

The Adulteration of Essential Oils - and the Consequences to Aromatherapy & Natural Perfumery Practice.

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Part 1. Oil Adulteration.

Introduction.

As far as adulteration is concerned, producers and distributors of essential oils are frequently painted as "the bad guys", but it should be pointed out that their oil customers frequently demand oils below the market price while still wanting to be told that they are authentic. In this climate, the honest oil trader may find it virtually impossible to survive on the margins he is allowed to make (many have already gone bust). For example, in the late 20th Century, lavender oil (*Lavandula angustifolia*) was being sold almost as a loss leader by many French producers as the market was unwilling to pay a realistic price; currently, the aroma industry is dominated by a handful of large and powerful international houses whose corporate buyers often attempt to drive raw material prices to impossibly low levels, not allowing workable profits to be made. This sets the scene for unethical practices.

Essential oils - a definition.

An essential oil (e.o.) is the volatile oil containing odiferous elements of the plant, produced by steam or hydro-distillation of aromatic vegetable plant matter. E. o. components arise via the secondary metabolism of plants and are stored within specialised structures; ideally they are isolated with minimum chemical changes from human intervention. Citrus oils, produced by the mechanical pressing of citrus peels, are also called essential oils, and, according to the International Standards Organisation (ISO), so are dry-distilled oils - such as cade oil (from the branches of *Juniperus oxycedrus*) and styrax pyrogenée (from *Liquidamber* spp).

E.o.'s should be produced by purely physical means, and be 100% pure and wholly derived from the named botanical source - but how are these standards to be guaranteed? No quality standards for the authentication of essential oils exist in aromatherapy, in spite of the revelations of gross adulteration of aromatherapy oils for retail sale (*Health Which* 2000). Professional aromatherapy organisations have failed to issue standards, in spite of individual schemes being put forward (Jones 1998) but, in contrast, other essential oil-using industries are served by the following standards.

The Pharmaceutical Trade: British Pharmacopoeia (BP) 2002 is published on recommendation of the Medicines Commission UK. Oils specifications are also published in the European Pharmacopoeia 4th edn 2002 (Eur. Pharm 4th edn); United States Pharmacopoeia (USP); also the pharmacopoeia's of individual nations such as China, India etc. Earlier editions of The British Pharmaceutical Codex (BPC) contains many essential oil standards still in use today.

Essential Oil Trade: Monographs on individual essential oils (EOA Standards) were produced by the Scientific Committee of the Essential Oil Association Inc.

Flavourings Industry: Food Chemicals Codex IV (1996, US) produced at the request of the FDA (1992), is widely used for guidance by the food flavourings industry.

Aroma Companies: Many larger established Flavour & Fragrance Houses have their own internal purchasing standards.

Independent Certifying Bodies: International Standards Organisation (ISO Standards TC 54) & Association Française de Normalisation (AFNOR) both have detailed standards for e.o.'s.*

*An example is ISO 3515 for Oil of Lavender (2001) which includes minimum and maximum percentages of thirteen substances, and their occurrence in French (spontaneous and clonal), Bulgarian, Russian, Australian and 'other origin' lavender oils. Limits for lavandulyl acetate, for example, are set at 2.0-5.0% in Bulgarian lavender oil by the standard.

Aromatherapy

Whilst it is apparent that the current BP or ISO standards may serve the needs of particular industrial sectors, they do not entirely address the unique needs of the aromatherapy profession, since:

Holistic aromatherapists demand that "pure" and "complete" oils are used, rather than oils only distilled for periods which are attractive economically, on a yield: fuel-consumption basis (although unnecessary energy 'wastage' may not be seen currently as a particularly "deep green" strategy!).

Many essential oils used in aromatherapy are particular to that industry, and not necessarily extensively used elsewhere e.g. *Ravensara aromatica*, Rosemary oil verbenone chemotype, *Helichrysum italicum* ssp. *serotinum* etc.

As well as "pure and natural", the words "wild-crafted", "organic" and "clinical grade" are frequently over-hyped descriptor terms used by both aromatherapy and by "naturals" traders, which need more careful definition prior to professional endorsement.

Natural perfumery

Natural perfumers are other potential users of pure essential oils. Grimshaw (1989) discussed "purist" perfumers (who employ no chemically produced or chemically modified ingredients), but also discussed reasons why others may wish to use up to 50% synthetics in formulations. This was, in a way, a prediction today's situation, whereby **aromachology perfumes** (worth £611 million for years 1999-2001 according to Mintel Database 2002) contain a proportion of synthetics stipulated by the perfume house, mixed in with the e.o.'s. The alleged psychopharmaceutical effects of these products still depend on the utilization of authentic essential oils in the formulation – as far as marketing claims/hype are concerned anyway. A realistic "in-practice" distinction between mass-marketed **aromatherapy perfumes** (as opposed to 100% e.o. blends) and aromachology perfumes, other than at a hypothetical level, has yet to be defined, since both commonly employ synthetics. The synthetics content can presumably have either symbiotic, neutral or opposing effects (mood changing etc.) to those claimed for the e.o.'s in the perfumes in question, hence the need for clinical testing of the finished formulations to support advertising claims.

Types of adulteration.

There are several distinct categories of adulteration:

1. Addition of single raw materials. This simple form of adulteration can be conveniently divided into two groups:

"Invisibles" – i.e. those materials undetectable by a gas chromatograph (GC) analysis operating under routine conditions to analyse essential oils.

"Visibles" – those materials normally detectable by GC

"Invisibles": an example of this type is the deliberate addition of vegetable or mineral oil to essential oils (Nour-el-Din *et al.* 1977) - rapeseed oil in the EU is a particularly cheap vegetable oil which has been used for this purpose. Theoretically the "total area" of the detectable components of the oil's gas chromatogram should be reduced by this latter type of adulteration, creating suspicion for the analyst and the need for further investigation. These adulterant materials may be revealed by aqueous alcohol solubility tests, and their presence further verified by using a different GC column & operating conditions (to detect mineral oil), or by derivatisation (for example the use of a methylating agent for vegetable oils – whereby the volatile methyl esters of the fatty acid components of glyceryl esters are revealed by subsequent GC analysis).

