11.190	88	curlone	Strong
11.380	78	benzyl benzoate	Strong
11.433	77	[1,1'-bicyclopropyl]-2-octanoic acid, 2'-hexyl-, methyl ester	

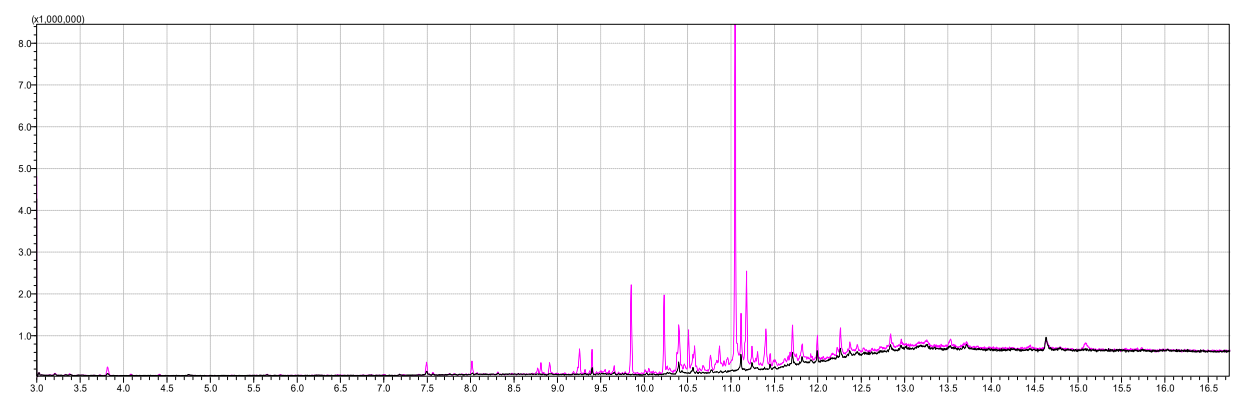

Figure 3 GC-MS chromatogram showing hexane method blank (black line) and the liquid from the MONQ Healthy blend aromatherapy vape pen (pink line)

Table 4 Character table recording the elution times and NIST17 library matches for analysis of GC-MS analysis of MONQ personal aromatherapy vape pen liquid –Mountain Blend

MOUNTAIN BLEND –MONQ Aromatherapy Vape Pen			
Retention Time	***NIST Match***	***NIST ID***	***Comments***
8.015	81	linalool	Low
8.773	93	levomenthol	Low
8.810	94	terpin-4-ol	Low
8.910	94	α-terpineol	Low
9.9235	90	linalyl acetate	Low
9.255	94	d-carvone	Medium
9.845	92	eugenol	Medium
10.013	86	copaene	Low
10.05	84	aromandendrene	Low
10.230	96	caryophyllene	Medium
10.378	85	α-caryophyllene	Low
10.405	88	α -curcumin	Low
10.462	84	trans-alpha-bergamotene	Low
10.510	86	eugenol	Low
10.582	90	cedrene	Medium
10.682	82	caryophyllene oxide	Low
10.763	89	aR-tumerol	Very Strong
10.867	92	caryophyllene oxide	Low
10.920	78	farnesense epoxide	Low
11.028	91	aR-tumerone	Low
11.178	92	curlone	Strong
11.402	93	benzyl benzoate	Low

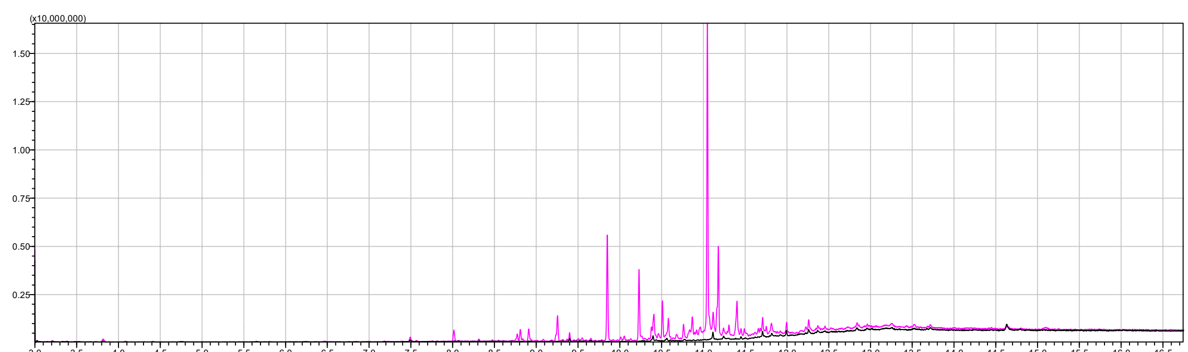

Figure 4 GC-MS chromatogram showing hexane method blank (black line) and the liquid from the MONQ Mountain blend aromatherapy vape pen (pink line)

