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Handbook of ESSENTIAL OILS

Science, Technology, and Applications

SECOND EDITION

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Edited by
K. Hüsnü Can Başer
Gerhard Buchbauer

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Preface

The overwhelming success of the first edition of the *Handbook of Essential Oils: Science, Technology, and Applications* prompted us to edit an updated version. All of our contributors of the first edition were animated by us either to cooperate again or to tell us if their articles of the first edition should be kept as they are. Moreover, we asked experts to write contributions on new topics not covered in the first edition. This was the case in two instances: Prof. Eva Németh-Zámboriné's Chapter 4 report on "Natural Variability of Essential Oils Components," which is based on her excellent plenary lecture at the 44th International Symposium on Essential Oils in Budapest in September 2013, and the important Chapter 18, "Adulteration of Essential Oil," by the perfumers Erich Schmidt and Jürgen Wanner. In the first treatise, the author discusses the reasons why variability of components often occurs in essential oils. The influence of molecular genetics in understanding the background of biodiversity in volatiles formation is elaborated with examples. Thinking on adulteration, the first idea is always a more or less criminal act, such as a fraud or a betrayal to the debit of customers; however, also to a certain extent, these natural products can lose their excellent quality by natural processes, such as storage under inferior conditions or improper access to air thus furnishing oxygenated artifacts. Now, these essential oils are adulterated. Other examples of such processes are described very impressively in this new chapter.

Chapters 3 through 6, 13 through 16, and 19 through 21 remained as they are written in the first edition. Now, they are listed as Chapters 3 (formerly 3), 5 through 7 (formerly 4 through 6), 15 (formerly 13) and 16 (formerly 19), 19 (14), 20 (15), and 23 (20). Especially as to the contribution of Prof. Maria Lis-Balchin (formerly Chapter 13, now Chapter 15), we thought that she has already reported all important facts on aromatherapy that was necessary to tell. Also, the interesting Chapter 25 on "Aroma-Vital Cuisine" by Maria M. Kettenring and Lara M. Vucemilovic, a well done example for the application of essential oils, really, has been kept as it is in the first edition, because an update of the recipes and thus creating a collection of old and new recipes for cooks does not meet the sense of this book. The chapter on "Biological Activities of Essential Oils" (formerly 9) has been updated and divided into two contributions, now Chapters 10 and 11. Chapter 24 "Storage, Labeling, and Transport of Essential Oils" and Chapter 26 "Recent EU Legislation on Flavors and Fragrances and Its Impact on Essential Oils" have been fully revised and updated.

We hope that also this second edition with the alluring mixture of former, updated, and even new contributions will satisfy the curiosity and needs of all of our readers who share with us the vivid interest in this fascinating natural topic, the essential oils.

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Editors



K. Hüsnü Can Başer was born on July 15, 1949, in Çankırı, Turkey. He graduated from Eskisehir I.T.I.A. School of Pharmacy with diploma number 1 in 1972 and became a research assistant in the pharmacognosy department of the same school. He did his PhD in pharmacognosy between 1974 and 1978 at Chelsea College of the University of London, London, United Kingdom.

Upon returning home, he worked as a lecturer in pharmacognosy at the school he had earlier graduated from and served as director of Eskisehir I.T.I.A. School of Chemical Engineering between 1978 and 1980. He was promoted to associate professorship in pharmacognosy in 1981.

He served as dean of the faculty of pharmacy at Anadolu University (1993–2001), vice dean of the faculty of pharmacy (1982–1993), head of the department of professional pharmaceutical sciences (1982–1993), head of the pharmacognosy section (1982–present), member of the university board and senate (1982–2001; 2007), and director of the Medicinal and Aromatic Plant and Drug Research Centre (TBAM) (1980–2002) in Anadolu University. He retired from Anadolu University in 2011. Since then, he has been working as a visiting professor in King Saud University, Riyadh, Saudi Arabia.

During 1984–1994, he was appointed as the national project coordinator of Phase I and Phase II of the UNDP/UNIDO projects of the government of Turkey titled "Production of Pharmaceutical Materials from Medicinal and Aromatic Plants," through which TBAM had been strengthened.

He was promoted to full professorship in pharmacognosy in 1987. His major areas of research include essential oils, alkaloids, and biological, chemical, pharmacological, technological, and biological activity research into natural products. He is the 1995 recipient of the Distinguished Service Medal of International Federation of Essential Oils and Aroma Trades (IFEAT) based in London, United Kingdom, and the 2005 recipient of "Science Award" (health sciences) of the Scientific and Technological Research Council of Turkey (TUBITAK) among others. He has published more than 700 papers in international refereed journals (more than 540 in SCI journals), 144 papers in Turkish journals, and 141 papers in conference proceedings. He communicated 940 papers in 262 symposia. He is the author of 52 books or book chapters.

Visit http://www.khcbaser.com for more information.



Gerhard Buchbauer was born in 1943 in Vienna, Austria. He studied pharmacy at the University of Vienna, from where he received his master's degree (Mag. pharm.) in May 1966. In September 1966, he assumed the duties of university assistant at the Institute of Pharmaceutical Chemistry and received his doctorate (PhD) in pharmacy and philosophy in October 1971 with a thesis on synthetic fragrance compounds. Further scientific education was practiced postdoc in the team of Professor C.H. Eugster at the Institute of Organic Chemistry, University of Zurich (1977–1978), followed by the habilitation (postdoctoral lecture qualification) in pharmaceutical chemistry with the inaugural dissertation entitled "Synthesis of Analogies of Drugs and Fragrance Compounds with Contributions to Structure-Activity-Relationships" (1979) and appointment as permanent staff of the University of Vienna and head of the first department of the Institute of Pharmaceutical Chemistry.

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In November 1991, he was appointed as a full professor of pharmaceutical chemistry, University of Vienna; in 2002, he was elected as head of this institute. He retired in October 2008. He is married since 1973 and had a son in 1974.

Among others, he is still a member of the permanent scientific committee of International Symposium on Essential Oils (ISEO); a member of the scientific committee of Forum Cosmeticum (1990, 1996, 2002, and 2008); a member of numerous editorial boards (e.g., *Journal of Essential Oil Research*, the *International Journal of Essential Oil Therapeutics*, *Scientia Pharmaceutica*); assistant editor of *Flavour and Fragrance Journal*; regional editor of *Eurocosmetics*; a member of many scientific societies (e.g., *Society of Austrian Chemists*, head of its working group "Food Chemistry, Cosmetics, and Tensides" (2000–2004), *Austrian Pharmaceutical Society*, and *Austrian Phytochemical Society*, vice head of the *Austrian Society of Scientific Aromatherapy*, and so on); technical advisor of IFEAT (1992–2008); and organizer of the 27th ISEO (September 2006, in Vienna) together with Professor Dr. Ch. Franz.

Based on the sound interdisciplinary education of pharmacists, it was possible to establish an almost completely neglected area of fragrance and flavor chemistry as a new research discipline within the pharmaceutical sciences. Our research team is the only one that conducts fragrance research in its entirety and covers synthesis, computer-aided fragrance design, analysis, and pharmaceutical/medicinal aspects. Because of our efforts, it is possible to show and to prove that these small molecules possess more properties than merely emitting a good odor. Now, this research team has gained a worldwide scientific reputation documented by more than 400 scientific publications, about 100 invited lectures, and about 200 contributions to symposia, meetings, and congresses, as short lectures and poster presentations.

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18 Adulteration of Essential Oils

Erich Schmidt and Jürgen Wanner

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18.1 INTRODUCTION

18.1.1 General Remarks

Requirements of governmental bodies in the use of natural products increased tremendously in the last years. Not only the directions by the flavor regulation but also the demand of the consumer organizations presses the industry and producers to supply solely natural products, often desired in "bio" quality. The green responsibility for natural products brought synthetic aroma chemicals in a poor light. Regarding the mass market for cosmetic products, genuineness in fragrances is very limited because of the sources of raw material. On the other side, prices for raw materials increase since years for natural flavor and fragrance ingredients. The acceptance of fragrance compounds containing essential oils and natural aroma chemicals is a way to fulfill the consumer demand for safe, effective, and too affordable finished products. But even today with that claims and in spite of fabulous analytical equipment and highly sophisticated methods, many adulterated essential oils are found on the market. The question arises, what are the motivations for such a serious condition? This and above all, the manner of adulteration and the matching analysis methods shall be treated within this chapter.

Essential oils are constituents of around 30,000 species of plants around the world. Only a few of them are used in today's flavor, cosmetic, animal feeding, and pharmaceutical industry as well

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as in aromatherapy. Observing the product range of producers and dealers, about 250–300 essential oils are offered in varying quantities. Within those oils, 150 can be characterized as important oils in quantity and price.

Consumption of these essential oils worldwide is tremendous. The data published by Lawrence (2009) show a total quantity of more than 120,000 metric tons. A monetary value can barely be calculated, as prices rise and fall over in a year; exchange rates in foreign currency and disposals within the dealer's community falsify the result. Gambling with natural raw materials and the finished essential oil takes place with all the important ones. Comparison of production figures and those of export and import statistic will vary sometimes obviously; discrepancy appears in higher amounts. Reunion Island's National Institute of Statistics and Economic Studies (INSEE, 2008–2009) reports for the year 2002 the low quantity of 0,4 to kg of "vetiver oil Bourbon" exported; the sold quantity in the market was more than ten times higher. From 2003 until today, no vetiver oil is produced on Reunion Island. The essential oils traded were proven pure and natural but were coming from other production areas.* However, "Bourbon" quality generated a much higher price. It must be assumed that the value of essential oils worldwide will be far above $10 billion and this dimension invites to make some extra money out of it.

Screening essential oil qualities from the market with high-end analytical equipment leads to detection of many adulterated essential oils. Alarming is the fact that these adulterations appear not only within the consumer market but also in industry and trade. The questions arise again, what is the explanation for such a behavior and what are the reasons? Adulterations are subject of many publications with high impact; most of them came out in the beginning of the nineties, when new analytical methods were developed and applied. However, adulteration of essential oils began a long time ago. This chapter deals with adulteration starting in history up to the present. High criminal energy must be responsible for such sacrileges. Financial advantages, market shares, monopole status, and sometimes simply a sportive action are only some reasons.

18.2 DEFINITION AND HISTORY

Observing the significant number of publications about essential oils using the Internet, the results show lack of knowledge, finding "extracted" essential oils, distilled ones by "supercritical fluids," distilled Jasmine oil, therapeutic essential oil, and so on. It is remarkable that in scientific publications of serious publishers, there is the same confusion and ignorance about the term "essential oil." Even in regulatory and institutional papers, this term is used falsely and unprofessionally. Essential oils are clearly defined by the International Standard Organization (ISO) in Draft International Standard (DIS) ISO 9235, 2013 (former 9235.2)—Aromatic natural raw materials—Vocabulary. This DIS war, revised 2013, tightened the strict definition for an essential oil:

> Essential oil: Product obtained from a natural vegetable raw material of plant origin, by steam distillation, by mechanical process from the epicarp of *Citrus* fruits, or by dry distillation, after separation of the aqueous phase—if any—by physical process.

Further it is mentioned that steam distillation can be carried out with or without added water in a still. No change has been made with the note that "the essential oil can undergo physical treatments (e.g., filtrations, decantation, centrifugation) which do not result in any significant change in its composition." This is a tightening of the old definition. Natural raw material also was updated in the definition: "Natural raw material of vegetal, animal or microbiologic origin, as such, obtained by physical, enzymatic or microbiological processes, or obtained by traditional preparation processes (e.g., extraction, distillation, heating, torrefaction, fermentation)." These definitions show clearly the importance of standards to avoid any adulteration during all stages of collection,

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production, and trade. Terms like "pure or natural essential oils" now are misleading as an essential oil has to fulfill this demand. Other regulatory elements are used for standardization like the pharmacopoeias around the world. Furthermore medicinal authorities and animal feeding product industry settled own regulatory standards. In the field of aromatherapy, no standards are settled although just in that field, the existence of ISO standards or pharmacopoeias as base should be implemented. The use of natural cosmetics is reported to result in a higher demand for oils but the quantity available on the market was and is insufficient to satisfy this developing market. Today many aromatic chemicals from "natural" source, produced by enzymatic reaction from sometimes not natural starters by microorganisms, help the producers to fill up that shortage. But are these chemicals really "natural"?

Adulteration is defined as "making impure or inferior by adding foreign substances to something" or "is a legal term meaning that a product fails to meet international, federal or state standards" (The Free Dictionary n.d. [a]). In this context all kinds of adulterations are enclosed like poisonous, economical, and unethical ones. In the United States, FDA regulations for food and cosmetics are the basis of law; in Europe consumer protection and food regulations as well as the cosmetic regulation have to be applied. Adulterations must not always be deliberated with criminal or unethical background. Environmental pollution like herbicides or pesticides cannot be prevented; these are unavoidable "adulterations." Negligently adulteration, basing on the lack of knowledge, still happens today. By selection of the botanical raw material, very often mistakes are done. Another instance is the missing of suitable production facilities and again, insufficient professionalism of the staff working there. The vocabulary for adulteration of essential oils is fanciful: extending, stretching, cutting, bouquetting, and rounding up to even "sophistication."

The history of adulteration reaches back to the turn of nineteenth to twentieth century. It is ongoing hand in hand with the establishment of chemistry and the development of synthetic aromatic chemicals. Already in 1834 it was possible to isolate cinnamaldehyde from cinnamon bark oil and as a result in 1856, it was synthesized. It is worth to know that Otto Wallach, a basic scientist for terpene chemistry at the University of Göttingen, already started in 1884 with the clarification of terpene structures. In 1885 he defined the contribution for the structure of pinenes, limonene, 1,8-cineole, dipentene, terpineol, pulegol, caryophyllene, and cadinene. The development of synthetic aroma chemicals found in essential oils and extracts started with the synthesis of vanillin in 1874 by W. Haarmann and F. Tiemann. Literature gives only a few information about adulteration and the authors' research is going back to publications from 1919. The company Schimmel & Co. in Leipzig, Germany, was founded in 1829 under the name Spahn & Büttner. In 1879 Schimmel was the first company to establish an industrial laboratory for the production of essential oils. Famous well-known scientists like Wallach (Isolation of Muscon) and Eduard Gildemeister (the famous books of Gildemeister and Hofmann) were there at work. In 1909 Schimmel started publishing the famous "Schimmel-Berichte" (Schimmel reports). These reports about progress in research on essential oils, containing composition of essential oils, isolation of chemicals, cultivation of fragrance plants, and adulteration of essential oils, were discussed. In the report from Schimmel-Berichte (April/October 1919), the following adulterations were reported: "Bergamot oil with phthalates and terpenes, bitter almond oil with raw benzaldehyde, cassia oil with phthalates, cinnamon oil Ceylon with camphor oil, lemon oil with water, lavender oil with phthalates and terpineol, peppermint oil with glycerol esters, menthol, phthalates and spirit, sandalwood oil with benzyl alcohol, star anise oil with fatty oils and rose oil with palmarosa oil and spermaceti."

That points out clearly that already at that time adulteration was omnipresent. Schimmel was the first company to create a synthetic neroli oil on the market. The cause of that was the establishment of a syndicate comprising of the South French growers and, what is more, frost and storm damages during orange flower blooming. In 1920, again in one of the "Schimmel-chronicles" (Schimmel-Bericht April/Mai 1920), the adulteration of lemon oil is reported: "Skilled mingled, with terpenes, sesquiterpenes and citral, the determination with specific gravity and optical rotation are not sufficient to detect adulteration in lemon oil." As a solution for that problem,

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preparation of as much as possible fractions, tested for the solution in ethanol of 89% Vol., was proposed. Rochussen (1934) reported that several oils were adulterated, as other than the permitted parts of the plant were added. The utilization of turpentine oil was the most used component for adulteration of those essential oils, bearing large amounts of terpenes. Mineral oils (paraffin, petrolatum, benzene, and Vaseline [petrolatum]) were reported to adjust the change of physical data forced by other adulteration chemicals. For the same purpose cedarwood, gurjun balsam, and copaiba balsam oils were applied because of their weak odor.