"Visible" diluents in this context include a number of solvents and perfumery materials. For example the following have been found in commercial essential oils: in a few instances resulting in a warning or prosecution by regulatory authorities:

Abitol (a primary hydroabietyl alcohol) – often used for extending resinoids.
Benzyl alcohol (now classified as a sensitiser by SCCNFP opinion)
Benzyl benzoate (now classified as a sensitiser by SCCNFP opinion; formerly widely used to extend resinoids)
Carbitol (diethylene glycol monoethyl ether or DEGME)
Diacetone alcohol
Dipropylene glycol (DPG)
Dipropylene glycol methyl ether (DPGME) and tripropylene glycol methyl ether (TPGME) - both of these substances are in air freshener technology.
Herculyn DÔ (hydrogenated methyl ester of rosin)
Isoparä (odourless kerosene fractions often used as a candle perfume diluent)
Isopropyl myristate (IPM)
Phthalate esters such as dibutylphthalate (DNP) or diethyl phthalate (DEP).
Triacetin (the anti-fungal compound glycerol triacetate - a popular food flavourings vehicle). 3,3,5-Trimethyl-hexan-1-ol.

Use of materials like isotridecyl acetate (ITDA, Fixateur 404), Herculyn D and Abitol, can be moderately difficult to spot, because the materials may show a myriad of late-eluting small peaks on a GC trace representing their different constituent isomers, which could be overlooked by an inexperienced analyst especially at low levels.

In all the above instances of "visible" and "non-visible" adulterants, the added material is merely a diluent, and makes no odour contribution of its own. Addition of 10-14% of such a material may pass un-noticed if the material is evaluated against a retained standard solely on an odour basis – even by an expert nose – but it will in all probability be revealed by subsequent physio-chemical testing e.g. added vegetable oil in patchouli oil can often be revealed by a solubility test in 90% alcohol at 20°C.

2. The addition of cheaper essential oils and adjuncts.

Blending in cheaper oils to meet a customers' target purchasing price, or to make additional profit for the producer, is commonplace in the oil trade. Some practices mentioned by Arctander (1960) - for example, the practice of extending of Amyris oil (*Amyris balsamifera*) with Cedarwood oil Virginia (*Juniperus virginiana*) and Copaiba Balsam (*Copaifera* spp.) – are unlikely to fool too many potential customers in these present & more educated times, but other more common adulteration practices still remain, which include:

Bergamot oil (*Citrus bergamia*): addition of lemon oil, rectified ho oil (*Cinnamomum* spp.) and acetylated ho oil Bitter orange oil (*Citrus aurantium* subsp. *aurantium*): addition of sweet orange oil (*Citrus sinensis*) & orange terpenes, plus trace amounts of character compounds.

Cedarwood oil Virginia (*Juniperus virginiana*): addition of cedarwood oil Chinese (*Cupressus funebris*).

Cinnamon bark oil (*Cinnamomum zeylanicum*): addition of cinnamon leaf oil.

Cinnamon leaf oil (*Cinnamomum zeylanicum*): addition of clove fractions, eugenol, cinnamic aldehyde etc.

Clove Bud oil (*Syzygium aromaticum*): addition of clove stem oil & isolates (eugenol) & eugenyl acetate.

Fir Needle oils (*Abies* spp.): addition of turpentine fractions, camphene, (-)- bornyl acetate etc.

Geranium oil Chinese (*Pelargonium* hybrids): addition of adulterated Indian geranium oil (which itself has been known to contain diphenyl oxide!)

Grapefruit oil (*Citrus paradisi*): addition of orange terpenes or sweet orange oil distilled + minor amounts of (+)-nootkatone & others.

Lavender oil (*Lavandula angustifolia*): addition of cheaper lavandin (*Lavandula x intermedia*) oil varieties; the addition of spike lavender oil (*Lavandula latifolia*); the addition of ho oil rectified (*Cinnamomum* spp) and acetylated ho or acetylated lavandin oils etc.

Lemon oil (*Citrus limon*): addition of orange terpenes, lemon terpenes & by-products (e.g. steam-stripped lemon oil). For lemon oil BP, expressed lime or grapefruit oil is added to poor grades to raise the UV absorbance level sufficiently to pass the BP specifications.

Nutmeg oil (*Myristica fragrans*): the addition of nutmeg terpenes, α -pinene, limonene, turpentine fractions etc.

Patchouli oil (*Pogostemon cablin*): addition of gurjun balsam (*Dipterocarpus* spp.); vegetable oils, Herculyn D; patchouli and vetiver distillation residues. The superior Indonesian patchouli oil is often blended with the cheaper Chinese oil.

Petitgrain oils (*Citrus* spp): addition of other citrus leaf oils & fractions, fatty aldehydes, linalyl acetate, orange terpenes etc.

Peppermint oil (*Mentha X piperita*): addition of cornmint oil (*Mentha arvensis*).

Sandalwood oil EI (*Santalum album*): addition of sandalwood terpenes, sandalwood fragrance chemicals etc.

Rosemary oil (*Rosmarinus officinalis*) addition of eucalyptus oil *Eucalyptus globulus* & camphor oil white (*Cinnamomum camphora*).

Verbena oil (*Lippia citriodora*): *L. citriodora* herb distilled over lemon oil.

Violet Leaf absolute (*Viola odorata*): addition of spinach absolute (*Spinacia oleracea*).

Ylang Ylang oil qualities (*Cananga odorata* subsp. *genuina*): addition of cananga oil (*Cananga odorata*), ylang ylang oil tails etc., ylang ylang oil reconstitutions.

And also addition of these synthetics to "convert" one oil to another:

Basil oil exotic: add linalol to convert to Basil oil Sweet (Arctander 1960).

***Eucalyptus globulus*:** add α -terpineol & others to convert to *Eucalyptus radiata*.

Geranium oil Chinese to Geranium oil Bourbon: addition of balancing materials (monoterpene alcohols and esters, especially formates), copper chlorophyll (for colour) and frequently a trace of dimethyl and/or dibutyl sulphides.