Table 5 Character table recording the elution times and NIST17 library matches for analysis of GC-MS analysis of MONQ personal aromatherapy vape pen liquid –Vibrant Blend

VIBRANT BLEND –MONQ Aromatherapy Vape Pen			
Retention Time	***NIST Match***	***NIST ID***	***Comments***
8.018	81	Carbonic Acid – nonyl vinyl ester	Low
8.913	77	.alpha.-Methyl-.alpha.-[4-methylpentyl]oxiranmethanol	Low
9.070	77	citronellol	Low
9.158	71	digitoxin (enolide)	Low
9.235	77	nerolidol	Low
9.292	80	ether dodecyl isopropyl	Low
9.518	85	carvacrol	Low
9.618	84	1-octanol	Possibly from Hexane solvent
10.003	76	1-chloroecosaine	Low
10.110	75	isopropyl tetradecyl ether	Low
10.205	81	(1S,2R,5R)-2-methyl-5-((R)-6-methylhept-5-en-2-yl)bicyclo[3.1.0]hexan-2-ol	Low
10.362	92	hexadecane	Low
10.872	75	pseudodiosgenin	Low
11.048	75	eicosaine	Low
11.098	87	heptacosane	Low

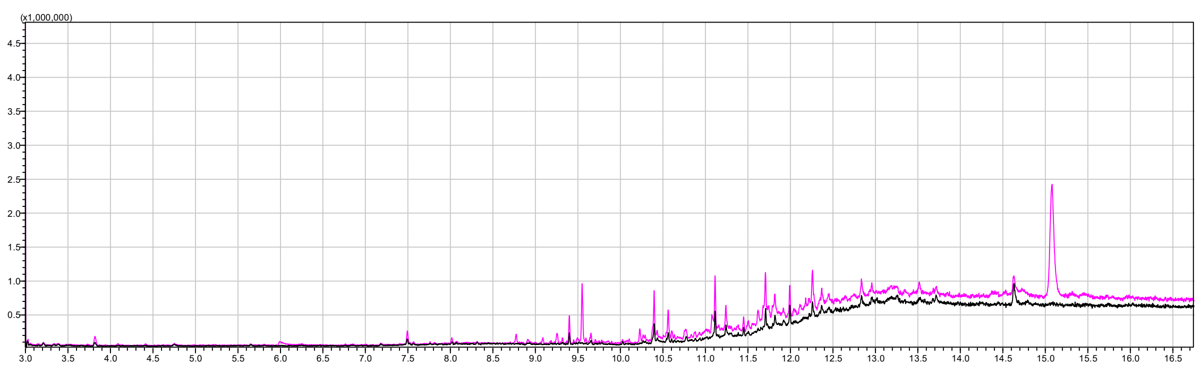

Figure 5 GC-MS chromatogram showing hexane method blank (black line) and the liquid from the MONQ Vibrant blend aromatherapy vape pen (pink line)

Table 6 Character table recording the elution times and NIST17 library matches for analysis of GC-MS analysis of MONQ personal aromatherapy vape pen liquid –Focus Blend

FOCUS BLEND –MONQ Aromatherapy Vape Pen			
Retention time	***NIST Match***	***NIST ID***	***Comments***
8.765	86	levamenthol	Low
8.912	86	L. α-terpineol	Low
9.082	77	dihydrocarveol	Low
9.075	79	cis-p-mentha-1(7),8-dien-2-ol	Low
9.082	85	dihydrocarveol	Low
9.223	82	d-carvone	Low
9.273	91	(-)-carvone	Low
9.852	83	aceteugenol	Low

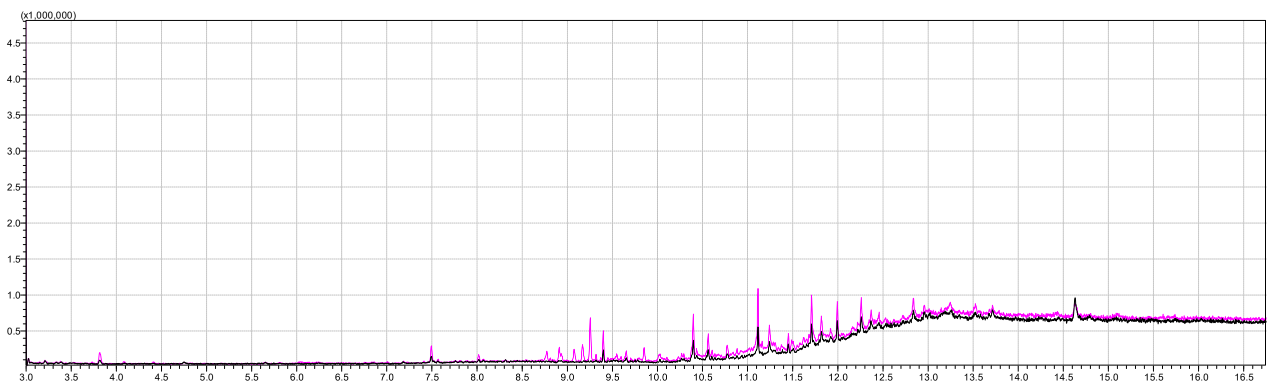

Figure 6 GC-MS chromatogram showing hexane method blank (black line) and the liquid from the MONQ Focus blend aromatherapy vape pen (pink line)