At that time, physical constants like optical rotation, density, refraction, boiling point, freezing point, residue on evaporation, ester value, acid value, carbonyl value, and water content could give satisfying data about the examined oils. In addition the so-called wet chemistry could give some more answers. Detection reaction, titration, photometry, and gravimetric analysis were used to achieve a more precise result about the genuineness of an essential oil (detailed information will follow). Lawrence (2000) published in Perfumer & Flavorist an original text of E.W. Bovill, RC Treatt, London, about the essential oil market in 1934. One key sentence was as follows: "Essential oils, largely because they are liquids, are easy to adulterate in ways which are difficult to detect and – it must be admitted they very frequently adulterated." At that time it was obvious that adulteration was a fight likely as between a thief and a policeman. Previously, adulteration was done by producers and brokers and customers had to believe the words about purity and nativeness. The only key of trustfulness could be the relationship between producers/traders and clients by permanent control in the harvest and production time locally. Ohloff (1990) confirmed that through hydrodistillation in industrial scale, progress was made in synthesizing compounds with effective methods. Camphor was one of the important chemical components (not only for the flavor and fragrance use but as starting substance for important "plastic" products), together with borneol. Starting with the synthesis of important aromatic chemicals at accessible prices like linalool, geraniol, phenylethanol, and the esters, the adulteration of essential oils started progressively. In a flash, essential oil components were available in sufficient quantities and at prices much cheaper than the essential oil itself. Another event was the problem of the two world wars. Especially in World War II, the acquisition of raw materials of natural or synthetic kind was no more possible at all. The military machine used nearly all raw material resources, chemists were forced to establish substitutes for essential oils, and that resulted in several new synthetic aroma chemicals, which now could be used to stretch or to substitute an essential oil. Thus, the ingenuity of corruption by essential oil producers increased rapidly, and even with general application of chromatographic analysis in industrial laboratories beginning in the 1980s, falsifications could hardly be detected. The authors noticed in the course of a visit to Provence area and producers of lavender and lavandin oil that starting with the analysis by gas chromatography using packed columns in 1979 led to the result that compositions of samples of essential oils were not adequate to literature of that time. By informing the producers about the establishment of chromatographic equipment, the quality was immediately rising. But not all was satisfying. So a "touristic" tour to the facilities of the producers was decided during harvest and production time, end of August 1981. Starting in Puimoisson and with the help of a little blue empty perfume bottle ("flacon montre," used at that time for lavender perfumes), the staff was asked for a little bit of lavender oil. By giving them some Francs, he had the opportunity to watch around the distillation facility. Together with the oil sample, information about special observations was noticed. The same happened in facilities of Riez, Montguers, Simiane, Richerenches, Rosans, Remuzat, Banon, and Apt, and from all production units oil samples could be collected and these were analyzed in the laboratory by gas chromatography. As a result, only one from 10 samples was in accordance with the requirements. The others were adulterated mainly by synthetic linalool and linalyl acetate (recognized by dihydro- and dehydrolinalool as well as dihydro- and dehydrolinalyl acetate) but also with too high contents of limonene, borneol, and camphor (by adding lavandin oil), far too little amounts of lavandulol and lavandulyl acetate. Adulteration by adding those chemicals after distillation could not have happened, as the samples he received were filled directly from the Florentine flask. Finally he was in luck to observe that before closing the cover of the plant, linalyl

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acetate was spread by means of a portable pump with tank directly on the lavender. The cover was closed and the essential oil now running from the Florentine flask could be seriously called as natural. Interesting was the observation of the distillation facilities. In four cases drums with linalool and linalyl acetate from BASF (Badische Anilin- & Soda-Fabrik) could be detected. Concerning linalool and linalyl acetate, it is worth to mention that 1 year later other drums were occurring from a producer in Bulgaria produced by another synthesis pathway without de- and dihydro components.* At that time it was a customary practice, as reported by Touche et al. (1981), to use relationships of *cis*-β-ocimene to *trans*-β-ocimene (R1) and *trans*-β-ocimene to octanone-3 (R2) as well as linalool + linalyl acetate to lavandulol + lavandulyl acetate (R3). Statistically collected values over many years guaranteed the correct relations.

Another observation was the use of lavender bushels together with some lavandin bushels. This could be transparently seen; they were larger in size and the leaves were characteristic of Lavandin Grosso. At least, there were other chemicals in a rack, such as camphor and geraniol, borneol, terpinen-4-ol, and β-caryophyllene. What should these chemicals be for? He was told that these are for special clients that are so-called comunelles, meaning a mixture of several charges from different farmers but from the same plant was established. The last production unit had constrained not only a laboratory but also a production unit with many chemicals. From that time on the author did no more trust any producer or trade companies.*

Today such a way of working is no more possible. Ordering an essential oil means, with all possible parameters, the properties are scanned and tested and standards must be fulfilled. In spite of all loyalty of a producer/customer relationship, only analytical, physical, and sensory examination is inevitable.

18.3 ADULTERATION

18.3.1 Unintended Adulteration

The term "adulteration" must be understood as a negative concept. It is in nearly all cases a criminal and unethical behavior. But there will be the possibility that it is an act of unwanted adulteration without any intent and has nothing to do with dishonesty. Reasons can be the lack of knowledge, bad equipment, wrong treatment of the plant material, not allowed methods for distillation and no good manufacturing practice. Of course, all these things should be avoided, but they happen.

Selection of correct plant material is the basic for the production of essential oils. It is clearly confirmed in every ISO standard, from which botanical source the oil has to be produced. When a cultivation of the biomass is possible, it must be guaranteed that the correct species is used. By using wild collected plants, the possible risk of collecting similar species or chemotypes cannot be excluded. As an example, essential oil of *Thymus serpyllum* L. from Turkey origin was sent for analysis. As a result, the oil was absolutely different from experience and literature. Thymol and carvacrol were too low, and linalool was higher as well as geraniol and geranyl acetate: What could have happened? People collecting the wild thyme took everything that look like a thyme plant without looking for the chemotypes (which is hardly possible by visual selection). Higher quantities of linalool and geraniol chemotypes were present. The oil was natural at all, but it is not to be used in flavor application or aromatherapy. A further example is the petitgrain oil: The petitgrain from Paraguay is not a bitter orange plant as such. *Citrus aurantium* L. is the true bitter orange plant used for production of the essential oil from peel, but also from leaves to obtain petitgrain oil "bigarade." The oil from Paraguayan plants is derived from *Citrus sinensis* L. Pers. × *Citrus aurantium* L. ssp. *amara* var. *pumila*. This is a hybrid from a sweet and bitter orange. The composition of that oil is different from the first because of its higher content of limonene, *trans*-β-ocimene, and mainly linalyl acetate (ISO/DIS 8901 and ISO/DIS 3064 2015). The Paraguayan

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oil was sold as bitter orange oil; however, curiously, the oil of petitgrain from Paraguay was much cheaper. A further mistake can be the inappropriate treatment of the biomass. Fresh distilled plant material results in a different final product than the oil from dried or fermented material. The question arises, is it a desired procedure or a mistake or an omission? Good manufacturing practice (GMP) is a standard application procedure in technologically developed countries, but what about emerging nations or those without any technical progress? Can such essential oils be traded or used?

Hydrodistillation is nowadays a highly developed, automated and computerized method to produce essential oils. Applying computer programs for detection- and production parameters observing all conditions. Starting with the plant material (moisture content, degree of maturity, oil content) followed by temperature, steam quantity, steam pressure, cooling temperature, and subsequent treatment, all is handled electronically. In spite of such a technology, mistakes can happen as in the case of distilling *Melissa officinalis* L. from a biological culture. When analyzing that oil by gas chromatography–mass spectrometry (GC-MS), a content of more than 5% of thymol was detected and confirmed three times. After discussing the problem with the producer, he guaranteed that only balm, fresh cut, was used. Contamination in the analyzed sample could be excluded as the second sample showed identical results. After checking all distillation equipment, the reason was found. Before producing Melissa oil, thyme was distilled and he discovered a small chamber within his own constructed cooling unit. There remained about 50–60 mL of oil and it was mixed up with balm oil in the course of hydrodistillation. This is a great financial loss for the producer taking account of the price.* Adulteration can also be done by, for example, adding basic solution to the vessel while distilling. Sometimes it is used to avoid artifacts formation from the plant acids. By raw materials with high-grade acids, for example, barks like massoia, this is sometimes applied. The neutralization of the pH value inhibits the ester formation or degradation reactions in the course of the processing. The use of cohobation is another fact for discussion. This method uses the recovery of volatiles from distillation of water, remaining solved and without separation by Florentine flask. As long as this water is returned to the distillation vessel, there is no problem at all. For some oils like thyme oil, it is of importance to use cohobation, because of the high content of phenols, which dissolve not insignificantly in distilled water. Venskutonis (2002) reports that "cohobation minimizes the loss of oxygenated compounds but increases the risk of hydrolysis and degradation." Similar is the situation with rose oil. Here the cohobation is an important step to receive a genuine reflection of rose odor. Treating the distillation water with salt (NaCl) or pH adjusting to remove plant acids seems to be discussed, as it will still remain an essential oil. By extracting the distillation water with any solvents, which then will be removed by distillation in vacuum, will lead to an extract. This extract, added to the distilled essential oil will result in the loss of the term "essential oil" by definition.! Codistillation is reported for several "essential oils." Mainly sandalwood oils are mentioned to be extracted and redistilled. Valder (2003) reported that the production method of sandalwood oil is sometimes the hexane extraction followed by codistillation with propylene glycol and finally a rectification is done. Baldovini (2010) stated that this method is not used anymore as it does not longer comply with ISO rules. Rajeswara (2007) tested the effect of codistillation using crop weed with several plant materials like citronella, lemongrass, palmarosa, *Corymbia citriodora*, and basil. He observed the increase of yields and also changes in composition. The author was able to observe a similar effect, analyzing bio lavender oil. The oil differed in composition from standards with higher ester content, higher oxidation compounds, and less 3-octanone. The cultivation was bio, without removing any weed. The percentage of weed was esteemed to be more than 30%. The oil did not match ISO standard.* Contaminated containers for production and transport are another possibility of adulteration. Reused drums, not cleaned, are often the cause for worst damage of the essential oil.

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Plastic containers contain phthalates as plasticizers and the hydrocarbons solve these. Phthalates contaminate even in smallest traces the oil for the use in natural finished products.

18.3.2 Intentional Adulteration

If anybody adulterates willfully and knowingly, it is a criminal act and never a wangle or minor offense. The target to cheat a customer by supplying an essential oil being not conform to any standard is a felony. There is no excuse for such an unethical behavior. Again, until now, adulteration can be found on essential oil market. Terms like cutting, stretching, blending, bouquetting, or sophistication try to moderate this act. The causes for adulteration are manifold and often related with economical and environmental reasons.

18.3.3 Prices

The cutting of essential oil happens in most cases because of the price. They are subject to falling and increasing prices, depending on crops and the demand but also speculation. The last is often the reason why essential oils come to market with adulteration in every dimension. All economical ambition is the increase of profit, the more the better to fulfill shareholders demand. Essential oil market makes no distinction as it is the same as the law of supply and demand. Speculation was always a reason to ameliorate the process. By hoarding raw materials, the profit can go up in high levels. Lavender oil is an example for that. Most of the farmers in France keep parts of the production in the cellars to wait as far as the new crop starts, until the price seems to be the best. Another example from the authors' experience was the competitive battle with cornmint oil in the 1980s. China and Brazil were the two countries with the highest production at that time. Both increased the cultivation area with the result that in the next season prices fall down tremendously because of oversupply. So Brazil decided to decrease the cultivations and in the next year, prices were going up like a rocket, as China too had decreased production, and demand of the market could not be fulfilled. At that time, most menthol was produced from cornmint and consumption was rising. Stabilization was achieved by supplement of sufficient synthetic menthol and India coming to the market as third production continent. Nowadays speculation has another dimension. Likely nearly all foodstuffs and plant crops are merchandized at the stocks and of course this has influence on the oil market. Increased prices in essential oil market cannot be transferred simply onto the industry of finished products. Mass market and global players will not accept any price advance, so the only solution is cutting or stretching. This situation leads to a competitive market, where adulterated essential oils with certificate of naturalness are sold.

18.3.4 Availability

The market changes from monopole situation for suppliers to buyers market within a few months. Shortage of biomass leads to a rapid increase of prices. The causes will be dryness in the production area, flood during harvest time, and ice and frost like in the case of citrus fruits in California or Florida. Tempests are crucial for destruction of stretches of land as well as deletion caused by insects or microbes. Annual rainfalls in tropic regions and lack of sunshine in Mediterranean region are reasons for partial failure. Unavailability or decreasing availability is a high risk for global players in the fine perfumery industry. The costs of launching a perfume worldwide exceed the amount of $100 million easily and one can imagine that the lack of an important natural raw material and therefore, unavailability of that perfume can lead to ruinous losses. In consequence of that, natural essential oils and also extracts are no more or only in small quantities added to the perfume compounds. It is easy to replace a natural product by a compound. Lemon oil, bergamot oil, rose oil, lavender oil, and many others can be substituted by basic synthetic compositions. Only some special oils like vetiver, sandalwood, patchouli, and orris are never perfect to compensate.

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18.3.5 Demand of Clients

The various fields of applications sometimes cause the client industries to require special essential oils. One of the reasons is of course the price. The end-consumer industry settles the prices for the market. Competitive prices have to be adjusted and the problem for the use of essential oils is that prices can change from crop to crop or even from week to week.

18.3.6 Regulations

A huge number of regulations have been established for the protection of consumers worldwide. Cosmetic regulation, flavor regulation, and REACH (*R*egistration, *E*valuation, *A*uthorisation and Restriction of *C*hemicals; the European chemical law) extend to the Globally Harmonized System of Classification and Labeling of Chemicals (GHS). All these provisions cover essential oils, but not per se. They are dealt with their contents. Most oils contain one or more chemicals, classified as toxic, allergic, sensitizing, or carcinogenic. Consequently these oils are no more or only in very limited quantities applicable. As an example, the methyl eugenol must be mentioned. Methyl eugenol is a native ingredient of distilled rose oil. Between 1.5% and 3.5% is found in this essential oil. Considering the European Cosmetic Regulation, the maximum dose in finished leave-on products might not exceed 0.001%, which results in the fact that rose oil is no more applicable in cosmetic products. The same pertains to pimento oil from leaves and berries, oil of myrtle, magnolia flower oil, laurel leaf oil, and bay oil. The demand of the industry is rose oil with nearly no or only traces of methyl eugenol. Applying various methods like fractioning and remixing and chemical reactions it was tempted to fulfill the regulation. The result was not as desired, as the odor was not a rose anymore and the finished product could not be named as "essential oil" or "natural."

To avoid an increase of cases of allergic contact dermatitis in the public, many essential oils nowadays have to labeled as "sensitizing—can cause allergic reaction"! Cosmetic producers as well as discounters with private labels avoid naming and labeling and thus, force the replacement of natural products. Essential oil producers offer natural oils with reduced values of sensitizers and this is not possible. Maybe the essential oils are treated in some way, but then are no more "natural," according to ISO standard.

18.3.7 Aging

It is impossible to characterize aging as adulteration without intent. Changes over storing, either correct or false, always result in quantitative modification of the composition. The formation of oxidative compounds like epoxides, peroxides, and hyperoxides is a dangerous effect. Terpenoids show the properties to be volatile but also to be thermolabile. The mentioned oxidative substances show the negative reaction activities coming in contact with the skin. Sensitizing and allergic reactions are consecutive. Countless components of essential oils are subject to oxidation reaction with atmospheric oxygen. Citrus oils implicate such chemicals. Dugo and Mondello (2011) confirm that the reaction of degradation of sabinene, limonene, γ-terpinene, neral, and geranial in lemon oil results in the enlargement of p-cymene, *cis*-8,9-limonene oxide, (*E*)-dihydro carvone, (*Z*)-carveol, 2,3-epoxy geraniol, and carvone. Particularly the increase of p-cymene in citrus essential oils is a distinct detection for heterodyning and aging. Sawamura et al. (2004) demonstrated in a poster compositional changes in commercial mandarin essential oil and detected in a 12-month test the decrease of limonene (60%) and myrcene (nearly 100%) and the increase of (*Z*)-carveol (from 0.0% to 2.8%) and carvones (from 0.2% to 3.0%). Brophy et al. (1989) could observe degradation in terpinen-4-ol, γ-terpinene, and β-pinene but rise of p-cymene and α-terpineol. Examination of peroxide value seems not to be the suitable tool to recognize aging of essential oils, as some of the reactions are reversible. Eucalyptus oil, rosemary oil, and of course citrus oils have demonstrated that warming of these oils in a closed system at 40°C reduces obviously the peroxide value.