Tangerine oil (*Citrus reticula* var. *tangerine*): addition of γ -terpinene, dimethyl anthranilate, α -sinesal & perilla aldehyde to convert to Mandarin oil (*Citrus reticulata* var. *mandarin*).

3. The addition of cheap (nature identical) synthetics to oils that naturally contain these materials. Little detailed guidance has been previously published in this area. The older work of Arctander (1960) mentions a number of adulteration practices, but the sophistication of customer quality control procedures probably means that of the noted practices are now too obvious for today's market. Looking at other published material on adulteration, Singhal et al. (2001) remarks on the adulteration of spice oils by simple additions of single raw materials e.g. the addition of synthetic citral to *Litsea cubeba* oil. My own guide to questionable practices include the following:

Anise oil (*Pimpinella* spp.): addition of technical grade anethol.

Basil oil exotic (*Ocimum* spp.): addition of methyl chavicol & linalol.

Benzoin resinoid (*Styrax* spp.): addition of small amounts of vanillin, benzyl benzoate, ethyl & benzyl cinnamates, benzoic acid etc. to enhance odour (or to pass off cheaper "Sumatra" grades as "Siam").

Bergamot oil (*Citrus bergamia*): addition of linalol and linalyl acetate.

Bitter almond oil (*Prunus amygdalus* var. *dulcis*): addition of, or passing off benzaldehyde, as the oil.

Buchu leaf oil (*Barosma betulina* & *B. crenulata*): addition to cutters of monoterpene sulphide fractions synthesised from the hydrogen sulphide treatment of pulegone, including *p*-menthan-8- thiol-3-one.

Cassia oil (*Cinnamomum aromaticum*): the addition of synthetic cinnamic aldehyde, methyl cinnamic aldehyde & coumarin.

Chamomile oil Roman (*Anthemis nobilis*): addition of isobutyl angelate and bisabolols.

Cinnamon bark oil (*Cinnamomum zeylanicum*): the addition of synthetic benzaldehyde, eugenol and cinnamic aldehyde.

Citrus oils: the addition of fatty aldehydes and monoterpene alcohols and esters to terpeneless and folded citrus oils.

Caraway seed oil (*Carum carvii*): the addition of limonene and (+)-carvone

Cardamom oil (*Elletaria cardamomum*): addition of linalyl acetate, 1,8-cineole and α -terpinyl acetate.

Celery seed oil (*Petroselinium crispum*): the addition of limonene + touches of alkyl phthalides.

Cognac oil: addition of ethyl esters of aliphatic acids e.g. ethyl oenanthate.

Coriander seed oil (*Coriandrum sativum*): addition of linalol and trace amounts of certain pyrazines. NB price fluctuates – not always economic!

Cypress oil (*Cupressus sempervirens*): addition of α -pinene, d-3-carene & myrcene.

Cumin seed oil (*Cuminum cyminum*): addition of cuminaldehyde and others.

Dill seed oil (*Anethum graveolens*): addition of α -phellandrene & limonene.

Elemi oil (*Canarium luzonicum*): addition of α -phellandrene & limonene.

Galbanum resinoid (*Ferula galbaniflua*): addition of β -pinene, undecatrienes and others.

Garlic oil (*Allium sativa*): addition of aliphatic sulphide mixtures containing 2-propenyl disulphide, 1-propenyl disulphide etc.

Jasmine absolute (*Jasminum spp*): reconstructions frequently added.

Juniper oil (*Juniperus communis* var. *erecta*): addition of terpene hydrocarbon mixtures containing α -pinene & d-3-carene, also Juniper branch oil and second grade oils from spoiled Juniper berries.

Labdanum resinoid (*Cistus ladaniferus*): formerly, the addition of DEP (now discontinued) or Abitol, with small amounts of ambroxan and *p*-methyl acetophenone to enhance odour.

Lavender oil, spike (*Lavandula latifolia*): addition of eucalyptus & white camphor oil fractions, spanish sage oil etc.

Lemongrass oil (*Cymbopogon spp.*): addition of citral.

Mentha citrata oil: addition of linalol + linalyl acetate.

Mustard oil (*Brassica nigra* & *B. juncea*): allyl isothiocyanate passed off as the oil (which is used in flavourings, but is banned in perfumery & aromatherapy). Neroli oil (*Citrus aurantium* subsp. *aurantium*): reconstructions frequently added to, or passed off as the authentic oil.

Origanum oil (*Origanum* spp.): addition of *para*-cymene and carvacrol.

Onion oil (*Allium cepa*): addition of aliphatic sulphide mixtures.

Palmarosa oil (*Cymbopogon martinii* var. *motia*): the addition of geraniol.

Petitgrain oil Paraguay (*Citrus aurantia* subsp. *aurantium*): addition of admixture of linalol, linalyl acetate, α -terpineol, geranyl & neryl acetates & trace amounts of pyrazines etc.

Pine needle oils (*Pinus* spp.): addition of (-)-bornyl acetate, isobornyl acetate, (-)-limonene, α -pinene, camphene etc.

Rose oil: reconstructions using damascones, β -ionone plus (-)-citronellol and other rose alcohols, plus rose sterptenes. Occasionally adulterated with β -phenylethyl alcohol, rhodinol fractions and cheaper rose oils (Morocco, Crimea etc.).

Rosemary oil (*Rosmarinus officialis*): addition of camphor, isobornyl acetate (+ *Eucalyptus* & turpentine oil fractions).

Rosewood oil (*Aniba* spp): addition of linalol, plus trace amounts of methyl heptenone, methyl heptenol, 3-octanol, *para*-methyl acetophenone etc.

Spearmint oil (*Mentha spicata*): addition of (-)-carvone.

Thyme oil (*Thymus* spp.): addition of *para*-cymene & thymol. "Red thyme oil" is often wholly synthetic.

Vetiver oil acetylated (*Vetivera* spp): the addition of cedrenyl acetate. Wintergreen oil (*Gaultheria procumbens*): the adding of, or passing off methyl salicylate, as the oil.