FOREST BLEND –MONQ Aromatherapy Vape Pen

All of these peaks correlate with various alkanes. They do not correspond to any known terpenes within the NIST17 library. This is likely due to instrument error, which was an ongoing issue over the course of this project with the GC-MS becoming overloaded with VG and PG samples causing it to give strange results. However, there is also a possibility some of these elutions could be due to the plastic labware in which the liquid was stored reacting with the hexane solvent. It could also be due to a quality control issue with the product itself.

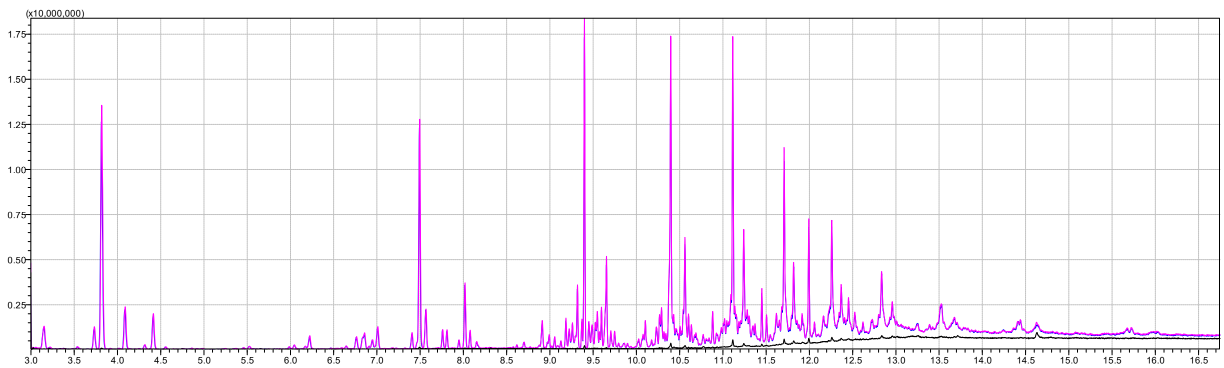

Figure 7 GC-MS chromatogram showing hexane method blank (black line) and two repeats of 1:40 dilution of MONQ Forest blend aromatherapy vape pen liquid (Pink=Forest 1; Blue=; Forest 2.)

ZEN BLEND –MONQ Aromatherapy Vape Pen

These peaks all correlate to the method blank, their increased intensity may be due to the reactions happening with the hexane and plastic labware, or different stock used for the blank. The dual peaked elution at ~13.25 minutes is a known column contaminant of this machine at this time. There are no identifiable terpenes in this sample.

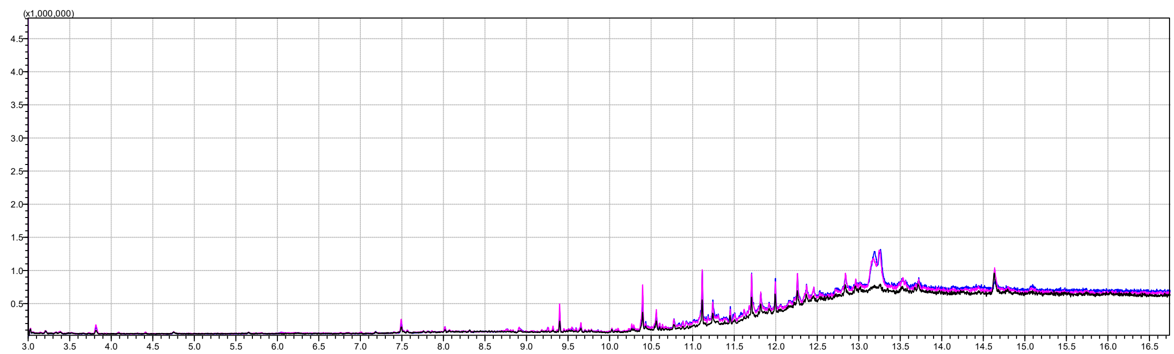

Figure 8 GC-MS chromatogram showing the hexane method blank (black line), and two separate samples of a 1:40 dilution of MONQ Zen blend aromatherapy vape pen fluid (Pink = Zen1, Blue = Zen2) over a time period of ~9 minutes to ~14 mins