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The most suitable method of recognizing aging is analysis by GC-MS. It has to be the responsibility of producers and traders to take care for the safe quality of their essential oils.

18.3.8 Cupidity

This of course is the main reason for adulteration. Cupidity is defined as "strong desire, especially for possessions or money; greed" (The Free Dictionary n.d. [b]). Although this is the main reason, here is not the place to discuss anything about cupidity as it is human fault.

18.3.9 Simple Sports?

The knowledge about absence of qualified analytical equipment on the side of the client seduces producers as well as the trade to cut essential oils. With the development of new chromatographic methods and an increase in sensitivity as well as selectivity, the inventiveness of the producers shows high wealth of ideas. Again, the race between a thief and a police could have been stopped by the costs of investments in analytical systems, but there seems no end by observing bad qualities on the market.

18.4 POSSIBLE ADULTERATIONS FOR ESSENTIAL OILS

18.4.1 Water

Adding of water is very simple and cheap. This is not possible for all essential oils but is likely for those possessing compounds with high affinity of binding to water. Conifer varieties and citrus oils are examples. Siberian pine (*Abies sibirica* Ledeb.) was supplied in August at 25°C–30°C. Visually no water could be detected. In January the oil came from stock (–5°C) and the quantity of water could be esteemed to 8%. Is that adulteration or natural behavior? Rajeswara Rao et al. (2002) mentioned water contents up to 20%, but that level seems too high by following GMP. Responsible for this effect are monoterpenes, and like conifer oils, citrus oils contain higher quantities of these compounds. Citrus expression techniques use a lot of water to spray away the oil/wax emulsion. Centrifugation will separate water and waxes from essential oil. Unfortunately also aldehydes and alcohols contained in the water/wax phase are removed. Cotroneo et al. (1987) observed this effect by comparing oils from manual sponge method without water and the industrial technical methods. An easy method to detect higher value of water in citrus oil is the visible cloudiness and the deposit of waxes when cooled down to 10°C. A validated method to detect the water content in essential oils is the Karl Fischer titration (ISO 11021, 1999).

18.4.2 Ethanol

Mostafa (1990) reports that ethanol is the main alcohol used in moderate quantities to dilute essential oils. Ethanol is a component of rose oil. The process of water/steam distillation forms alcohol in certain quantities. In the chromatographic profile in ISO 9842, 2003, essential oil of rose, the value of ethanol is specified from 0% to 7%, depending on the origin (maxima: Bulgaria 2%, Turkey 7%, and Morocco 3%). The question arises whether the alcohol is coming directly from the process or if the distilled water is washed and extracted by alcohol and this extract is added to the essential oil. If it is the case it is no more an essential oil. Rose oil is a very expensive product and 1% or 2% of added ethanol increases the profit.

18.4.3 Fatty Oils or Mineral Oils

As mentioned in the introduction, adulteration with fatty oils in history is well known. Rochussen (1920) confirms that and mentions paraffin, petrolatum, and castor oil. The easiest way to detect

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was the blotting paper test. Essential oils containing fatty oils leave behind a lasting grease spot. By testing the solubility in ethanol of 90%, a blur occurs, showing the presence of fatty oils. Only castor oil is soluble in ethanol but can be detected by evaluation of nonvolatile residue content method. Since more than three decades, such an adulteration became no more apparent.

18.4.4 High Boiling Glycols

For a long time such adulterations remained undiscovered. High boiling materials like polyethylene glycols could not be detected with normal GC-MS method, even at 280°C column temperature. It takes many hours before these chemicals leave the column and give a long, small hill in chromatogram. Not only expensive essential oils like sandalwood, vetiver, and orris were cut with those chemicals but also extracts like rose absolute. By happenstance the author found the stretching of this rose absolute. Typically this product is of deep red color and its use in white cosmetic cream was impossible. A high vacuum distillation was performed to remove the color, which really seemed to be not a problem. The end temperature was 200°C in high vacuum but still about 20% remained in the retort. Never a component of rose absolute with that boiling point was detected or mentioned in the literature before.* At least in 1974, Peyron et al. reported about the use of hexylene glycol up to 40% in vetiver and cananga oils. By treatment with water, separation by rectification, and liquid gas chromatography, it could be detected. In 1991, John et al. reported the thin-layer chromatography method for detection. This method is simple and effective and can be used for routine analysis.

18.4.5 Oils from Other Parts of the Same Species or Other Species with Similar Essential Oil Composition

This is a very simple method to stretch. Clove leaves are easier to harvest and bring similar oil as the stem or bud oil. Clove bud oil is nearly three times higher in price than clove leaf oil. Therewith the concocting of that oil makes sense. Pimento berry oil and leaf oil are closely related in composition. The price of the berry oil is double the leaf oil; consequently, adulteration is interesting. By GC-MS method and regarding minor components, this immixture will be detected. Eucalyptus oil from China is derived from *Cinnamomum longepaniculatum* (Gamble) 1,8-cineole type. Either the whole oil sold as eucalyptus oil or *Eucalyptus* sp. added is clearly not allowed and must be seen as adulteration.

18.4.6 Related Botanical Species

Something of the kind can only happen in the origin, where biomass is growing and distillation is proceeded. Some plants have relatives, which then might have comparable oil compositions. Ylang oil is produced from the flowers of *Cananga odorata* (Lam.) Hook. F. Thomson forma *genuina*. The other form of *Cananga odorata* is cananga oil with *Cananga odorata* (Lam.) Hook. F. Thomson forma *macrophylla* as plant source. On the market, cananga oil is cheaper than ylang oil. Instead of mixing the distilled oils of both, the flowers are distilled together. Furthermore flowers of climbing ylang-ylang *Artabotrys uncinatus* (Lam.) Merill are sometimes added before distillation. When producing patchouli oil, the vessel is filled with leaves, and to avoid that they stick together during processing, branches of the gurjun tree (*Dipterocarpus alatus* Roxb. Ex G. Don and *Dipterocarpus turbinatus* C.F. Gaertn.) are added to avoid that. As a result, the oil of patchouli is contaminated with α-Gurjunene (up to 3%),which is not part of a pure patchouli oil (private information to the author).

* Based on the authors' private information.

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18.4.7 Fractions of Essential Oils

This is one of the most applied adulteration methods. Fractions arise in all cases, when essential oils or extracts are concentrated, washed out, and rectified or are residues of removal from centrifugation and distillation as well as from recovery of water streams of expressed citrus peels. Heads and tails from rectification are added to similar essential oils, like peppermint terpenes to mint oil. Essential oils that are high in terpene content, cheap, and available on the market are citrus, eucalyptus, *Litsea cubeba*, cornmint and mint, petitgrain, spearmint, vetiver, lavender and lavandin, cedarwood, citronella, clove, and *Corymbia citriodora* terpenes. Especially citrus terpenes from various species are appropriate for stretching. Dugo (1993) recognized the contamination of bitter orange oil by the use of sweet orange and lemon oils and its terpenes. By applying liquid chromatography (LC), high-resolution gas chromatography (HRGC), and GC-MS methods, they confirmed the addition of less than 3% of these components. Limonene is the main component of many citrus oils. On the other side, limonene is a component of nearly all essential oils in variable quantities. Limonene is a chiral terpene; chirality describes a basic property of nature. Some molecules show the property to have three-dimensional structures that possess a chiral center. These molecules show a basic property of nature, to look like image and mirror image, but both cannot be aligned. It is a matter of common knowledge that chemical compounds in essential oils tend to be chiral and the preference of one form can be observed. In contrary synthetic molecules are racemic, meaning both forms of the molecule are balanced. This is a real fingerprint for detection of adulterations with terpenes owing different chiral properties. On normal GC columns a separation of chiral molecules is impossible. With the use of columns coated with modified cyclodextrins, those mirror-imaged molecules will be separated and appear as two different signals. In 1995 bergamot essential oils, sold in 10 mL bottles to end users, were subject for investigation of the German journal ÖKO Test. This magazine, dealing with naturality and safety of consumer products, charged the University of Frankfurt, Prof. Mosandl, to check the authenticity of the oils by chiral separation of linalool and linalyl acetate. Thirty-seven of fifty-three samples could be confirmed not being natural and "pure." In a private letter of Prof. König (University of Hamburg) to the author, he confirmed: "Linalool and linalyl acetate are present in bergamot oil only in pure R-enantiomers. That for bergamot oil—unnatural—(*S*)-linalyl acetate cannot be found even in traces." The invention of this technique by König, Mosandl, Casabianca, and Dugo, improved by Mondello, Bicchi, and Rubiolo, was a huge step to diminish the cases of adulterations in essential oils. Limonene occurs in D-, L-, and DL-form in nature. Limonene in sweet orange oil is always in D-form; most of the pine oils show pure L-forms. For the detection of stretching, other terpenes and terpene alcohols are used like α- and β-thujene, α- and β-pinene, α- and β-camphene, α- and β-sabinene, δ3-carene, linalool, β-borneol, myrtenol, linalyl acetate, menthol, *cis*- and *trans*-terpineol, 1-phenylethanol, nerolidol, terpinen-4-ol, and many more. Whenever the chiral distribution differs from normal proportion, a manual intervention was made. However, the last sentence has to be relativized carefully. Chiral composition of camphor and borneol in natural essential oil of rosemary is very variable, depending on the origin! Another method of stretching is the use of distilled residues of expressed citrus peels. After citrus production process, some quantities of essential oil components still can be found in the "waste products." In addition, Reeve et al. (2002) report of the use of distilled mandarin oil, produced from recovered water streams from spiking and pressing. Because of the contact with acidic juice, the composition of such an oil shows significant difference in composition from peel oil. This oil can be sold as "distilled" mandarin oil, but never be used to be blended with pressed oils. Such a handling must be seen as adulteration and is not covered by ISO definitions.

18.4.8 Natural Isolates

Single chemicals can be derived from essential oils by methods of fractioning and rectification. As this procedure is from a natural source, of course, this chemical must be characterized as natural.

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Numerous components are offered on the market like citral from *Litsea cubeba* oil, geraniol from palmarosa oil, linalool from ho oil, coriander oil or lavandin, pinenes from different *Pinus* species, citronellal from *Corymbia citriodora*, cedrol from cedarwood, or even the santalols from different sandalwood species. All these are added to "finish" essential oils. As already mentioned before, synthetic chemicals can no more be applied as enantiomeric separation is a state of the art today and will convict the matter of fact of adulteration. By using synthesized, correct chiral compounds, the detection is hardly to be recognized but with NMR method, but this is an expensive analysis.

18.4.9 Chemically Derived Synthetic Compounds, Which Are Proved to Appear in Nature

Compounds formerly named "nature identical" are to be defined by law since some years as synthetic products. These molecules have been found in nature by analytical proof and are published in an authorized scientific journal. The term "nature identical" is no more valid and allowed in Europe in relation to flavor and fragrance substances. Such molecules are identical with those appearing in nature but are produced by a synthetic process. These processes contain undesired by-products. The use of such synthetic compounds is easy to detect, as by-products from manufacturing can easily be detected by GC-MS systems. On the other hand, chiral separation will help to confirm adulteration.

18.4.10 Steam Distilled Residues from Expression

After an expression process of citrus oils either in exhausted peels or in centrifugation residues, carryovers of the volatiles will remain. These can be removed by distillation with high-strung steam, and oils acquired are colorless and still have the smell of the starting material. Components are similar those of the expressed oils but contain for reason of the production method some oxidized chemicals. Nevertheless these oils are used to adulterate the expressed oils, and with that process the naturality per definition by ISO standard is lost. The main ingredient is limonene, followed by myrcene and γ-terpinene. Traces of aldehydes still can be found. Such adding can be detected by observing higher values of oxidation compounds.

18.4.11 Enzymatically Produced Chemicals (Natural by Law)

Enzyme is defined as "Any of numerous proteins or conjugated proteins produced by living organisms and functioning as biochemical catalysts" (The Free Dictionary n.d. [c]). Enzymes of microorganisms in a medium with added nutrients produced such molecules. Isolation has to be done by any physical process to isolate the desired molecule designed by the biosystem. Although this manufacturing process is authorized within the applicable legal requirements for the use of such enzymes in the EU, it is somewhat uncertain. These processes will as a rule not happen in nature. Within a plant, production of hydrocarbons takes place without microorganisms by conversion of a starting material and by enzymatic reaction within a cell system. The question arises if that molecule from microorganisms can really be named as natural, particularly if it is generated from "any" starters. In the authors' mind, adding such a compound must also be named as adulteration within the definition of essential oil, which does not allow such a process and any additives.

At least it must be mentioned that the mixture of essential oils from the same species but from different geographical sources cannot be called an adulteration. They fulfill the requirements of standards as long as the specific provenance is not laid down in the specification. Lavender oil from France can be mixed up with Chinese, Bulgarian, and Russian origin and is still lavender oil (*Lavandula angustifolia* Mill.), but may not be sold as "French lavender." Furthermore if an essential oil from the same species but from another geographical area is recognizably different in

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composition, it must be mentioned in standards and certificates. One example is geranium oil from North Africa compared to other origins. 10-epi-γ-Eudesmol is only present in North African oil. The cause for this is adaptation to different conditions in a longer period.

18.5 METHODS TO DETECT ADULTERATIONS

18.5.1 Organoleptic Methods

18.5.1.1 Appearance and Color

Appearance is the visual aspect of a thing or person. In this case it is the appearance of the "essential oil," starting with color, going on with mobility, and finally with the odor itself. The color of essential oils is dependent on the starting material. Citrus oils are colored weak yellowish with lemon oil, light green to darker green with bergamot oil, and orange to brownish red with orange and mandarin oils. The color is dependent on the degree of ripeness of the fruits but they alter with storage and influence of light and warmth. Hereby the age of such an essential oil can be detected. Colors too will appear in hydrodistilled essential oils. The normal case for these oils is colorlessness accompanied by mobile fluidness. Weak yellow color can appear in oils like cardamom, rosewood, tarragon, or turpentine. Oils of lavender and lavandin are pale yellow and mobile; sandalwood oil is almost colorless to golden yellow but viscous. Lemongrass and citronella Java-type oils are pale yellow to yellowish brown; the latter is slightly viscous. Geranium oil has various shades, starting from amber yellow to greenish yellow. Yellow to light brown is the rectified oil of clove leaf, but the crude oil is black. Vetiver and patchouli oils are yellow to reddish brown; both are viscous or even highly viscous. The oils of thyme (*Thymus vulgaris* L. and *Thymus zygis* (Loefl.) L.) are red. The color results from a reaction between thymol and iron of the still. Divergent colors have to be observed critical as these can be a result of aging. Citrus oils can be clear at ambient temperature but becomes cloudy at lower temperatures. Sometimes waxes can undergo precipitation by storage under 4°C.

18.5.1.2 Odor

The odor is the most important factor for the application of essential oils. The highest quantity of usage is in the flavor and fragrance industry. Hatt et al. (2011) confirmed that the human sense of scent is the most important, even if the visual is dominating today. Smelling influences the human being by affecting the limbic system and is responsible for feelings and instincts, to trigger hormonal effects and activate pheromone production. The fact, that the human species has 350 gene receptors for smelling but only 4 for seeing will demonstrate the vital necessity of this sense.