Ylang ylang oil (*Cananga odorata* var. *genuina*): addition of benzyl acetate, methyl benzoate, *para*-cresyl methyl ether, geranyl acetate, benzyl benzoate, benzyl cinnamate, cedarwood oil and others or complete reconstitutions/bases.

Boelens (1997) described four types of odourants in essential oils: character compounds, essential compounds, balance compounds and artifacts. Adulterants such as monoterpene hydrocarbons, being balance compounds in Boelens scheme above, do little for the characteristic odour of the cut oils, since the added materials have little odour value in themselves. In practice, the addition of certain adulterants "flattens" the odour profile of the authentic oil, or otherwise dilutes or represses some true character, sparkle and richness. To compensate for this, a practiced oil counterfeiter will add small amounts of character compounds. Taking the example of Cypress oil *Cupressus sempervirens* var. *stricta*, the oil is often adulterated by the addition of the monoterpene hydrocarbons α -pinene and d-3-carene, which creates a crude terpinic aspect. The addition of a small amount of deca-2-(E),4-(Z)-dienyl isovalerate to the somewhat insipid cutting agent, will give a better impression of the oil's normal character, a lead which follows on from the work of Garnero *et al.* (1978) who identified the compound above in cypress shoots, and found it strongly reminiscent of the typical odour of cypress oil.

Commercial oils, adulterated by such synthetics, can often fool the less sophisticated nose, or satisfy those oil customers buying to a price, where authenticity is sometimes not a primary consideration. Depending on exact market conditions, some oils have a selling price which is so cheap that it is generally unrewarding for a trader to reconstitute, or even add, nature identicals to the product, except for some solvent-like diluents. This category includes the following oils:

Sweet orange oil (*Citrus sinensis*)

Clove leaf oil & stem oils (*Syzygium aromaticum*)

Citronella oil (*Cymbopogon* spp.): (but the oil has been known to have been crudely adulterated with dipentene and citronella terpenes)

Camphor oil white (*Cinnamomum camphora* fractions)

Commmint oil (*Mentha arvensis* subspp).

***Eucalyptus globulus* oil**

Tea tree oil (*Melaleuca alternaria*) (NB a collapse in market price means potential adulterants such as terpinen-4-ol and a- & g-terpinenes can now be as expensive as the oil).

Other oils are difficult to reconstitute with anything other than diluents because the major components are not commercially available; this class of oils includes patchouli oil, vetiver oil and to some extent ginger oil.

4. The addition of isolates or natural components to essential oils e.g. the addition of pure natural eucalyptol ex *E. globulus* oil (*Eucalyptus globulus*) to rosemary oil (*Rosmarinus officinalis*) or rectified ho oil (very high in (-)-linalol) to lavender and bergamot.

5. The addition of bases or reconstituted essential oils to genuine oils & absolutes. It is particularly economically attractive to extend high value floral absolutes such as rose (*Rosa* spp.), jasmín (*Jasminum grandiflora* other spp.) and osmanthus (*Osmanthus fragrans* var. *auranticus*), and the more valuable oils such as neroli oil and rose otto, and this practice occurs extensively within the trade.

6. The addition of individual unnatural components to oils and aromatic raw materials.

Absolutes have been traditionally produced for the consumption of the perfumery industry, but are being increasingly employed in aromatherapy (in spite of using un-natural solvents in their manufacture). Revelations that materials such as Linden Blossom absolute (*Tilia* spp.) contain hydroxycitronellal, or that Gardenia absolute (*Gardenia* spp.) has added styrallyl acetate, or that added Schiff's bases have been found in floral absolutes, should not therefore come as a complete surprise. It has been suggested that if the synthetic fragrance compound is added in to the aromatic plant material during manufacture, the added material will "blend in" better. In other instances, absolutes may well contain perfume bases or reconstitutions, rather than a single key character compound.

7. The addition of "normal" oils to oils certified as "organic".

Many certifying bodies (EcoCert, Soil Association) seemingly rely on inspection regimes, batch tracking and the separate & secure storage of organic oils, rather than independently analysing finished oils for pesticide levels, a situation inviting abuse by unscrupulous traders. There is also the huge question of "who inspects the inspectors", the lack of external expert input into their protocols, and the denial of free public access to the records of these bodies – but these topics (and many others) are another issue! The labelling laws seem so lax in the EU, that an advertising claim on a bottle, such as "contains organic orange oil" would not invite prosecution if the oil contained 0.1% organic orange oil and 99.9% non-organically produced orange oil – and who, specifically, is charged with checking anyway, and who has the necessary expertise? Further, it has been revealed that on more than one occasion, oils from endangered species have been about to be certified as "organic" by one particular organisation with seemingly little eco-awareness, and with little experience of complexities of the oil trade (Wildwood 2002), to which the president of the association concerned is reported to have replied that certification is "...bound to be an evolving process, very often in uncharted territory". And so many of us "at the coal-face" feel that the whole organic oils exercise, although possibly very noble in its intent, is not particularly watertight.

8. Passing off one essential oil as another.

"Old chestnuts" in this category include the following deceptions:

Cinnamomum camphora (var. cineole type) **fractions** (1,8-cineole rich) were passed off extensively **as Eucalyptus globulus** oil by certain Chinese suppliers in early 1998. Cineol rich fractions from *Cinnamomum longepaniculatum* may be used for the same purpose.

Cornmint oil from *Mentha arvensis* var. *piperascens* **as peppermint oil** *Mentha x piperita* (however, Chinese and Brazilian "peppermint oils" do derive from *M. arvensis* subsp.).

Melissa oil is frequently concocted from mixtures of Citronella oil, Litsea cubeba, lemon oil (Citrus limon) and various other isolates & synthetics.

Micromeria fruticosa (Turkish pennyroyal) & Hedeoma pulegioides (American pennyroyal) have been passed off **as pennyroyal oil** (*Mentha pulegium*).

Treemoss resinoid (Pseudoevernia furfuracea) is passed off as or use as an extender for **Oakmoss resinoid** (Evernia prunastri).

Petitgrain oil terpeneless (*Citrus aurantium* subsp. *aurantium*) has a former tradition of being passed off **as rosewood oil** (*Aniba* spp.). **Petitgrain oil**

Paraguay may be passed off **as petitgrain bigarade**.

Siberian silver fir needle oil *Abies siberica* has been often passed off **as Siberian pine oil** *Pinus siberica*.

Sweet orange oil Brazil has been passed off as the finer **Sweet orange oil Florida**.

Star anise oil *Illicium verum* has been passed off **as anise oil (Pimpinella spp)**, and synthetic anethole is passed off as star anise oil (in certain anise type liquors!).

Tolu balsam (Myroxylon balsamum var. a-genuinum..): some aroma concerns acknowledge that the commercial products offered are not genuine (Biolandes 1997), offered products often being blends of *Styrax* spp. and aroma chemicals etc.

.... and three additional categories which are arguably more about "contamination" than adulteration:

9. Co-gathering one species with another.

This may be deliberate or accidental, and might arise through lack of botanical education of the involved gatherers, especially those involved in "wild-crafting".

Artemisia mesaltantica ("Blue Armoise") and A. atlantica growing in Morocco are often co-gathered or deliberately passed off as **Moroccan Armoise oil (A. herba-alpa)**. However the situation is further complicated by the fact that there are at least four chemotypes of *A. herba-alpa* (!) according to Richard *et al.* (1984).

Litsea enosma & L. mollifolia as well as other *Litsea* species are used to produce the oil of **Litsea cubeba** according to Zhaobang (1995).

Massoy essential oil (from bark of Cryptocarya massoy), containing up to 90% of lactones, is used in flavourings. Joulain (1994) reports that bark has been frequently been gathered with the bark of other species in the past (which in the case of bark from *Cinnamomum* spp., might contain high levels of safrrole, of toxicological concern).

Nardostachys grandiflora is said to be often co-gathered with **Valeriana wallichii** according to Traffic International (1999) and that published chemical compositions of essential oils from these species are similar – see <http://www.users.globalnet.co.uk/~nodice/new/magazine/products/products.htm>.

10. GM-Free Essential Oil Production Methods.

Many EU oil-buying customers require oils to have been produced by plants free of gene modification, or free of Genetically Modified Organisms (GMO's) themselves, and to be free of their use, and use of their derivatives during manufacture, according to Commission Regulations 50/200, 1139/98, 90/220/EEC and 2092/91/EGW Biological Agriculture. Certificates to show that plant material was polymerase chain negative may need to be provided by suppliers to prove GM-free status.

11. Oils produced from non-irradiated vegetable sources.

Many essential oil customers require oils certified as being manufactured from non-irradiated sources at any point of the production process, in order to comply with EU Directives 99/2/EC and 99/3/EC, and so may require statements that the original oil-source vegetable raw material, (including spices), have not been gamma-ray irradiated. Suitable sources of g-radiation are usually taken to be cobalt-60, caesium-137 and electron/X-ray irradiation from an electron accelerator. It is of note that certain terpene compounds are changed by irradiation – Kawashima K. (1981) found that α -terpinene, g-terpinene and terpinolene levels drop at a dose of 7.5 kGy, whilst other spices such as cardamom and coriander change flavour attributes at the same dose Bachman & Gieszcynska (1973). Rapid tests are now available to establish irradiation status - cumin seeds for example can be subjected to the rapid detection method for g-irradiation, developed by Satyendra *et al.* 1988.

Spotting adulteration.

Odour evaluation.

A trained perfumer or essential oils analyst might immediately spot a QC problem with an oil sample before committing the oil to more sophisticated analytical tests – in fact the e.o. may simply be rejected on a poor or uncharacteristic odour profile. So, in spite of great technological advancements in analysis techniques (sometimes bordering on scientific imperialism), the trained nose can still be the single most important arbiter of quality, and it is important to keep sight of this fact.

Analysis procedures.

Quality control procedures for essential oils in aromatherapy have already been (briefly) discussed by Garner (1999).

Physiochemical methods.

These standard tests have long been used to help establish the identity of oil, on the basis that normally produced genuine oils have characteristic values for:

Specific gravity/ Relative density

Refractive index

Optical Rotation

Tests are normally carried out at ambient temperature (20°C or 25°C) if the oils are normally liquids at this temperature; guaiacwood oil (*Bulnesia sarmienti*) & rose otto (*Rosa* spp.) etc., are normally solid at these temperatures and may be evaluated at 40°C.

Oils are sometimes traded on the basis of "USP figures" or "BP figures". This means that the oils conform the relevant specifications in spite of synthetic additions, but obviously do not conform to the "100% wholly derived from the named botanical source" requirements of the pharmacopoeia monographs.

Chemical tests.

[The first two tests are indicative of contamination rather than adulteration, but are included as examples of requirements in sister industries].

Heavy metals – in essential oils used as food flavourings, a limit of 3mg/Kg arsenic, 10mg/Kg lead, 1mg/Kg cadmium and 1mg/Kg mercury is imposed under Council Directive 88/388/EEC.

Pesticides - this is a troubled area. Buchbauer (1998) reports that Schiler and his team at the free University of Berlin found levels of 34 organochlorine pesticides in 72 of 110 commercial essential oils tested, but in reality the exorbitantly high costs of testing for pesticides (dedicated equipment, trained operators etc.) often do not square realistically with the economic resources available to e.o. producers and suppliers. This situation is further driven by requirements in EC directives for food ingredients, and often puts companies in as Catch-22 situation of potentially losing potential e.o. flavourings business, or falsifying information to the customer.

Acid value

Ester value, saponification values etc – these wet chemical tests are now largely superseded by more accurate techniques e.g. high performance capillary GC.

Instrumental methods.

Suitable combinations of instrumentation used to spot adulteration include:

GC-MS

GC-FTIR (Fourier transform Infra-Red)

GC-13C NMR (Nuclear Magnetic Resonance -see Kubeczka & Formáček 2002)

HPLC-MS.