In times of high sophisticated analytical technique with highest resolutions, the human sense of smell is not replaceable. Especially small traces of aromatic chemicals with very low odor threshold are quickly identifiable. Examples are vanillin from vanilla beans and maltol, found in malt and caramel. For perfumer beginners, a single chemical is easy to recognize again. Essential oils are multicomponent mixtures, varying sometimes in wider limits. In spite of electronic noses a well-trained perfumer can recognize more nuances. Common human nose might be duped by adulterations, but not those of professional perfumers. At least the sense of taste is used to recognize adulteration. A good example is rose oil. Part of a drop applied to the tongue and mixture with synthetic geraniol or citronellol reflects in soapy and bitter taste.*

18.5.1.3 Physical–Chemical Methods

These are inevitable in spite of many analytical certificates according to regulations. In history these values have been the only reference points to confirm naturalness. Although the bandwidth is

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often too wide, the values can be used as simple and easy to establish proof-samples. Gross mistakes and adulteration can be detected as well as aging of the oil. A series of ISO standards have been established especially for the quality control of essential oils:

Relative density (*ISO 279*): This is defined as the relation between the mass of a defined volume of substance at 20°C and the mass of a comparable volume of water at the same temperature.

Refractive index (*ISO 280*): This is the ratio of the sine of the angle of refraction, when a ray of light is passing from one medium into another. Three decimals are obligatory for the result.

Optical rotation (*ISO 592*): This is the property of defined substances, to turn the plane of polarized light.

Residue on evaporation (*ISO 4715*), 1978: This is the residue of the essential oil after vaporization on the water quench, using defined conditions and is expressed in percent (*m/m*).

Determination of acid value (*1242*): This value shows the number of milligrams of potassium hydroxide required to neutralize free acid in one gram of essential oil.

Miscibility with ethanol (*ISO 875*), 1999: For that key figure, a defined quantity of an essential oil is added to an also defined mixture of distilled water and ethanol at 20°C. As result a blur occures visual. The value before obscuration is the value for a clear solution.

Determination of ethanol content (*ISO 17494*): This is the method of detecting the quantity of ethanol contained in an essential oil by GC analysis on a suitable column.

Determination of the carbonyl value—free hydroxylamine method (*ISO 1271*): The determination is done by converting the carbonyl compounds to oximes by reaction with free hydroxylamine liberated in a mixture of hydroxylammonium chloride and potassium hydroxide.

Determination of water content—Karl Fischer method (*ISO 11021*): A reagent (Karl Fischer reagent without pyridine) is used for the reaction of the absorbed water from an essential oil. The reagent is produced by titration, using a Karl Fischer apparatus. The final result of the reaction is obtained by an electronic method.

Determination of phenol content (*ISO 1272*), 2000: The transformation of phenolic compounds into their alkaline phenol esters, then soluble in aqueous phase, in a defined volume of essential oil is proceeded. The volume of unabsorbed portion is measured.

Determination of peroxide value (*ISO 3960*): The peroxide value is the quantity, expressed as oxygen, to oxidize potassium iodide under specific conditions, divided through the mass of testing substance.

Determination of freezing point (*ISO 1041*): This is the highest temperature during freezing of an undercooled liquid.

Iodine number: Kumar and Madaan (1979) report in their chapter the use of iodine number. This method is a measure of the unsaturation of a substance (as an oil or fat) expressed as the number of grams of iodine or equivalent halogen absorbed by 100 g of the substance (Merriam-Webster, n.d.). By using the iodine monobromide-mercuric acetate reagent for iodination, a much better result could be achieved, as reported by Kumar. With this method adulterations on essential oils can be successfully detected.

Thin-layer chromatography: This method belongs to wet chemistry (see Section 18.5.2).

All earlier-mentioned methods are still involved in the concerning standards and are useful tools to confirm naturalness and purity of essential oils.

18.5.1.4 Calculation of Relationship Coefficient

This method might be derided by some people, but once a relationship is recognized, it will help to detect mixtures with natural fractions or isolates. Touche et al. (1981) presented reference factors

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for identification of adulteration in French lavender. The ratio between (*Z*)-β-ocimene and (*E*)-β-ocimene (R_1), (*E*)-β-ocimene and 3-Octanone (R_2), and linalool + linalyl acetate to lavandulol + lavandulyl acetate had been determined using GC data of production values over more than 5 years. Schmidt (2003) showed in accordance to that the differentiation of the minor components of lavender oils as the possibility to detect even the provenance of the oil. Of course such a calculation of relationship can be done for many other essential oils. As long as typical minor substances are present, it is a valuable tool for genuineness control.

18.5.2 Analytical Methods

18.5.2.1 General Tests

Before the availability of sophisticated analytical instrumentation, only rather simple tests could be carried out to detect adulterations in essential oils (EOs), and it can be assumed that at that time many falsifications went undetected and adulterations were widespread. To determine the identity and purity of EOs, several elementary methods are described in the European Pharmacopoeia (Ph. Eur.) and ISO Standards (ISO/TC 54).

Physicochemical properties like relative density (Ph.Eur. 7.0/2.02.05.00, ISO 279), refractive index (Ph.Eur. 7.0/2.02.06.00, ISO 280), optical rotation (Ph.Eur. 7.0/2.02.07.00, ISO 592), and flash point (ISO/DIS 3679:2013-08) should be within certain limits and numerical values can be found in the literature, on the Internet, and in ISO standards. Solubility tests can indicate the presence of contaminants (solubility in ethanol ISO 875), for instance, a turbidity of a solution of an EO in carbon disulfide is an indication of water (Ph.Eur. 7.0/02.08.05, ISO 11021). To detect fatty oil adulterations, a drop of the EO is placed on a filter paper and after 24 h no visible grease spot must be left behind (Ph.Eur. 7.0/2.08.07.00). Determination of the evaporation residue serves the same purpose and detects nonvolatile or low volatile compounds (Ph.Eur. 7.0/2.08.09.00, ISO 4715). An instrumentally more demanding method would be thermogravimetry

Basic chemical tests can also provide useful information on the properties of EOs. Ester and saponification value (Ph.Eur. 7.0/2.05.02.00, ISO 7660, 1983), content of free and total alcohol (ISO 1241), carbonyl value (ISO 1279, 1996), and phenol content (ISO 1272, 2000) are helpful indicators of identity and purity of EOs.

Last but not least sensory evaluation is important especially in perfume houses and should be carried out by experts or a panel of fragrance professionals most effectively done with the help of reference samples of known status.

But some parameters alone determined by the earlier-mentioned methods tell us nothing about the composition of the tested EO. Therefore, separation of the oil into their individual components is necessary.

18.5.2.2 Thin-Layer Chromatography

One of the oldest and easiest separation methods is thin-layer chromatography (TLC), which nevertheless has the disadvantage of having a quite low separation efficiency. A drop of the EO is placed at one end of a plate (glass, aluminum foil) coated with a thin layer of an adsorbent (silica gel, diatomaceous earth, aluminum oxide, cellulose) as the stationary phase and placed in a glass chamber filled with a small amount of an appropriate solvent as a mobile phase. The solvent is drawn up the plate through capillary forces and separates the oil into several fractions. These fractions or spots can be made visible by derivatization with a chromophoric reagent or by inspection under UV light. A reference oil of known purity and composition must be analyzed simultaneously or under the same conditions for comparison (Ph. Eur. 7.0/2.02.26.00); (Jaspersen-Schib and Flueck, 1962; Phokas, 1965; Atal and Shah, 1966; Sen et al., 1974; Kubeczka and Bohn, 1985; Nova et al., 1986; John et al., 1991).

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18.5.2.3 Gas Chromatography (GC, GLC, HRGC, GC-FID, GC-MS)

The analysis of natural products can be demanding especially in the field of EO research since an EO can contain 300 or more components at a concentration ranging from more than 90% to a few ppm or less of the total oil amount. A separation technique of high efficiency is needed and gas liquid chromatography (GLC) or simply GC is one of the most importantly used analytical methods for volatile compounds and is especially suited in EO and fragrance analysis. With the introduction of capillary columns, the separation performance increased dramatically and theoretical plate numbers of more than 1×10^5 are commonplace and provide gas chromatograms of high resolution (HRGC).

A small amount of the EO (nanograms to micrograms) is injected with a microliter syringe into a closed evaporation chamber (injector) held at elevated temperature (>200°C) where the sample evaporates and the vapor is transferred by an inert carrier gas (N_2, H_2, He, Ar) as the mobile phase to a quartz capillary column of an internal diameter ranging from 0.05 to 0.53 mm and a length between a few meters up to a hundred meters or more. The inner wall of the capillary is coated with a thin film of a liquid phase (polysiloxane, polyglycol) in which separation takes place through permanent absorption and desorption of the sample compounds between liquid and mobile phase. The column is mounted in an oven either held at constant temperature or more often programmed at a constant rising temperature rate and permanently flushed with the carrier gas. The substances pass through the column and leave at a reproducible time (retention time) and then need to be registered by an appropriate detector. The most common and versatile detector for organic compounds is the flame ionization detector (FID) in which the eluted substances are burnt in a tiny hot hydrogen/air flame producing charged carbon ions that are collected on an anode. The resulting current is registered after amplification by an electrometer. Amplitude and signal (peak) area are proportional to the amount of the corresponding compound. Retention times depend on many device parameters, and therefore, instrument-independent retention indices calculated according to Kovats (1958) or Van Den Dool and Kratz (1963) are used instead and can be compared with literature data for substance identification. To verify identification, a second analysis on a column of different polarity must be performed, and retention times or indices on both columns must match the data from the assumed compound or of those from a reference substance. This two-column approach is also useful to detect peak overlapping, which occurs quite often on single-column GC analysis of natural complex substances. Substance amounts are calculated from the individual peak areas as a percentage of the total peak areas of all substances multiplied by a substance-dependent correction factor, which accounts for the different sensitivities of each compound to FID detection.

These correction factors should be determined at least for the major compound found in an EO relative to a standard substance (preferably a *n*-paraffin) whose factor is typically set to 1. Unfortunately many EO compounds are not available commercially in pure form or easily synthesized, so for convenience all correction factors are set to 1 to a first approximation.

Chromatographic profiles and percentage composition of all important commercial essential oils and many oil-bearing plants can be found in the ISO standards and the literature of essential oil research (Formácek, 2002; Kubezka and Formácek, 2002; Adams, 2007), and genuine EOs should fit into this image within certain limits. Chemical profiling in this manner can detect adulterations if additional or missing peaks are found or percentage composition deviates substantially. Addition of synthetic compounds can in some cases be disclosed by the presence of by-products or impurities left behind from the synthesis pathway. Dihydro linalool, for example, is an intermediate in the industrial production of linalool and is always present in small amounts (≪1%), and if a trace of this substance is found in an EO, it is a clear indicator of an adulteration with synthetic linalool.

However, fingerprinting of EOs by GC with FID detection alone cannot reveal the chemical identity of detected peaks if any deviations from the expected profiles occur. In that case further

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information is needed besides the chromatographic data, and mass spectrometry provides detailed information on the structure of the separated compounds. High-resolution gas chromatography coupled with a mass spectrometric detector (HRGC-MS or simply GC-MS), most commonly quadrupole ion trap detectors in EO analysis, together with sophisticated chromatographic software and special mass spectral libraries of essential oil components (Adams, 2007; König, Joulain, Hochmuth 2004, Mondello, 2011) separates and identifies most components of an EO. Since some classes of compounds show very similar mass spectra, like some groups of mono- and sesquiterpenes, retention indices must be taken into account as a second criterion for an unequivocal identification. Usually a GC-MS run is performed for identifying the EO components and a second GC-FID run for peak area, respectively, for percentage composition determination. Normally this is done on two different instruments. Identical capillary columns must be used in both GCs, and device parameters must be adjusted properly to obtain closely similar chromatographic profiles for both detectors to facilitate peak allocation between the two chromatograms. Peaks identified in the mass spectrometry (MS) chromatogram must be correctly assigned in the FID chromatogram for peak integration. Sometimes this is proving difficult since separation on two instruments is never exactly equal especially if one column ends up in a high vacuum (MS) and the other at atmospheric pressure (FID). Therefore, a series of closely eluting peaks may not be resolved in the same manner on both columns. To overcome this problem, an FID-MS splitter can be used since here the separation takes place on one GC column and the effluent is split to both detectors, MS and FID. To detect peak overlapping, the same procedure should be undertaken on another capillary column of different polarity. In EO analysis GC separations are preferably performed on 95% dimethylpolysiloxane/5% diphenylpolysiloxane and on Carbowax 20M columns. In the end you come up with two FID and two MS chromatograms, each of the two carried on different capillary columns. These results in two analyses that should for the most part coincide and accept the overlapping peaks on the other GC column. EO analysis performed in this way confirms the chemical composition of an EO and detects adulterations with exogenic substances if they are amenable by GC, that is, volatile. Adulterations with gas chromatographic undetectable substances like very high boiling vegetable or mineral oils can be disclosed by a change in the percentage composition or a too low total peak area. Specific marker compounds or a diastereomeric isomer distribution can also be used for authentication of EO (Teisseire, 1987).

In some unusual EOs not all detected peaks can be identified by MS library search because of missing entries but, with the necessary experience, it can often be estimated in which group of natural substances these peaks belong by looking at the mass spectrometric pattern. All in all an extensive experience in EO analysis and a thorough knowledge of the scientific literature is needed to evaluate the authenticity of an EO and detect adulterations. If this is the case the analyst should be in a position to disclose falsifications with cheaper or inappropriate EOs, synthetic natural substances, solvents, or other unnatural chemicals.

18.5.2.4 Chiral Analysis (Busch and Busch, 2006)

Many EOs contain substances with asymmetric carbon atoms, that is, there are two molecular forms of these molecules that behave like image und mirror image and cannot be brought into alignment. This is the reason why EOs rotate the oscillating plane of light and the resulting overall optical rotation is measured by a polarimeter. However, the enantiomeric distribution of single substances cannot be determined in this way because separation is needed in the first place. This will be enabled by GC, and additional separation of optically active compounds into their enantiomers can be achieved if a chiral selector is added to the stationary phase of the GC column. Cyclodextrin derivatives have proven to be very useful and chiral capillary columns with different optically active cyclodextrin selectors are commercially available. The enantiomeric separation should be done at rather low temperature since the interaction between the chiral molecules and the chiral selector is rather weak; therefore, the temperature gradient of the GC program should be low and the carrier

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gas flow is higher than the optimal flow. If the appropriate chiral GC column is used, the enantiomeric distribution of certain EO components can be determined, and since nature often prefers one enantiomer over the other, the determination of the enantiomeric excess is a valuable tool to detect adulterations by synthetic components that are racemic and change the enantiomeric ratio. The optical purity of authentic EOs can be found in the literature (Mosandl, 1998; Busch and Busch, 2006; Dugo and Mondello, 2011).

18.5.2.5 GC-GC and GC×GC (Two-Dimensional Gas Chromatography, ^{2}D GC)

Gas chromatography is one of the most widely deployed methods in analytical chemistry to investigate organic sample material due to its simple ease of use, the ready availability of sophisticated inexpensive instrumentation, and the large amount of qualitative and quantitative information that can be retrieved if the appropriate configuration is employed. Especially the high separation efficiency for volatiles makes GC very suitable to investigate complex mixtures and sample matrices. But for some applications, the separation performance is not sufficient when it comes to very complex mixtures like odors, flavors, crude oil products, and foodstuff. Co-elution with other analytes or sample matrix elements causes problems in detection and quantitation especially when the analytes differ greatly in their concentration. This problem can be solved by cutting out the co-eluting part of the chromatogram and a subsequent second chromatographic separation of the excised effluent preferably on a stationary phase of different polarity. This technique, called heart-cutting or two-dimensional GC (^{2}D GC or GC-GC), is done with the help of a diverting valve or a Deans switch. The sought-after substances are then resolved in the second GC column.

An even more elaborate technique recently developed not only cuts out one or several parts of the first GC column but virtually cuts the whole 1D chromatogram into small equal pieces (each several seconds long), refocuses each effluent, and separates it on a short second GC column within a very short time (several seconds). Refocusing, which here means stopping the effluent of the first column for several seconds onto a very small area and then releasing the focused substances into the second column, is done with a cryogenic modulator, and this procedure is repeated until all substances are eluted from the first GC column. The overall separation efficiency is calculated by multiplying the theoretical plate numbers of the two columns, which results in rather large figures and a separation efficiency, which cannot be achieved by simple GC alone. Instruments and the appropriate data acquisition software for this comprehensive ^{2}D GC (or GC×GC) are now commercially available. Instead of a 1D chromatogram, a 2D contour plot is generated, and since the "half-widths" of substances are very small, detection must proceed very fast, so in case a mass spectrometer is used as a detector, a fast scanning quadrupole or a time-of-flight MS (TOF-MS) must be chosen. Such an instrument arrangement like GC×GC-TOF-MS leaves almost nothing to be desired for EO analysis (Marsili, 2010).