Whilst these techniques are undoubtedly useful, the cost of trained operator time and the necessary allocation of sophisticated & expensive hardware for the time-consuming methodology and number crunching capabilities, all for what is essentially a basic QC task asking a question "is this material genuine or not?", is often seen as inappropriate by aroma company management. Less capital intensive techniques therefore might be deemed more practical and appropriate, and nowadays, it might be considered that minimum equipment ("entry level") for oil authentication work would be a GC/MS facility and access to chiral chromatography techniques.

Thin layer chromatography (TLC). This technique requires a less capital-intensive investment, and for example is a requirement for essential oil testing (in conjunction with a battery of other tests), in the BP monograph (2002) on Citronella oil (*Cymbopogon winterianus*). This requires identification of the oil by TLC and gas chromatography (reference chromatogram with eight identified compounds is supplied, the test oil must be within prescribed limits), as well as requiring prescribed physio-chemical testing.

In another area, TLC has been used to identify the geographical origins of olibanum resins (Harefield 1984).

Fingerprints.

A slightly older review of the value of "fingerprints" in e.o. analysis is given by Lamparsky (1987), the subject of which centres on: pattern recognition of GC traces of e.o.'s, examples of character compounds (which Lamparsky terms impact compounds) and unique components of certain oils, as well as the enantiomeric analysis technique (see below). Nowadays, GC analysts have the additional resource of commercial available MS libraries, which compare the spectrum of each compound detected with a library of reference spectra, identifying the closest matches, and giving a confidence level estimation of each tentative identification.

Marker Compounds.

Many oils have unique or unusual marker compounds, which can be used as standards of authentication, such that some essential oil customers will only purchase oils, which have certain levels of these marker compounds. An introduction to this topic is given by Teissaire (1987). The table below gives a few analytical hints:

Essential Oil	Examine for:	Indications
Anise oil	g-himalchene, pseudoisoeugenyl 2-methylbutyrate and foeniculum.	To distinguish between <i>Pimpinella anisum</i> and <i>Illicium verum</i> .
Anise Oil (<i>Illicium verum</i>)	<i>cis</i> -anethole	Levels >0.5% may indicate addition of synthetic anethole.
Anise oil (<i>Illicium verum</i>)	Anisic alcohol & anisic aldehyde	Levels >0.7% may indicate excessive oxidation.
<i>Artemisia vulgaris</i>	Vulgarone	Authenticity indicator.
Cinnamon bark oil & Cassia oils (<i>Cinnamomum</i> spp)	Absence of 5-phenyl penta-2,4-dienal.	Impurity in added synthetic cinnamic aldehyde
Geranium oil (N. African type)	(-)-(4R)-6,9-Guaiadiene; 10-epi-g-eudesmol.	Authenticity indicators
Lavender oil (<i>Lavandula angustifolia</i>)	α -Santalene, (-)-lavandulol, (-)-lavandulyl acetate etc.	Authenticity indicators
Lemon oil, Mandarin oil etc.	d-3-Carene	Its virtual absence (<0.01%) might indicate no orange oil or orange terpenes have been added to cheapen the oil.
Patchouli oil Indonesian (<i>Pogostemon cablin</i>)	α -Gurjunene	Concentration found is proportional to concentration of added gurjun balsam.
Patchouli oil Indonesian (<i>P. cablin</i>)	Patchulol	Range 26-40% for authentic oils.
Rosewood oil (<i>Aniba</i> spp)	Eremophilene	Authenticity indicator
Rose otto (<i>R. centifolia</i>)	(Z)-9-nonadecene	Authenticity indicator in steroptene fraction of otto in which it occurs at fairly constant levels*

Table 1: A few analytical hints in essential oil examination.

*according to Ohloff and Demole

For oil authentication, analysts sometimes rely on estimation of the concentrations of non-commercially available e.o. components, which would be reduced from their normal concentration by the deliberate addition of adulterants. For example celery seed oil naturally contains a certain level of b-selinene; addition of adulterants such as orange terpenes would reduce the concentration of b-selinene to below acceptance range criteria.

Spotting minor impurities in adulterants by GC.

The GC can be employed to detect the presence of synthetic additives to essential oils from tell-tale impurities in the synthetics themselves. Added natural camphor in oils such as Rosemary and Sage may be detected by the presence of impurities such as α -pinene, camphene, b-pinene, cineole, fenchone, fenchol, camphene hydrate and methylcamphenilol (BP 2002). Similarly in rosemary, fir needle and pine needle

oils, the presence of added borneol may be detected by examination of borneol: isoborneol ratios, or (-)-bornyl acetate by looking at bornyl acetate: isobornyl acetate.

Impurities in synthetic linalol made via the older acetylene or b-pinene routes (e.g. dehydrolinalol, dihydrolinalol, tetrahydrolinalol, plinol & others), don't occur naturally in linalol-containing essential oils such as lavender (*Lavandula angustifolia*). Therefore the presence of dehydrolinalol etc, used to be relatively easy way of identifying synthetic linalol in a lavender oil GC chromatogram. Nowadays, cleaner linalol can be manufactured via the a-pinene process and its' presence in oils is less easily detectable. Cheaper grades containing impurities are still employed however, so that in cheap linalyl acetate (which is derived from linalol), it is still often possible to detect traces of, dihydrolinalyl-, pinanyl- or plinyl acetates, thus giving away its' origin.

Enantiomeric analysis.