18.5.2.6 ^{13}C NMR (Nuclear Magnetic Resonance)

Another useful method to validate the identity of an EO offers the spectroscopy of the magnetic properties of ^{13}C nuclei. ^{13}C NMR spectroscopy is a very useful technique regularly used to elucidate the structure of individual substances and has been applied by Kubeczka and Formácek (2002) to a large number of essential oils and individual reference compounds. Even though it is not very common to apply this technique to substance mixtures, as it is with an EO, the spectrum of a genuine oil is very distinct and characterized by the chemical shifts, signal multiplicities, and intensities of all components. Functional groups can be identified by chemical shifts and provide information on chemical structures of individual substances within the oil. No separation of the oil into its components is necessary, and therefore, the approach is simply measuring a solution of the oil in an NMR tube and comparing the spectrum with literature data or reference oils and compounds. Unfortunately NMR instruments are rarely found in traditional analytical laboratories, and thus, this method has not gained the significance in the field of EO analysis it deserves.

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18.6 IMPORTANT ESSENTIAL OILS AND THEIR POSSIBLE ADULTERATION

18.6.1 Ambrette Seed Oil

This is a very expensive oil. The main ingredients are (*E*,*E*)-farnesyl acetate (≈60%) and ambrettolide (≈8.5%). Synthetic ambrettolide is used as a fixative in perfumery, is nearly odorless but has exalting properties. Compared to natural ambrette seed oil, the price of ambrettolide is only 10%. Detection of naturality can be done by isotope ratio mass spectrometry (IRMS).

18.6.2 Amyris Oil

ISO standard 3525 shows character and data for this oil. Blending (professional term within dealers and perfumers for adulterating) is done by Virginia cedarwood oil, α-terpineol, and copaiba balm. Also, elemol distilled from elemi resin is used. Detection is done by GC-MS.

18.6.3 Angelica Oils

The fruit oil contains up to 76% of β-phellandrene and α-pinene (13%) as main ingredients. As no chiral values for the β-phellandrene are described in literature, it must be assumed that adulteration is done by this compound. β-Phellandrene is naturally available by geranyl diphosphate cycling (BRENDA, BRaunschweig ENzyme DAtabase) 2007. For adulteration copaiva balm, gurjun balsam, lovage root oil, and amyris oil were used in the past. Cheap α-pinene can also be used to "improve" the composition.

18.6.4 Anise Fruit Oil

ISO standard 3475 shows character and data for that oil. This oil often is produced not only from the fruits but also from the whole aerial part. Values of *cis*- and *trans*-anethole are then reduced in smaller amount. Adulteration with star anise oil can be easily detected. Anise fruit oil does not contain any foeniculin, but star anise oil contains up to 3% (ISO 11016). On the other hand pseudo-isoeugenol-2-methylbutyrate is a component of anise fruit oil (0.3%–2%, ISO 3475) and does not appear in star anise oil. The midratio of *cis*-anethole to *trans*-anethole in anise seed oil in relation to star anise oil is 0.3%:0.6%. Synthetic anethole, fennel terpene limonene (80°), and terpineol were used for blending.

18.6.5 Armoise Oil

This oil is often blended with α- and β-thujones from cheaper sources like *Thuja orientalis*. Furthermore camphor or white camphor oil and camphene are used. GC-MS method is used to detect these adulterations.

18.6.6 Basil Oils

ISO standard 11043 shows character and data for the methyl chavicol–type oil. Synthetic methyl chavicol is used to adulterate that oil and can be detected by NMR. Basil oil from linalool type will be adulterated by synthetic linalool but can easily be detected by chiral separation. Casabianca (1996) found the minimum value *R*-(–)-linalool with 99.8% and is congruent with the authors' results.

18.6.7 Bergamot Oil

ISO standard 3520 shows character and data for that oil. As mentioned in the text, this oil was adulterated with synthetic linalool and linalyl acetate but also with bergamot terpenes and distilled oil

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from bergamot peel residues. Blending was done with limonene (80°), nerol, geranyl acetate, petitgrain oil Paraguay, rosewood oil, and citral from *Litsea cubeba* oil. This oil was subject to many studies with chiral separation. König, 1992, Mosandl, 1991, Dugo, 1992, and Casabianca, 1996a, reported similar values for linalool and linalyl acetate. (*R*)-(−)-linalyl acetate is present always in purity higher than 99.0% and (*R*)-(−)-linalool with a minimum of 99.0% too. (*S*-pinene value varies between α)-(−)- 68.0% and 71.1%, (*S*-pinene between 91.1% and 92.6%β)-(−)-, and (*R*)-(+)-limonene between 97.4% and 98.0% (Juchelka and Mosandl, 1996a). Blending is done by terpinyl acetate, citral synthetic, *n*-decanal, *n*-nonanal, nerol, limonene (80°), and sweet orange terpenes. Dugo and Mondello (2011) published the following data for chiral ratios: (*S*)-thujene (0.5%–1.3%):(α)-(+)-(*R*)-thujene (98.7%–99.5%); (α)-(−)-(*R*)-(+)-α-pinene (31.0%–36.1%):(*S*)-(−)-α-pinene (63.9–69.0); (1*S*,4*R*)-(−)-camphene (85.7%–92.7%):(1*R*,4*S*)-(+)-camphene (7.3%–14.3%); (*R*)-(+)-β-pinene (7.6%–10.3%):(*S*)-(−)-β-pinene (89.7%–92.4%); (*R*)-(+)-sabinene (13.7%–19.8%):(*S*)-(−) (80.2%–86.3%); (*R*)-(−)-α-phellandrene (43.1%–54.7%):(*S*)-(+)-α-phellandrene (45.3%–56.9%); (*R*)-(−)-β-phellandrene (24.1%–36.9%):(*S*)-(+)-β-phellandrene (63.1%–75.9%); (*S*)-(−)-limonene (1.2%–2.1%):(*R*)-(+)-limonene (97.9%–98.8%); (*R*)-(−)-linalool (97.8%–99.5%):(*S*)-(+)-linalool (0.5%–2.2%); (*S*)-(−)-citronellal (>98%):(*R*)-(+)-citronellal (<2%); (*R*)-(−)-linalyl acetate (99.1%–99.9%):(*S*)-(+)-linalyl acetate (0.1%–1.0%); (*S*)-(+)-terpinen-4-ol (22.4%–44.7%):(*R*)-(−)-terpinen-4-ol (55.3%–77.6%); (*S*)-(−)-α-terpineol (14.0%–68.5%):(*R*)-(+)-α-terpineol (31.5%–86.0%); (*S*)-(−)-citronellol (12.0%–20.0%):(*R*)-(+)-citronellol (80.0%–88.0%); (*S*)-(−)-terpinyl acetate (36.0%–44.0%):(*R*)-(+)-terpinyl acetate (56.0%–64.0%).

18.6.8 Bitter Orange Oil

ISO standard 3517 shows character and data for this oil. In the past, sweet orange oil was used as well as orange terpenes and distilled bitter orange residues from production for adulteration. Limonene of high purity from other citrus fruits was also applied. Dugo (2011) shows the similarity of the components between bitter and sweet orange oil. McHale et al. (1983) report the use of grapefruit oil, as it contains higher concentrations of coumarins and psoralens. Today the adding of purified components from other citrus sources is used. Mingling up with sweet orange oil can be detected by measuring the δ-3-carene and camphene content. As bitter orange oil contains only traces of δ-3-carene, the sweet oil goes up to 0.1%. Also, the ratio δ-3-carene/camphene can be used for detection (Dugo et al., 1992). Chiral values for components are reported by Dugo and Mondello (2011): (*R*)-(+)-α-pinene (89.7%–97.4%):(*S*)-(−)-α-pinene (2.6–10.3); (1*S*,4*R*)-(−)-camphene (35.8%–47.6%):(1*R*,4*S*)-(+)-camphene (52.4%–64.2%); (*R*)-(+)-β-pinene (6.1%–7.9%):(*S*)-(−)-β-pinene (92.1%–93.9%); (*R*)-(+)-sabinene (49.4%–80.6%):(*S*)-(−)-sabinene (19.4%–50.6%); (*R*)-(−)-α-phellandrene (60.1%–74.9%):(*S*)-(+)-α-phellandrene (25.1%–39.9%); (*R*)-(−)-β-phellandrene (0.6%–5.7%):(*S*)-(+)-β-phellandrene (94.3%–99.4%); (*S*)-(−)-limonene (0.5%–0.8%):(*R*)-(+)-limonene (99.2%–99.5%); (*R*)-(−)-linalool (61.2%–89.8%):(*S*)-(+)-linalool (10.2%–38.8%); (*S*)-(−)-citronellal 42.5%:(*R*)-(+)-citronellal (57.5%); (*R*)-(−)-linalyl acetate (99.2%–99.4%):(*S*)-(+)-linalyl acetate (0.6%–0.8%); (*S*)-(+)-terpinen-4-ol (67.5%–71.5%):(*R*)-(−)-terpinen-4-ol (28.5%–32.5%); (*S*)-(−)-α-terpineol (6.6%–29.8%):(*R*)-(+)-α-terpineol (93.4%–70.2%).

18.6.9 Bitter Orange Petitgrain Oil

ISO standard 8901, 2010 shows character and data for this oil. This is also known as "petitgrain oil bigarade." Adulteration is done by using petitgrain oil from Paraguay, heads and tails from fractioning of that oil (to receive the petitgrain oil "terpene free"), and rectified orange terpenes and limonene. Blending is done with synthetic linalool, linalyl acetate, geraniol, geranyl acetate, and α-terpineol. Detection is done by GC-MS as well as by multidimensional enantiomeric separation systems. Juchelka (1996) published the following chiral ratio values: (*R*)-(−)-linalyl acetate (98.0%–99.1%):(*S*)-(+)-linalyl acetate (0.2%–2.0%); (*R*)-(−)-linalool (66.4%–90.2%):(*S*)-(+)-linalool

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(9.8%–33.6%); (*S*)-(−)-α-terpineol (26.4%–28.4%):(*R*)-(+)-α-terpineol (71.6%–73.6%). Mondello and Dugo (2011) reported following chiral ratios: (*R*)-(+)-α-pinene (6.7%–12.0%): (*S*)-(−)-α-pinene (93.3%–88.0%); (*R*)-(+)-β-pinene (0.1%–1.1%): (*S*)-(−)-β-pinene (98.8%–99.9%); (*S*)-(−)-limonene (29.2%–39.2%): (*R*)-(+)-limonene (60.8%–70.8%); (*R*)-(−)-linalool (66.4%–90.2%):(*S*)-(+)-linalool (9.8%–33.6%); (*R*)-(−)-linalyl acetate (93.4%–99.1%):(*S*)-(+)-linalyl acetate (0.9%–6.6%); (*S*)-(+)-terpinen-4-ol (47.9%–67.4%):(*R*)-(−)-terpinen-4-ol (32.6%–52.1%); (*S*)-(−)-α-terpineol (27.5%–28.4%):(*R*)-(+)-α-terpineol (71.6%–72.5%).

18.6.10 Cajeput Oil

The oil obtained from *Melaleuca cajuputi* Powell is the natural variety. To adulterate this oil, other species like *Eucalyptus* ssp. or *Cinnamomum camphora* (1,8-cineole type) are used. Eucalyptus terpenes, α-phellandrene, and α-terpineol were used for blending. Detection is done by GC/MS with the smaller components like α-selinene, α-humulene, and α-terpineol.

18.6.11 Camphor Oil

This oil is produced from *C. camphora* Sieb. wood in China and Taiwan. Many chemotypes and also varieties from *Cinnamomum* species exist, but the true camphor oil is meant. This oil is rarely adulterated because of the cheap price but, fractioned and enriched in camphor up to 90%, is used to mix up camphor containing oils like rosemary, Spanish sage, and spike lavender. Smallest traces of safrole (a carcinogenic compound) shows the use of *C. camphora* higher boiling fractions.

18.6.12 Cananga Oil

ISO standard 3523 shows character and data for the oil, produced from *C. odorata* (Lam.) Hook. F. et Thomson forma *macrophylla*. It can be adulterated by linalool from lavandin oil showing nearly equal chiral values and β-caryophyllene from clove oil fractions. Blending is done with benzyl acetate, linalool, α-terpineol, geraniol, α-terpinyl formate, methyl benzoate, and allo-ocimene, all synthetically produced further from Virginia cedarwood oil, copaiva balm oil, clove leaf oil, and gurjun balsam oil. Cananga oil is used to adulterate ylang-ylang oil, as yield is higher and the flowers have a higher distribution in nature. Detection is done by GC/MS and by multidimensional chiral separation. Bernreuther et al. (1991) mentions the ratio for linalool with (*S*)-(+) 2% and (*R*)-(−) 98%.

18.6.13 Caraway Oil

ISO standard 8896 shows technical data for that oil. Synthetic carvone is used for adulteration. European pharmacopoeia requires contents of (+)-limonene 35.5%–45.0%, (+)-carvone 50.0%–65.0%, and (−)-carvone with a maximum of 0.7% for a pure quality. ISO 8896 standard evaluated a content of *cis*-dihydrocarvone from 0.3% to 1.2%.

18.6.14 Cardamom Oil

ISO standard 4733 shows character and data for that oil. This oil is produced mainly in India, Sri Lanka, and Guatemala. Adulteration is done by 1,8-cineole from eucalyptus or camphor oil, α-terpinyl acetate, and linalool. The adding of 1,8-cineole is hard to be discovered; linalool ratio is reported by Dugo (1996) and Casabianca (2011) to be between 7% of (*R*)-(−)-linalool to 93% of (*S*)-(+)-linalool and 100.0% (*R*)-(−)-linalyl acetate to 0.0% (*S*)-(+)-linalyl acetate. The presence of δ-terpinyl acetate seems to show adulteration as to nowadays it is only detected in clary sage oil. δ-Terpinyl acetate is a side product in synthetic terpinyl acetate.

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18.6.15 Cassia Oil

ISO standard 3216 shows character and data for the Chinese type oil. Main component is *trans*-cinnamaldehyde. Synthetic cinnamaldehyde as well as coumarin is used for adulteration. This oil is often used for the adulteration of cinnamon bark oil. That is easy to detect, as coumarin is not a component of that oil. If *o*-methoxy cinnamaldehyde is found in cinnamon bark oil, it is a sign for adulteration with cassia oil. The naturality of the cinnamaldehyde can be detected by the combination of GC–combustion– IRMS (GC-C-IRMS) and GC-P-IRMS.

18.6.16 Cedar Leaf Oil

Adulteration is done by adding thujones from other species. According to Ravid (1992), the chiral value of (–)-fenchone is 100%. The thujones were found to be (–)-α-thujone 90% and (+)-α-thujone 10% (Gnitka 2010).

18.6.17 Cedarwood Oils

ISO standard 9843 shows character and data for Virginia cedarwood oil. This is obtained from *Juniperus virginiana* L., whereas the Texas oil is distilled from *Juniperus ashei* J. Buchholz. If these two oils are mixed together, the status of essential oil per definition is lost. Another adulteration is adding cedrol of cheaper cedarwood from Chinese oil (*Chamaecyparis funebris* (Endl.) Franco). Production is done by cooling down the oil and separating the crystals by filtration. As by-products, the terpenes will also serve as adulterations for other cedarwood oils. This is only valid for cedarwood oils coming from the species *Juniperus*. Recognition of adulterations is done by GC-MS.

18.6.18 Celery Seed Oil

ISO standard 3760 shows character and data for this oil. Blending is done with α-terpineol, limonene from orange oil, rectified copaiba oil, lovage root oil, and amyris oil. Detection is best done by GC-MS.

18.6.19 Chamomile Oil Blue

ISO standard 19332 shows character and data for that oil. Adulteration is done mainly by synthetic α-bisabolol, chamazulene, and (*E*)-βfarnesene. The method developed by Carle et al. (1990) and Carle (1996) using $\delta^{13}C$ and δD isotopes with IRMS is a real tool for naturality assay.