Essential oils invariably contain substances with one or more asymmetric carbon atoms, which give rise to different optical isomers (enantiomers). The distribution ratios of these different enantiomers (as determined by GC using chiral columns) can be used as a powerful tool to detect oil adulteration by nature-identical synthetics. A review of enantioselective GC analysis is provided by Mosandl (1998); uses of enantiomeric analysis in establishing lavender oil authenticity is reported by Kreis and Mosandl (1992) and for bergamot oil by Cotroneo *et al.* (1992). A few of examples of specific oil constituents showing high levels of optical purity include:

Essential Oil	Enantiomeric Ratio	Enantiomeric Excess	Reference
Bergamot oil: (Citrus bergamia)	(4R)-(-)-linalol to 100%; (4S)-(+)-linalol 0%.	100	Cotroneo et al. (1992)
Fennel oil (<i>Foeniculum vulgare</i>)	(4R)-(+)-a-phellandrene 100%; (4S)-(-)-a-phellandrene 0%	100	Cassiabanca (1996)
Ho leaf oil (<i>Cinnamomum</i> spp)	(4R)-(-)-linalol 96.2%; (4S)-(+)-linalol 3.8%	92.4	Bernreuther & Schreier (1991)
Lavender oil (<i>Lavandula angustifolia</i>)	(4R)-(-)-linalyl acetate >99%; (4S)-(+)-linalyl acetate <1.0%	98 +	
Lavender oil (<i>Lavandula angustifolia</i>)	(4S)-(+)-linalol usually <5% but up to 15% during very prolonged hydrodistillation. Over 15% of 4S-(+)- linalol indicates adulteration with racemic linalol	85 (worst case) 95 normal	Kreis & Mossandl (1992)
Melissa oil (<i>Melissa officinalis</i>)	(3R)-(+)-methyl citronellate 99.0%; %; (3S)-(-)-methyl citronellate 1.0%	98	Lawrence (2000)
(Synthetic linalool)	(4R)-(-)-linalol 50.0%; (4S)-(+)-linalol 50.0%	0	

Table 2. Enantiomeric information on some essential oils.

Certain of these optical purity figures may be useful in spotting the addition of synthetic racemic adulterants (such as linalol), but obviously the addition of racemic ho leaf oil to bergamot or lavender oil may be harder to spot on this basis alone, due to the similarities in optical purity of the constituent linalol. Dugo (2001) discusses enantiomeric techniques for the differentiation of citrus oils, as summarised in the table below:

Oil	Can be differentiated from:	On the basis of the enantiomeric ratios of:
Bergamot oil	Lemon oil	Linalol
Bergamot oil	Mandarin oil	b-Pinene, sabinene, linalol
Bergamot oil	Sweet orange oil oil	Sabinene & limonene

Lemon oil	Mandarin oil	b-Pinene, sabinene, limonene, linalol
Lemon oil	Sweet orange oil	Sabinene & limonene
Lemon oil	Bitter orange oil	Linalol
Mandarin oil	Sweet orange oil	Sabinene & limonene
Mandarin oil	Bitter orange oil	b-Pinene, sabinene, linalol
Sweet orange oil	Bitter orange oil	Sabinene & limonene

Table 3. Differentiation of Citrus Oils by Enantiomeric Distribution of Certain Volatiles after Dugo *et al.* (2001).

Isotopic analysis.

Measurements of ratios of $^{12}\text{C}:^{13}\text{C}$ distribution by natural isotope spectrometric methods, or by isotopic analysis of the $^{12}\text{C}:^{13}\text{C}$ ratios in pyrolytic CO_2 are mentioned by Lawrence (2000) as two of six possible ways to determine the natural and genuine status of e.o. components. $2\text{H}:1\text{H}$ site specific natural fractionation of single components represents a third possible way in Lawrence's scheme, and this technique is explored for essential oils containing linalol and linalyl acetate by Hannaguelle *et al.* (1992). Hener (1992) maintains that the combination of enantioselective GC together with stable isotope ratio analysis is a powerful tool for this purpose of establishing the 100% natural status of an aromatic raw material. The expense of these techniques, however, surely limits their more universal application, although Schmidt *et al.* (2001) report that isotopic analysis has become an important tool in flavourings for checking the natural status of ingredients such as benzaldehyde and vanillin.

Component ratios.

This technique relies on the tendency of specific components to adhere to given set of component ratios in given e.o.'s oils, which can be determined by simpl GC or GC/MS analysis. For example Lawrence (1997) provides twelve component ratios for Midwest Native Spearmint oil (*Mentha spicata*) against which unauthenticated oils of that type may be compared. In an earlier article on the same theme, Shu & Lawrence (1996) examined samples of peppermint and Indian lemongrass oils, and claim to show, in the case of the Indian lemongrass oil examination, that since the component ratios of the examined oil fall between that of E. Indian and W. Indian lemongrass oils, that the oil must have been adulterated. In general terms, this technique is useful and well within the ability and budget of the average commercial oils analyst.

[Background note – the situation in other trades using essential oils:

As analysis standards in essential oil trading become more demanding, many essential oil buyers (especially in the flavourings sector) buying oils and oleoresins as food grade now require a Certificate of Naturalness fulfilling the requirements of European Directive 88/388/EC. Buyers may additionally require assurance that oils are produced by purely physical means (steam distillation, separation of oil from distillation waters by gravity etc.) and that no additional chemicals other than water are used in the manufacture of these products. Information on the addition of any other components (such as anti-oxidants BHA/BHT or tocopherols, colourants, residual solvents according to CPMP/ICH/283/95 etc) may also be requested.

In other areas, customers may require information on dioxin content to determine that the material complies with EC Regulation EC466/2001. Information on dioxin-like PCB's and polycyclic aromatic hydrocarbons may be further requested].

Part 2.

The Consequences of Adulteration to Aromatherapy & Natural Perfumery Practice.

Background.

Aromatherapists and natural perfumers have long required that their e.o.'s are genuine, but conversely, the essential oil trade has traditionally offered oils to the perfumery and flavourings trades on a "buyer beware" principle. Therefore, the finding in the *Which*

Health report (Health Which 2001) on aromatherapy oils, citing a case where a labelled sandalwood oil turned out to be a synthetic sandalwood aroma chemical, cannot come as a complete surprise.