18.6.20 Chamomile Oil Roman

This essential oil, distilled from *Chamaemelum nobile* (L.) All., possesses a series of angelates in various concentrations. Bail (2009) published the following values for angelates and specific esters: isobutyl angelate 32.1%, 2-methylbutyl angelate 16.2%, isobutyl isobutyrate 5.3%, methyl 2-methylbutyrate 1.9%, prenyl acetate 1.4%, 2-methylbutyl 2-methylbutyrate 1.2%, and 2-methylbutyl acetate 1.2%. As most of these compounds are available as synthetic chemicals, adulteration can be done easily. Detection is done by the combination of GC-C-IRMS.

18.6.21 Cinnamon Bark Oil

Only in some pharmacopoeia monographs this oil is dealt with a chromatic profile. Adulteration is done once by adding cassia oil. This can easily be detected by any quantity of coumarin, never to be

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found in pure cinnamon bark oil. On the other hand, adulteration by synthetic cinnamaldehyde will be detected by the combination of GC-C-IRMS and GC-P-IRMS as the assessment of synthetic and natural cinnamaldehyde is possible. Sewenig et al. (2003) reported that the chiral ratio of linalool is (*R*)-(–) 95% and (*S*)-(+) 5%.

18.6.22 Cinnamon Leaf Oil

ISO standard 3524 shows character and data for this oil. As eugenol is the main component, rectified clove leaf oil or isolated eugenol from this oil is used. Also β-caryophyllene, synthetic benzyl benzoate, and *trans*-cinnamaldehyde are added. Blending is done by α-terpineol. Casabianca (1998) reported the ratio of (*R*)-(–)-linalool and (*S*)-(+)-linalool to be 64.0% and 36.0%, respectively.

18.6.23 Citronella Oil

ISO standard 3849 shows character and data for the Sri Lanka–type oil and ISO standard 3848 for the Java-type oil. Synthetic citronellal, citronellol, and geraniol were used. In addition citronella oil terpenes were used to cover such adulterations. Lawrence (1996a) mentions the ratio of citronellal in Java type with *R*-(+) enantiomer is 90%. Casabianca (1996) found chiral ratio of (*R*)-(+)-citronellol and (*S*)-(–)-citronellol to be 75.0%–79.0% and 21.0%–25.0%, respectively.

18.6.24 Clary Sage Oil

The chiral ratio of (*R*)-(–)-linalool and (*S*)-(+)-linalool is 80.6%–94.0% and 6.0%–19.4%, respectively, and of (*R*)-(–)-linalyl acetate and (*S*)-(+)-linalyl acetate is 93.0%–98.1% and 1.9%–7.0%, respectively, dependent on fresh or ensilaged biomass, found by Casabianca (1996).

18.6.25 Clove Oils

ISO standards 3141, 3142, and 3143 dealing with the oil from buds, leaves, and stems show character and data for these oils. The best and most expensive oil is from buds. Adulterations with oils from leaves and stem are used for adulteration. For those other oils, residues from isolation of natural eugenol are applied. Sometimes β-caryophyllene from other sources was used too. Detection can be made by GC-MS analysis.

18.6.26 Coriander Fruit Oil

ISO standard 3516 shows character and data for this oil. It was subject to adulteration with linalool, as it is the main component. By using chiral separation, Braun and Franz (2001) found the chiral ratio for linalool as follows: (*S*)-(+) from 64.8% to 87.3% and (*R*)-(–) from 12.7% to 35.2%, while Casabianca (1996) found (*S*)-(+) 87% to (*R*)-(–) 13%. Chiral ratio in limonene according to Casabianca (1996) is (*R*)-(+) 62.0%–93.0% and (*S*)-(–) 7.0%–38.0%.

18.6.27 Corymbia Citriodora Oil

ISO standard 3044 shows character and data for this oil. Adulteration is done by synthetic citronellal as well as by citronellol. Detection is done by GC-MS but better by multidimensional chiral separation. The chiral ratio of linalool is (*R*)-(–) 100.0% and (*S*)-(+) 0.0%, citronellal (*R*)-(+) 55.6%–57.2% and (*S*)-(–) 42.8%–44.4%, and citronellol (*R*)-(+) 49.0%–54.0% and (*S*)-(–) 46.0%–51.0%, found by Casabianca (1996).

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18.6.28 Corn Mint Oil

ISO standard 9776 shows character and data for this oil. Adulterations were carried out using synthetic menthol, menthyl acetate and menthone, terpenes from fractioning of corn mint oil, and limonene. Detection is done by enantiomeric separation by GC-MS. Mosandl (2000) reported the following chiral ratios: (1*S*)-(α)-pinene (56.5%–73.5%):(1*R*)-(α)-pinene (26.5%–43.5%); (+)-(1*S*)-(−)-β-pinene (49.1%–55.6%):(1*R*)-(+)-β-pinene (44.6%–50.9%); (1*S*)-(−)-limonene (98.1%–99.9%):(1*R*)-(+)-Limonene (0.1%–1.9%); (1*R*,3*R*,4*S*)-(−)-menthol (min 99.9%):(1*S*,3*S*,4*R*)-(+)-menthol (max 0.1%); and (4*R*)-(−)-piperitone 21.0%:(4*S*)-(+)-piperitone <0.1%.

18.6.29 Cumin Fruit Oil

ISO standard 9776 shows character and data for this oil. The main components are cumin aldehyde, p-mentha-1.3-dien-7al, p-mentha-1,4-dien-7al, γ-terpinene, and β-pinene. Blending is done by orange terpenes, p-cymene, and piperitone. Detection is done by GC-MS and by the combination of GC-C-IRMS and GC-P-IRMS as assessment of synthetic and natural cuminaldehyde is possible.

18.6.30 Cypress Oil

Adulteration is done by either turpentine oil or α- and β-pinene. Further, blending is done using δ-3-carene and cedrol from cedarwood Chinese type. AFNOR (1992) presents data in the standard NF T 75-254. Using chiral GC as his method of analysis, Casabianca (1996) determined that the enantiomeric ratio of α-thujene in cypress oil was as follows: (1*R*)-(+)-α-thujene (45%):(1*S*)-(−)-α-thujene (55%).

18.6.31 Dill Oils

Dill weed oil is dominated by α-phellandrene, limonene, and carvone. Dill ether and the absence of dill apiol are further criteria for that oil. Dill seed oil contains mainly carvone and dihydrocarvone. Adulteration is done using phellandrenes, distilled limonene coming from orange terpenes, synthetic carvone, and dihydrocarvone. Detection is done by 2D enantiomeric separation. Lawrence (1996) reports the following ratios for dill seed oil: (+)-limonene 98.4%:(−)-limonene 1.6%; (+)-carvone 98.7%:(−)-carvone 1.3%; (+)-*trans*-carveol 33.3%:(−)-*trans*-carveol 66.7%; and (+)-*cis*-carveol 100%:(−)-*cis*-carveol 0%. The authors own findings from biocultivated oil was (+)-carvone 98.4%:(−)-carvone 1.6%; (*S*)-(−)-α-pinene 4.0%:(*R*)-(+)-α-pinene 96.0%; (+)-limonene 95.4%:(−)-limonene 4.6%; (*S*)-(−)-β-phellandrene 0%:(*S*)-(+)-β-phellandrene 100%; and (*R*)-(−)-α-phellandrene 100%:(*R*)-(+)-α-phellandrene 0%.

18.6.32 Dwarf Pine Oil

ISO standard 21093 shows character and data for this oil. In the past, addition of turpentine oil was used for adulteration; later the essential oil of the needles of *Pinus maritima* was used, as the composition showed very close data of compounds. Chiral analysis gives helpful results. Kreis et al. (1991) reported chiral ratio in monoterpenes as follows: (1*R*,5*R*)-(+)-α-pinene (44%):(1*S*,5*S*)-(−)-α-pinene (56%); (1*R*,5*R*)-(+)-β-pinene (2%):(1*S*,5*S*)-(−)-β-pinene (98%); and (4*R*)-(+)-limonene (40%):(4*S*)-(−)-limonene (60%).

18.6.33 Elemi Oil

ISO standard 10624 shows character and data for this oil. Elemol and elemicin are the lead compounds. As minor component 10-epi-γ-eudesmol must be detected between 0.2% and 0.3% to ensure naturness. Adulteration is done by limonene, α-phellandrene, and sabinene. Detection is done by GC-MS.

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18.6.34 Eucalyptus Oil

ISO standard 770 shows character and data for this oil. Schmidt (2010) reported the Chinese eucalyptus oil coming from Sichuan province and is derived from *C. longepaniculatum* (Gamble). This must be labeled and it is not correct to mix this up with true eucalyptus varieties. Adulteration is done by 1,8-cineole from various *Cinnamomum* varieties. The detection is not easy as all these adulterations are natural.

18.6.35 Fennel Oil Sweet

Although this oil should be produced solely from fruits, more often the whole aerial parts are used. Adulteration is done by synthetic anethole or from other sources like star anise oil. Blending is done too with star anise oil and limonene + 60°. Analysis is done by GC-MS or by multidimensional enantiomeric separation. According to Ravid (1992), the chiral ratio of (+)-fenchone is 100%–0%.

18.6.36 Fennel Oil Bitter

ISO standard 17412 shows character and data for this oil. It is often mixed up with the herb oil derived from bitter fennel and sweet fennel produced from the whole aerial parts of the plant. Blending is done by sweet fennel oil, star anise oil, and fennel terpenes. Analysis is done by GC-MS or by multidimensional enantiomeric separation. Ravid reported that the chiral ratio of (+)-fenchone is 100%:0%. Casabianca (1996b) confirms a chiral ratio of (4*R*)-(+)-α-phellandrene 100%:(4*S*)-(–)-α-phellandrene 0%.

18.6.37 Geranium Oils

ISO standard 4731 shows character and data for that oil. This important and high-price product is and was often the target for adulteration. Blending was done with synthetic geraniol, citronellol, limonene—60°, terpinyl formate, synthetic rhodinol, α-terpineol cristallin, and distilled bergamot oil. Lawrence (1996) showed results of chiral separation of citronellol, *cis*- and *trans*-rose oxide, menthone, isomenthone, and linalool for geranium oils from different origins. The chiral ratio of (*R*)-(–) linalool and (*S*)-(+)-linalool is 42.0%–55.0% and 45.0%–58.0% found by Casabianca (1996b). In 1996 he published the following chiral data: (from various origins) (*R*)-(–)-citronellol (18.0%–43.0%) and (*S*)-(+)-citronellol (50.0%–82.0%); (2*R*,4*S*)-(+)-*cis*-rose oxide (24.0%–38.0%):(2*S*,4*R*)-(–)-*cis*-rose oxide (62.0%–76.0%); (2*R*,4*R*)-(–) *trans*-rose oxide (70.0%–76.0%):(2*S*,4*S*)-(+)-*trans*-rose oxide (72.0%–76.0%); (1*S*,4*R*)-(+)-menthone (>99.0%):(1*R*,4*S*)-(–)-menthone (<1%); and (1*S*,4*S*)-(–)-isomenthone (>99.0%):(1*R*,4*R*)-(+)-isomenthone (<1.0%); Lawrence (1999) confirmed further the values: (2*R*,4*S*)-(+)-*cis*-rose oxide (35.5%–49.3%):(2*S*,4*R*)-(–)-*cis*-rose oxide (50.7%–64.5%); (2*R*,4*R*)-(–) *trans*-rose oxide (49.2%–62.4%):(2*S*,4*S*)-(+)-*trans*-rose oxide (37.6%–50.8%); and (2*S*,4*R*)-(–)-*cis*-rose oxide ketone (50.5%–62.6%):(2*R*,4*S*)-(+)-*cis*-rose oxide ketone (37.4%–49.5%).

18.6.38 Grapefruit Oil

ISO standard 3053 shows character and data for this oil. Pure oils possess as marker the compound nootkatone from traces up to 0.8%, depending on the fruit status. This compound is used for blending, together with *n*-octanal, *n*-nonanal, *n*-decanal, and synthetic citral. Adulteration is performed by orange terpenes and distilled grapefruit residues from expression and limonene—80°. Detection must be done exclusively by multidimensional enantiomeric separation. Dugo and Mondello (2011) published the following chiral data: (*R*)-(–)-α-pinene (0.3%–0.8%):(*S*)-(+)-α-pinene (99.2%–99.7%); (*R*)-(+)-β-pinene (62.0%–76.8%):(*S*)-(–)-β-pinene (23.2%–38.0%); (*R*)-(+)-sabinene (98.4%–98.5%):(*S*)-(–)-sabinene (1.5%–1.6%); (*S*)-(–)-limonene (0.5%–0.6%):(*R*)-(+)-limonene

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(98.4%–98.5%); (*R*)-(−)-linalool (32.0%–43.0%):(*S*)-(+)-linalool (57.0%–68.0%); (*S*)-(−)-citronellal (16.6%–21.4%):(*R*)-(+)-citronellal (57.0%–68.0%); (*S*)-(−)-α-terpineol (1.2%–3.3%):(*R*)-(+)-α-terpineol (96.7%–99.8%); and (*S*)-(+)-carvone:(*R*)-(−)-carvone 34.8%.

18.6.39 Juniper Berry Oil

ISO standard 8897 shows character and data for this oil. *J. communis* oil is often mixed up with *J. oxycedrus*. As marker for that the myrcene content is rising up. Real markers are germacrene D- and δ-cadinene. The sesquiterpene fraction gives more information. Further on, addition of fractions of juniper berry oil from rectification as well as adding juniper branches oil is made. Kartnig et al. (1999) published some chiral data comparing self-distilled and commercial qualities of juniper berry α- and β-pinene, limonene, and terpinen-4-ol oils. Chirality was recognized as useful components for quality control of that oil. Mosandl et al. (1991) report a ratio for (*S*)-(−)-α-pinene 77%:(*R*-)-(+)-α-pinene 23%.

18.6.40 Lavandin Oils

ISO standard 3054 and 8902 shows character and data for Abrial and Grosso lavandin oil. This oil is adulterated by adding acetylated lavandin, lavandin distilled heads and tails, camphor oil white, Spanish sage oil, and spike lavender oil. Blending is done by terpinyl acetate, turpentine oil, methyl α-terpineol and ethyl amyl ketone, hexyl ketone, and geranyl acetate, all from synthetic source. Chiral ratio of linalool is from (*R*)-(−) 64.8% to 87.3% and (*S*)-(+) from 12.7% to 35.2%. Chiral ratio of linalyl acetate according to the findings of Casabianca (1996a) is (*R*)-(−) from 98.1% to 100.0% and (*S*)-(+) from 0.0% to 1.0%). Renaud (2001) reported chiral ratios for linalool and linalyl acetate as follows: (3*R*)-(−)-linalool (94.5%–98.2%):(3*S*)-(+)-linalool (1.8%–5.5%) and (3*R*)-(−)-linalyl acetate (99%–100%):(3*S*)-(+)-linalyl acetate (0%–1%). Lawrence (1996a) published the following chiral ratios: (3*R*)-(−)-linalyl acetate (98.3%–100%):(3*S*)-(+)-linalyl acetate (0.0%–1.7%); (3*R*)-(−)-linalool (95.0%–96.6%):(3*S*)-(+)-linalool (3.4%–5.0%). (2*S*,5*S*)-*trans*-linalool oxide (4.2%–23.3%):(2*R*,5*R*)-*trans*-linalool oxide (76.7%–95.8%); (2*R*,5*S*)-*cis*-linalool oxide (82.9%–95.8%):(2*S*,5*R*)-*trans*-linalool oxide (4.2%–17.1%); (*R*)-(−)-lavandulol (96.2%–99.0%):(*S*)-(+)-lavandulol (1.0%–3.8%); and (*R*)-(−)-terpinen-4-ol (1.6%–10.9%):(*S*)-(+)-terpinen-4-ol (89.1%–98.4%).