Whilst many e.o. used in aromatherapy are sourced from commercial oil trade outlets, other items are offered by smaller dedicated aromatherapy oil producers. In attempt to make themselves uniquely positioned in the oil market, aromatherapy oil suppliers have previously boasted that their oils are distilled longer and under gentler conditions to produce superior oils. This is a complete nonsense – longer treatment can only encourage greater artefact production via thermal degradation, and any perceived more pleasing odour effect is possibly due the increased oil complexity (via the creation of artefacts). Even more curious is the easy acceptance of hype that CO₂ extracts are suitable for the aromatherapy community. In many cases the CO₂ extracts are of unknown composition and toxicity, extractions are not standardised (depending on operating conditions CO₂ extracts can resemble either resinoids or essential oils, and all stages in between) and the use of any co-solvents during processing is often omitted by suppliers. Furthermore the concentration of pesticide residues during the CO₂ extraction of spices are from seven to fifty three times greater than the values obtained by use of conventional solvents, according to Guba R. (2002).

Consequences.

The adulteration of essential oils leads to the following concerns:

1. Toxicity of the adulterant(s).

Phthalates such as DEP are still occasionally found as adulterants in essential oils. Phthalate esters have been withdrawn as ingredients by many cosmetics manufacturers on toxicity grounds; specific phthalates (DEHP & DBP) are classified by the EU as reproductive toxicants. Whilst consumer pressure groups campaign for awareness in this area (see for example: <http://www.nottoopretty.org/>); the other side of the argument can be viewed on <http://cms.phthalates.com/index.asp?page=3>.

Traces of residual organic solvents (such as hexane and cyclohexane) in oils and absolutes are found as a result of extraction & co-distillation practices.

The presence of pesticides in tainted e.o.'s in cosmetics has been described as a serious health & safety issue by Buchbauer (1998); their inevitable presence in aromatherapy oils is an unresolved issue.

2. The interference of adulterants on the expected physiological or psychophysiological effects of the essential oil.

Point 2 above has long been a concern of aromatherapists, but proof of adverse effects has been harder to find, although the following section below might furnish the beginnings of a case:

Chiral Issues from added adulterants..

Addition of racemic synthetics to natural essential oils may distort the enantiomeric ratios of the naturally occurring substances within the make-up of oils, and thus may have an untoward effect on the expected physiological outcome. The logic behind this may be predicted from data accrued on the contrasting physiological effects of different chiral isomers of the same substance. A few examples are given below:

Huenberger *et al.* (2001) have demonstrated that inhalation of (+)-limonene caused increases in systolic blood pressure and changed alertness and restlessness in subjects, whereas (-)-limonene only affected blood pressure.

(-)-Carvone was reported to increase pulse rate, diastolic blood pressure and restlessness whereas (+)-carvone increased systolic and diastolic blood pressure. Traynor (2001) reports that when evaluated by Contingent Negative Variation, (+)-rose oxide confers relaxing physiological effects, whilst (-)-rose oxide (which occurs in Bulgarian Rose oil and geranium oils) possesses a significantly higher stimulative effect.

Sugawara *et al.* (2000) looked at the effects of 10 mins inhalation of the different linalool isomers [(-)-linalol purified from lavender, (+)-linalol from coriander, and synthetic (+/-)-linalol] inhaled before and after work. Effects were examined by sensory scoring and portable forehead surface EEG measurements. They found inhaling (-)-linalol after hearing environmental sounds produced a more favourable impression produced a more favourable impression in the sensory test but was accompanied by a greater decrease in beta waves after than before work. Conversely with mental work, there was a tendency for agitation accompanied by an increase in beta waves. (+/-)-Linalol gave results similar to (-)-linalol, but (+)-linalol gave the reverse results.

Buchbauer (1998) maintains that each constituent of an essential oil contributes to the beneficial or adverse effects of the oil. I contend that changing the distribution of chiral components of oils by deliberate adulteration with racemic synthetic odourants may in fact change the properties of the oil.

Glossary.

Adulteration: the purposeful addition of cheaper alternative oils, oil fractions, by-products, isolates, natural or non-natural synthetics etc., to reduce the cost price of the oil.

Aliphatic: a series of compounds built up from carbon backbones with the specific formula C_nH_{2n+2} .

Bases (perfumery): complete accords/compositions added to fragrances to form part of an entire note e.g. rose base, jasmin base, etc. May not always be as complex or true to nature as reconstituted oils

Buyer Beware: the operating policy whereby an essential oil or other raw material is offered to a customer without guarantees, and the onus is on the customer to assess its purity or status.

Chiral compounds: the ability of a specific substance, containing one or more asymmetric carbon atoms, to occur as 2 or more optical isomers (**enantiomers**). These chiral components may have different odour profiles e.g. (-)-carvone is minty, (+)-carvone is caraway-like.

Coeur: heart or body of a fragrance or raw material.

Commercial essential oils: essential oils available in quantity, which it is understood, are not necessarily pure.

Compound: an aroma industry term for a fragrance mixture, often with more components than a blend, and liable to comprise (mainly) synthetics. Do not confuse with substance!

Coupage: (in this context) an amount of extender added to an essential oil.

Extender: an agent (single or compound) used for the purpose of adulterating or extending an oil.

Extending: a term for adulteration almost implying a degree of legitimacy.

Isolate: a specific fraction of an essential oil. May be composed of a single chemical e.g. eugenol from Clove oil.

Oil blend: a (hopefully) harmonious mixture of essential oils.

Organic oil: a more expensive essential oil, which has been derived from vegetable matter which has been grown in a pesticide free environment, but which still liable to have a pesticide content reflecting background contamination/incorporation.

Sophisticating an oil: a scientifically inappropriate trade term for extending or adulterating an oil.

PQ quality: A slightly older expression, standing for "Perfume Quality" i.e. not necessarily pure, but the odour profile should strongly remind of the named oil, and the performance in product should be satisfactory.

Reconstituted oil: An oil made from nature identical synthetics, to look like analytically as far as possible -, and to give an accurate odour impression of -, the named essential oil.

Abbreviations Glossary.

(-)- laevorotatory isomer

(+)- dextrorotatory isomer

BP British Pharmacopoeia published by the Pharmaceutical Press.

DEP Diethyl phthalate

EC European Community

EU Economic Community

EO essential oil

EI East Indian

GC gas chromatography

GC/MS coupled gas chromatography with mass spectroscopy

ISO International Standards Organisation

TLC Thin layer chromatography

WI West Indian

USP United States Pharmacopoeia

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