18.6.41 Lavender Oil

ISO standard 3515 shows character and data for oils from various origins. Most applied adulterations were already discussed in the text. Chiral separations showed the most effective results in analysis. The ratio of linalool is (*R*)-(−) 98.0%–100.0% and (*S*)-(+) 0.0%–2.0% found by Casabianca (1996a). He too found that the chiral ratio of linalyl acetate is (*R*)-(−) from 98.0% to 100.0% and (*S*)-(+) from 0.0% to 2.0%. Stoyanova reports the following data for Bulgarian lavender oil: (3*R*)-(−)-linalyl acetate (100%):(3*S*)-(+)-linalyl acetate (0%); (3*R*)-(−)-linalool (95.0%–96.6%):(3*S*)-(+)-linalool (3.4%–5.0%); and (3*R*)-(+)-camphor (27.4%–52.2%):(3*S*)-(−)-camphor (47.8%–78.6%). Kreis and Mosandl (1992) published the following data: (2*S*,5*S*)-*trans*-linalool oxide (3.9%–23.3%):(2*R*,5*R*)-*trans*-linalool oxide (76.7%–96.1%); (2*R*,5*S*)-*cis*-linalool oxide (82.9%–95.8%):(2*S*,5*R*)-*trans*-linalool oxide (4.2%–17.1%); (*R*)-(−)-lavandulol (89.8%–100%):(*S*)-(+)-lavandulol (0%–10.2%); (*R*)-(−)-terpinen-4-ol (0%–10.9%):(*S*)-(+)-terpinen-4-ol (89.1%–100%); and (3*R*)-(−)-linalool (95.1%–98.2%):(3*S*)-(+)-linalool (18%–4.9%).

18.6.42 Lemon Oil

ISO standard 855 shows character and data for that oil. Adulteration is done by distilled lemon oil from residues, orange terpenes or limonene from orange terpenes, and synthetic citral from

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Litsea cubeba oil. Lemon oil washed as residues from production of terpene-free oil is preferably used, as these contain still all components of the pure lemon oil. Also lemon terpenes and heads of distilled grapefruit oils could be found. Blending is done by using synthetic decanal, nonanal, octanal, and citronellal from *Corymbia citriodora* oil. Detection is made by GC-MS and mainly by multidimensional enantiomeric separation with various methods (see part of methods). Mondello (1998) reports some constituents with chiral ratios as follows: (*R*)-(+)-β-pinene 6.3%:(*S*)-(−)-β-pinene 93.7%; (*R*)-(+)-sabinene 14.9%:(*S*)-(−)-sabinene 85.1%; (*S*)-(−)-limonene 1.6%:(*R*)-(+)-limonene 98.4%; (*S*)-(+)-terpinen-4-ol 24.7%:(*R*)-(−)-terpinen-4-ol 75.3%; and (*S*)-(−)-α-terpineol 75.2%:(*R*)-(+)-α-terpineol 75.2%. Further on, Dugo and Mondello (2011) gave the following data: (*R*)-(+)-α-pinene (25.5%–31.5%):(*S*)-(−)-α-pinene (68.5%–74.5%); (1*S*,4*R*)-(−)-camphene (86.2%–92.4%):(1*R*,4*S*)-(+)-camphene (7.6%–13.8%); (*S*)-(−)-β-pinene (93.2%–95.7%):(*R*)-(+)-β-pinene (4.3%–6.8%); (*R*)-(+)-sabinene (12.4%–15.0%):(*S*)-(−)-sabinene (85.0%–87.6%); (*R*)-(−)-α-phellandrene (46.9%–52.6%):(*S*)-(+)-α-phellandrene (47.4%–53.1%); (*R*)-(−)-β-phellandrene (31.1%–53.9%):(*S*)-(+)-β-phellandrene (46.1%–68.9%); (*S*)-(−)-limonene (1.4%–1.6%):(*R*)-(+)-limonene 98.4%–98.6%); (*R*)-(−)-linalool (52.0%–74.5%):(*S*)-(+)-linalool (25.5%–48.0%); (*S*)-(−)-citronellal (89.5%–94.8%):(*R*)-(+)-citronellal (5.2%–10.5%); (*S*)-(+)-terpinen-4-ol (12.0%–26.2%):(*R*)-(−)-terpinen-4-ol (73.8%–88.0%); and (*S*)-(−)-α-terpineol (66.4%–82.0%):(*R*)-(+)-α-terpineol (18.0%–33.6%).

18.6.43 Lemongrass Oil

ISO standard 4718 shows character and data for this oil. Adulteration is done by adding synthetic citral or citral from *Litsea cubeba* oil. Blending is done with addition of geranyl acetate and 6-methyl-5-heptene-2-one. Detecting is done by GC-MS and multidimensional chiral separation. Wang et al. (1995) reported chiral ratio for linalool to be (3*S*)-(+)-linalool 30.9%:(*3*R)-(−)-linalool 69.1% and (*R*)-(−)-linalool 58.0%:(*S*)-(+)-linalool 42.0%.

18.6.44 Lime Oil Distilled

ISO standard 3809 shows character and data for that oil. Adulteration is done by adding limonene from different sources, synthetic terpineol and γ-terpinene from lime terpenes as well as from heads of the production of terpene-free lemon oil. Detection must be done by multidimensional chiral separation. Dugo and Mondello (2011) report the following data for chiral ratios: (*S*)-(−)-β-pinene (96.0%–96.8%) (*R*)-(+)-β-pinene (3.2%–4.0%); (*S*)-(−)-limonene (5.5%–8.7%):(*R*)-(+)-limonene (91.3%–94.5%); (*R*)-(−)-linalool (49.8%–80.0%):(*S*)-(+)-linalool (50.0%–50.2%); (*S*)-(+)-terpinen-4-ol (42.3%–45.0%):(*R*)-(−)-terpinen-4-ol (55.0%–57.7%); and (*S*)-(−)-α-terpineol (53.3%–56.8%):(*R*)-(+)-α-terpineol (46.7%–43.2%).

18.6.45 Lime Oil Expressed

ISO standard 23954 shows character and data for this oil. Blending is done by adding limonene from different sources, citral from *Litsea cubeba* oil and γ-terpinene from lime terpenes as well as from heads of the production of terpene-free lemon oil. Detection is done by GC-MS, looking for absence of δ-3-carene (not even in traces) and multidimensional enantiomeric separation with various methods (see part of methods). Dugo and Mondello (2011) reported the following chiral ratios: (*S*-)-(−)-(β)-pinene (98.7%–90.9%):(*R*)-(+)-β-pinene (9.1%–10.3%); (*R*)-(+)-sabinene (18.2%–23.4%):(*S*)-(−)-sabinene (76.6%–81.8%); (*S*)-(−)-limonene (0.4%–2.7%):(*R*)-(+)-limonene (97.3%–99.6%); (*R*)-(−)-linalool (54.4%–69.3%):(*S*)-(+)-linalool (30.7%–45.6%); (*S*)-(+)-terpinen-4-ol (18.6%–24.9%):(*R*)-(−)-terpinen-4-ol (75.1%–81.4%); and (*S*)-(−)-α-terpineol (74.5%–80.8%):(*R*)-(+)-α-terpineol (19.2%–25.5%).

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18.6.46 *Litsea cubeba* Oil

ISO standard 3214 shows character and data for this oil. Adulteration is done by adding synthetic citral and can be detected by GC-MS by checking the values for geranial and neral but also for isogeranial and isoneral.

18.6.47 Mandarin Oil

ISO standard 3528 shows character and data for this oil. α-Sinensal is a marker for mandarin essential oil. Adulteration is made by synthetic methyl-*n*-methyl anthranilate, as well as methyl anthranilate. Orange terpenes and limonene (80°), dipentene, citronellal, and citral are used for blending. Detection is done by GC-MS but improved results show multidimensional chiral separation. Dugo and Mondello (2011) reported the following chiral data: (*S*)-(+)-α-thujene (0.3%–1.9%):(*R*)-(−)-α-thujene (98.1%–99.7%); (*R*)-(+)-α-pinene (41.7%–54.5%):(*S*)-(−)-α-pinene (45.5–58.3); (1*S*,4*R*)-(−)-camphene (31.8%–72.6%):(1*R*,4*S*)-(+)-camphene (27.4%–68.2%); (*R*)-(+)-β-pinene (87.8%–99.1%):(*S*)-(−)-β-pinene(0.9%–12.2%);(*R*)-(+)-sabinene(71.3%–83.5%):(*S*)-(−)(16.5%–28.7%); (*R*)-(−)-α-phellandrene (44.3%–55.0%):(*S*)-(+)-α-phellandrene (45.0%–55.7%); (*R*)-(−)-β-phellandrene (0.4%–3.0%):(*S*)-(+)-β-phellandrene (97.0%–99.6%); (*S*)-(−)-limonene (1.5%–2.91%):(*R*)-(+)-limonene (97.1%–98.5%); (1*R*,4*R*)-(+)-camphor (17.0%–36.5%):(1*S*4*R*)-(−)-camphor (63.5%–83.0%); (*R*)-(−)-linalool (13.0%–22.7%):(*S*)-(+)-linalool (77.3%–87.0%); (*S*)-(−)-citronellal 3.9%–9.2%):(*R*)-(+)-citronellal 90.8%–96.1%); (*S*)-(+)-terpinen-4-ol (9.5%–23.8%):(*R*)-(−)-terpinen-4-ol (76.2%–90.5%); and (*S*)-(−)-α-terpineol (66.1%–75.9%):(*R*)-(+)-α-terpineol (24.1%–33.9%).

18.6.48 Melissa Oil (Lemon Balm)

This expensive essential oil was and is often the target of adulteration: citronella oil (*Cymbopogon winterianus* or *Cymbopogon nardus*), lemongrass oil, lemon oil citral, and geraniol rose oxides, natural and synthetic. Lawrence (1996) reports the chiral values of (*R*)-(+)-citronellal in that oil being 97.2%–98.2%. Also the value for (*R*)-(+)-methyl citronellate is at a minimum of 99.0% By using GC analysis with different chiral GLC phases, Schultze (1993) confirmed citronellal from other sources as follows: lemongrass oil (*S*)-(−) 30%–55%, (*R*)-(+) 70%–45%; citronella oil (*S*)-(−) 10%–15%, (*R*)-(+) 90%–85%; and catnip oil (*S*)-(−) 98%–99.9%, (*R*)-(+) 2%–0.1%. Schultze also published δ-values to confirm naturality.

18.6.49 *Mentha citrata* Oil

The chiral ratio of linalool is (*R*)-(−) 42.0%–55.0% and (*S*)-(+) 45.0%–58.0% found by Casabianca (1996a). Chiral ratio of linalyl acetate according to the findings of Casabianca (1996) is (*R*)-(−) from 98.6% to 99.0% and (*S*)-(+) from 1.0% to 1.4%.

18.6.50 Mountain Pine Oil

ISO standard 21093 shows character and data for that oil. *Pinus maritima* shows nearly identical values and adulteration is hardly to recognize. As long as the price of the oil of *P. maritima* was one-fifth of the *Pinus mugo*, it was sold in large quantities as mountain pine oil. Now prices are nearly identical and mixing up makes no sense anymore. Adulteration is done by α-pinene, β-pinene, δ-3-carene, (−)-limonene, myrcene, β-phellandrene, and *l*-bornyl acetate from various sources.

18.6.51 Neroli Oil

ISO standard 3517 shows character and data for that oil. Adulteration is made by geraniol from palmarosa oil, linalool from rose wood oil, orange oil sweet, citral from *Litsea cubeba* oil, and

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petitgrain oil Paraguay. Blending is done by methyl-*n*-methyl anthranilate, methyl anthranilate, synthetic phenyl ethyl alcohol, and synthetic indol. Detection is done by GC-MS and multidimensional chiral separation. Casabianca (1996a) found the chiral ratio for linalool with (*S*)-(+) 12.0–13.8 and (*R*)-(–) 86.2%–88.0% and for linalyl acetate with (*S*)-(+) 1.8–5.0 and (*R*)-(–) 95.0%–98.2%. Dugo and Mondello (2011) reported the following chiral ratios: (*R*)-(+)-α-pinene (2.2%–13.6%):(*S*)-(–)-α-pinene (86.4%–97.8%); (*R*)-(+)-β-pinene (0.1%–0.8%):(*S*)-(–)-β-pinene (99.2%–99.9%); (*S*)-(–)-limonene (1.9%–6.9%):(*R*)-(+)-limonene (93.1%–98.1%); (*R*)-(–)-linalool (70.8%–81.5%):(*S*)-(+)-linalool (18.4%–29.2%); (*R*)-(–)-linalyl acetate (95.4%–98.2%):(*S*)-(+)-linalyl acetate (1.8%–4.6%); (*S*)-(+)-terpinen-4-ol (36.0%–37.6%):(*R*)-(–)-terpinen-4-ol (62.4%–64.0%); (*S*)-(–)-α-terpineol (28.1%–39.8%):(*R*)-(+)-α-terpineol (60.2%–71.9%); and (*R*)-(–)-(*E*)-nerolidol (0.4%–1.8%):(*S*)-(+)-(*E*)-nerolidol (98.2–99.6).

18.6.52 Nutmeg Oil

ISO standard 3215 shows character and data for this oil. Adulteration is done by monoterpenes α- and β-pinene, sabinene from different sources, and α- and β-phellandrene as well as synthetic linalool, terpinen-4-ol and α-terpineol. In the past, safrole from sassafras oil was used for blending. Detection is done by GC-MS, looking for the quantities of safrole and myristicin; in addition 2D chiral separation is recommended. König et al. (1992) reported the following chiral ratios: (+)-α-thujene 10.3%:(–) α-thujene 89.7%; (–)-α-pinene 79.3:(+)-α-pinene 20.7%; (–)-camphene 100%:(+)-camphene 0%; (+)-β-pinene 41.9%:(–)-β-pinene 58.1%; (–)-α-phellandrene 0%:(+)-α-phellandrene 100%; (+)-sabinene 89.5%:(–)-sabinene 10.5%; (+)-δ-3-carene 0%:(–)-δ(–)-3-carene 100%; (–)-β-phellandrene 7.7%:(+)-β-phellandrene 92.3%; and (–)-limonene 60.9%:(+)-limonene 39.1%.

18.6.53 Orange Oil Sweet

ISO 3140 shows character and data for this oil. Adulteration is done by adding orange terpenes or purified limonene. Casabianca (1996) found chiral ratio for linalool with (*S*)-(+) 86 to (*R*)-(–) 14. Hara et al. (1999) reported chiral ratios of several hydrocarbon components as follows: (1*R*,5*R*)-(+)-α-pinene (99.6%–99.7%):(1*S*,5*S*)-(–)-α-pinene (0.3%–0.4%); (1*R*,5*R*)-(+)-β-pinene (66.1%–77.8%):(1*S*,5*S*)-(–)-β-pinene (22.2%–33.9%); (4*R*)-(+)-limonene (99.1%–99.4%):(4*S*)-(–)-limonene (0.6%–0.9%); (3*S*)-(+)-linalool (82.5%–96.3%):(3*R*)-(–)-linalool (3.7%–17.5%); (4*R*)-(+)-α-terpineol (97.0%–98.4%):(4*S*)-(–)-α-terpineol (1.9%–3.0%); and (3*R*)-(+)-citronellal (31.3%–87.8%):(3*S*)-(–)-citronellal (12.2%–68.7%).

18.6.54 Origanum Oil

This oil is from *Origanum vulgare* L. ssp. *hirtum*. Adulteration is done with synthetic thymol and carvacrol or with limonene from different sources. The chiral ratio of linalool is (*R*)-(–) 82.0% and (*S*)-(+) 18.0% found by Casabianca (1996).

18.6.55 Palmarosa Oil

ISO 4727 shows character and data for this oil. As the main component is geraniol, adulteration is done by synthetic geraniol. Detection must be done by the combination of GC-C-IRMS and GC-P-IRMS as the assessment of synthetic and natural geraniol is possible.

18.6.56 Parsley Oil

ISO standard 3527 shows character and data for this oil. Adulteration is done by turpentine oil or pure α-pinene, β-pinene, and elemicin from elemi resinoid. 1,2,3,4-Tetramethoxy-5-allyl benzene is

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a key compound up to 12% as well as apiol. Blending is done by celery grain oil, nutmeg oil, and carrot seed oil. Detection is done by GC-MS system.

18.6.57 Pine Oil Siberian

ISO 10869 shows character and data for this oil. Adulteration is done by turpentine oil, *l*-bornyl acetate synthetic, terpinyl acetate, and Virginia cedarwood oil. Detection is done either by GC-MS or chiral separation. Ochocka (2002) determined the enantiomeric ratios of four monoterpene hydrocarbons in *A. sibirica* oils (Korean source): (1*R*,5*R*)-(+)-α-pinene 25.4%:(1*S*,5*S*)-(−)-α-pinene 74.6%; (1*R*,5*R*)-(+)-β-pinene 13.2%:(1*S*,5*S*)-(−)-β-pinene 86.8%; (4*R*)-(+)-limonene 6.8%:(4*S*)-(−)-limonene 93.2%; and (3*R*)-(+)-camphene 4.8%:(3*S*)-(−)-camphene 95.2%.

18.6.58 Patchouli Oil

ISO standard 3757 shows character and data for this oil. This very complex oil is adulterated by gurjun balm oil (see text). Blending is done by patchouli terpenes, cedarwood oil, pepper oil, white camphor oil, and guaiac wood oil. Detection is done by GC-MS.

18.6.59 Pepper Oil

ISO standard 3061 shows character and data for this oil. Blending is done with turpentine oil, α-phellandrene from other sources, limonene from orange terpenes, and clove leaf oil terpenes. The chiral ratio of linalool is (*R*)-(−) 81.0%–89.0% and (*S*)-(+) 11.0%–19.0% found by Casabianca (1996a). König et al. (1992) reported the following chiral ratios: (+)-α-thujene 100%:(−)-α-thujene 0%; (−)-α-pinene 74.6%:(+)-α-pinene 25.4%; (−)-camphene 66.6%:(+)-camphene 33.4%; (+)-β-pinene 2.6%:(−)-β-pinene 97.4%; (+)-δ3-carene 2.6%:(−)-δ3-carene 97.4%; (−)-α-phellandrene 0%:(+)-α-phellandrene 100%; (−)-β-phellandrene 100%; and (−)-limonene 61.7%:(+)-limonene 38.3%.

18.6.60 Peppermint Oil

ISO standard 856 shows character and data for that oil. Adulteration is done by synthetic menthol, menthol from cornmint oil, and fractions of peppermint terpenes. Detection is done by chiral separation using 2D enantiomeric columns on GC-MS system. The chiral ratio of 3-octanol is (*R*)-(−) 94.1%–100.0% and (*S*)-(+) 0.0%–5.9% according to values found by Casabianca (1996b). Mosandl (2000) reports the following ratios: (1*S*)-(−)-α-pinene (45.1%–68.1%):(1*R*)-(+)-α-pinene (31.9%–54.9%); (1*S*)-(−)-β-pinene (41.7%–53.6%):(1*R*)-(+)-β-pinene (46.4%–58.3%); (4*S*)-(−)-limonene (74.4%–98.3%):(4*R*)-(+)-limonene (1.7%–25.6%); (1*R*,3*R*,4*S*)-(−)-menthol (min 99.9%):(1*S*,3*S*,4*R*)-(+)-menthol (max 0.1%); and (4*R*)-(−)-piperitone 2.0%–13.0%:(4*S*)-(+)-piperitone 87.0%–98.0%.

18.6.61 Petitgrain Oil Paraguay Type

ISO standard 25157 shows character and data for this oil. A marker for natural quality is (*Z*,Z)-farnesol with 2.0%–3.5%. Adulteration is done by limonene (80°) and rectified orange terpenes. The chiral ratio of linalool is (*R*)-(−) 71.4%–73.3% and (*S*)-(+) 26.7%–28.6% found by Casabianca (1996a).

18.6.62 Pimento Oils

ISO standard 4729 shows character and data for the oil from *Pimento dioica* (L.) Merr. (Pimento oil Jamaica type). ISO standard 3045 shows character and data for the oil of *Pimenta racemosa*

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(Mill.) J.W. Moore (Bay oil). The difference between the oils is the content of eugenol and myrcenol. Pimento berry oil shows a content of more than 80% of eugenol and traces of myrcene, while bay oil contains maximum 56% of eugenol but up to 30% of myrcene. Eugenol from cinnamon leaf oil is added as adulteration for both oils and myrcene from other sources. Detection is done by GC-MS.

18.6.63 Rose Oil

ISO standard 9842 shows character and data for oils from various sources. Blending is done with synthetic phenyl ethyl alcohol, synthetic rhodinol, and geraniol from palmarosa oil. Geranium oil, ylang oil, rose absolute, and palmarosa oil are used for "finishing." Detection has to be done by GC-MS system and by chiral separation with multidimensional GC-MS. Kreis and Mosandl (1992) report the enantiomeric ratios for (*S*)-(−)-citronellol with >99%; (2*S*,4*R*)-*cis*-rose oxide as well as (2*R*,4*R*)-*cis*-rose oxide show a purity higher than 99.5%.

18.6.64 Rosemary Oil

ISO standard 1342 shows character and data for oils from various sources. Turpentine oil, synthetic camphor, and limonene from orange terpenes are used to blend this oil. Adulteration is done by 1,8-cineole from eucalyptus or white camphor oil. Detection is usually done by GC-MS but also by multidimensional chiral separation. Enantiomeric ratio of linalool is reported by Casabianca (1996a) to be (*R*)-(−) 23 to (*S*)-(+) 77. Kreis (1991a) published the following values of chiral separation: (−)-borneol (84.6%–97.8%):(+)-borneol (2.2%–15.4%) and (−)-isoborneol (29.1%–53.8%):(+)-isoborneol (46.2%–70.9%). König et al. (1992) reported the following chiral ratios: (+)-α-thujene 33.7%:(−)-α-thujene 66.3%; (−)-α-pinene 41.7%:(+)-α-pinene 58.3%; (−)-β-pinene 84.4%:(+)-β-pinene 15.6%; (−)-camphene 83.3%:(+)-camphene 16.7%; (−)-α-phellandrene 0%:(+)-α-phellandrene 100%; (−)-β-phellandrene 2.5%:(+)-β-phellandrene 97.5%; and (−)-limonene 64.8%:(+)-limonene 35.2%. The authors own results analyzing pure oils showed the following ratios: (+)-β-pinene 26.0%:(−)-β-pinene 74.0%; (−)-α-pinene 42.0%:(+)-α-pinene 58.0%; (*S*)-(−)-sabinene 66.5%:(*R*)-(+)-sabinene 33.5%; and (*S*)-(−)-camphene 31.4%:(*R*)-(+)-camphene 68.6%.

18.6.65 Rosewood Oil

ISO standard 3761 shows character and data for this oil. Blending is done by synthetic linalool, α-terpineol, geraniol, and heads of rosewood oil. Eremophilane is a marker according to Lawrence (1999) with values of 0.3%–0.9%. Detection is done by GC-MS on a chiral column. Casabianca (1996a) found chiral ratio for linalool with (*S*)-(+) 50.0%–51% and (*R*)-(−) 49.0%–50%. Lawrence (1999) reported the following chiral ratios: (3*R*)-(−)-linalool 10.0%:(3*S*)-(+)-linalool 90.0%. (For information, rosewood leaf oil ratio is (3*R*)-(−)-linalool 22.2%:(3*S*)-(+)-linalool 77.8%).

18.6.66 Sage Oil (*Salva officinalis*)

ISO standard 9909 shows character and data for this oil. As the main components α-thujone β-thujone are known, adulteration is done with thuja oil or cedar leaf oil. β-Caryophyllene, 1,8-cineole, and borneol from other sources are used. α-Humulene is a marker with up to 12% total content. Detection is done by GC-MS analysis.

18.6.67 Sage Oil Spanish Type

ISO standard 3526 shows character and data for this oil derived from *Salvia lavandulifolia* Vahl. Adulteration is done by eucalyptus oil, camphor oil chemotypes like 1,8-cineole and camphor,

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α-pinene, and limonene from different sources. Blending is done with synthetic linalyl acetate und terpinyl acetate. Detection can be achieved by using GC and GC-MS analytical equipment.

18.6.68 Sandalwood Oil

ISO standard 3518 shows character and data for the oil derived from *Santalum album* L. This very expensive oil with source from India is no longer sold on the market these days. Today other varieties from various sources like New Caledonia (*Santalum austrocaledonicum* Vieill. and *Santalum spicatum* (R.Br.) A. DC) from Australia are used in flavor and fragrance industry. α- and β-Santalols are the main components and responsible for the fragrance. Adulteration will take place with cheaper varieties, coming from Australia. This is very simple to detect as *Santalum spicatum* contains *cis*-nuciferol in high amounts. The same can be seen with *S. austrocaledonicum*, where the *cis*-lanceol can be found in high amounts compared to the other oils. Braun et al. (2005) found in analyzing *S. album* oil the following chiral substances and values: (1*R*,4*R*,5*S*)-α-acorenol (0.22%), (1*R*,4*R*,5*R*)-β-acorenol (0.11%), (1*R*,4*S*,5*S*)-epi-α-acorenol (0.13%), and (1*R*,4*S*,5*R*)-epi-β-acorenol (<0.01%).

18.6.69 Spearmint Oils

ISO 3033-1-3033.4 shows character and data for these oils from various varieties and hybrids. Adulteration is done by synthetic levo-carvone. Detection is done by enantiomeric separation by GC-MS. Coleman (2002), Lawrence and Cole (2011) report the chiral ratio of monoterpenes in native spearmint to be (1*R*,5*R*)-(+)-α-pinene-(40.3%):(1*S*,5*S*)-(−)-α-pinene (59.7%); (1*R*)-(+)-camphene (99.9%):(1*S*)-(−)-camphene (0.1%); (1*R*,5*R*)-(+)-β-pinene (48.7%):(1*S*,5*S*)-(−)-β-pinene (51.3%); and (4*R*)-(+)-limonene (1.9%):(4*S*)-(−)-limonene (98.1%). Further on, Nakamoto (1996) gave results about *cis*- and *trans*-carveol as follows: (2*R*,4*S*)-(+)-*trans*-carveol (15%):(2*S*,4*R*)-(−)-trans-carveol (85%) and (2*S*,4*S*)-(+)-cis-carveol (4%):(2*R*,4*R*)-(−)-*cis*-carveol (96%). Mosandl (2000) reports the following chiral ratios: (1*S*-)-(−)-α-pinene (59.7%–62.4%):(1*R*)-(+)-α-pinene (37.6%–40.3%); (1*S*)-(−)-β-pinene (51.3%–52.1%):(1*R*)-(+)-α-pinene (98.1%–98.8%); (−)-camphene (<0.1%):(+)-camphene (>99.9%), and (4*S*)-(+)-carvone (99.1%–99.9%):(4*R*)-(−)-carvone (0.1%–0.9%).

18.6.70 Spike Lavender Oil

ISO standard 4719 shows character and data for that oil. Adulteration is done by white camphor oil, 1,8-cineole distilled from eucalyptus oil, synthetic camphor, and linalool. Blending is done with terpenes from eucalyptus oil, turpentine oil, *n*-bornyl acetate, lavandin, rosemary oil, HO leaf oil, and α-terpineol. Detection can be made by GC-MS and by multidimensional chiral separation. Ravid (1992) mentions the chiral ratio of terpinen-4-ol as (4S)-(+)-terpinen-4-ol 93%:(4R)-(−)-terpinen-4-ol 7%.

18.6.71 Star Anise Oil

ISO standard 11016 shows character and data for the oil. This oil is not the target for adulteration but is itself an oil for the adulteration of oils containing *trans*-anethole. For the detection of possible adulteration by limonene and α-pinene trace components, *cis*- and *trans*-bergamotene and foeniculin are markers with quantities according to the standard.

18.6.72 Tarragon Oil

ISO standard 10115 shows character and data for this oil. Adulteration and blending are done by synthetic anethole, eugenol from cinnamon leaf oil, synthetic estragole, and *cis*- and *trans* ocimene

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from other sources. Detection is done either by GC-MS or by 2D chiral separation. The chiral ratio of linalool is (*R*)-(–) 80.0%–90.0% and (*S*)-(+) 10.0%–20.0% found by Casabianca (1996a).

18.6.73 Tea Tree Oil

ISO standard 4730 shows character and data for this oil. Adulteration is done by adding α-pinene and β-pinene from different sources and γ-terpinene from terpenes resulting from citrus concentration production. Synthetic terpinen-4-ol is also used. Detection is done by GC-MS and by using multidimensional chiral analytical equipment. Cornwell et al. (1995) reported the following chiral ratios for steam distillation of whole branches: (+)-sabinene 58%:(–)-sabinene 42%; (+)-α-pinene 91%:(–)-α-pinene 9%; (+)-α-phellandrene 49%:(–)-α-phellandrene 51%; (+)-β-phellandrene 71%:(–)-β-phellandrene 29%; (+)-limonene 62%:(–)-limonene 38%; (+)-terpinen-4-ol 65%:terpinen-4-ol 35%; and (+)-α-terpineol 80%:(–)-α (–)-terpineol 20%.

18.6.74 Thyme Oil

ISO standard 14715 shows character and data for the oil of *T. zygis* (Loefl.) L. For real thyme oil (*T. vulgaris* L.), no ISO standard is available. Origanum oil and marjoram oil from Spain were used for adulteration in earlier times. Also synthetic thymol and carvacrol were applied components. Pure essential oil of thyme is red, as most of the steel vessels contain iron and this reacts with the thymol. White thyme oil, sold in high quantities, is mostly blended and synthetic. By using multidimensional chiral analytical equipment starting in 1990, Herner et al. 1990 reported the following enantiomeric ratios: (1*S*)-(–)-α-pinene 89%:(1*R*)-(+)-α-pinene 11%; (1*S*)-(–)-β-pinene 96%:(1*R*)-(+)-β-pinene 4%; and (4*S*)-(–)-limonene 70%:(4*R*)-(+)-limonene 30%. Kreis et al. (1991) reported the chiral ratio of borneol as follows: (–)-borneol (98.1%–99.6%):(+)-borneol (0.4%–1.9%). The chiral ratio of linalool is (*R*)-(–) 94.5%–99.0% and (*S*)-(+) 1.0%–5.5%. Casabianca (1996a) showed the following value for chiral ratios of linalyl acetate, although this is a minor component: (3*R*)-(–)-linalyl acetate (93.8%–99.2%):(3*S*)-(–)-linalyl acetate (0.8%–6.2%).

18.6.75 Turpentine Oil

ISO standard 21389 shows character and data for this oil. It is distilled from *Pinus massoniana* Lamb. and produced in China. Adulteration is rarely observed, but this oil is a source for the production of α-pinene and β-pinene. Both compounds are used for the adulteration of various essential oils containing these monoterpenes in higher value. Another type of turpentine oil is distilled from *Pinus pinaster* Aiton but is less of interest for adulterations as the quantities produced are minor.

18.6.76 Vetiver Oil

ISO standard 4716 shows character and data for this oil. Limonene chiral ratio is (*R*)-(–) with 100%. The identical value was reported by Möllenbeck (1997) for α-terpineol. He too found a ratio of linalool to be (*R*)-(–) 80% and (*R*)-(+) with 20% as well as terpinen-4-ol to be (*R*)-(–) 66% and (*S*)-(+) with 34%. Because of use GC×GC-TOF for detection it will be impossible to adulterate this essential oil.

18.6.77 Ylang-Ylang Oils

ISO standard 3063 shows character and data for this oil, distilled from the flowers of *C. odorata* (Lam.) Hook. F. Thomson forma genuina. Adulteration is done either by one of the fractions of cananga oil (extra, first, second, or third) or with various fractions like heads and tails. The mingling up of various fractions like extra, I, II, or III will not touch the purity or naturality of that oils as long

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as all are from the same botanical source. *C. odorata* (Lam.) Hook. F. Thomson forma *macrophylla* is used for adulteration. Blending is done by linalyl acetate, benzyl acetate, synthetic geraniol from various sources, methyl benzoate, benzyl alcohol, methyl salicylate, all synthetic and bay leaf oil, cedarwood terpenes, lavandin residues, and traces of ethyl vanillin (synthetic). Detection is done by GC/MS and by multidimensional chiral separation. Casabianca (1996a) reported the chiral ratio for linalool to be (*S*)-(+) 1.6–3.0 and (*R*)-(–) 97.0%–98.4%.

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