UNIMORE
UNIVERSITÀ DEGLI STUDI DI MODENA E REGGIO EMILIA

Dipartimento di Scienze della Vita

La produzione degli oli essenziali e i requisiti di qualità

Coltivazione e utilizzi delle piante officinali

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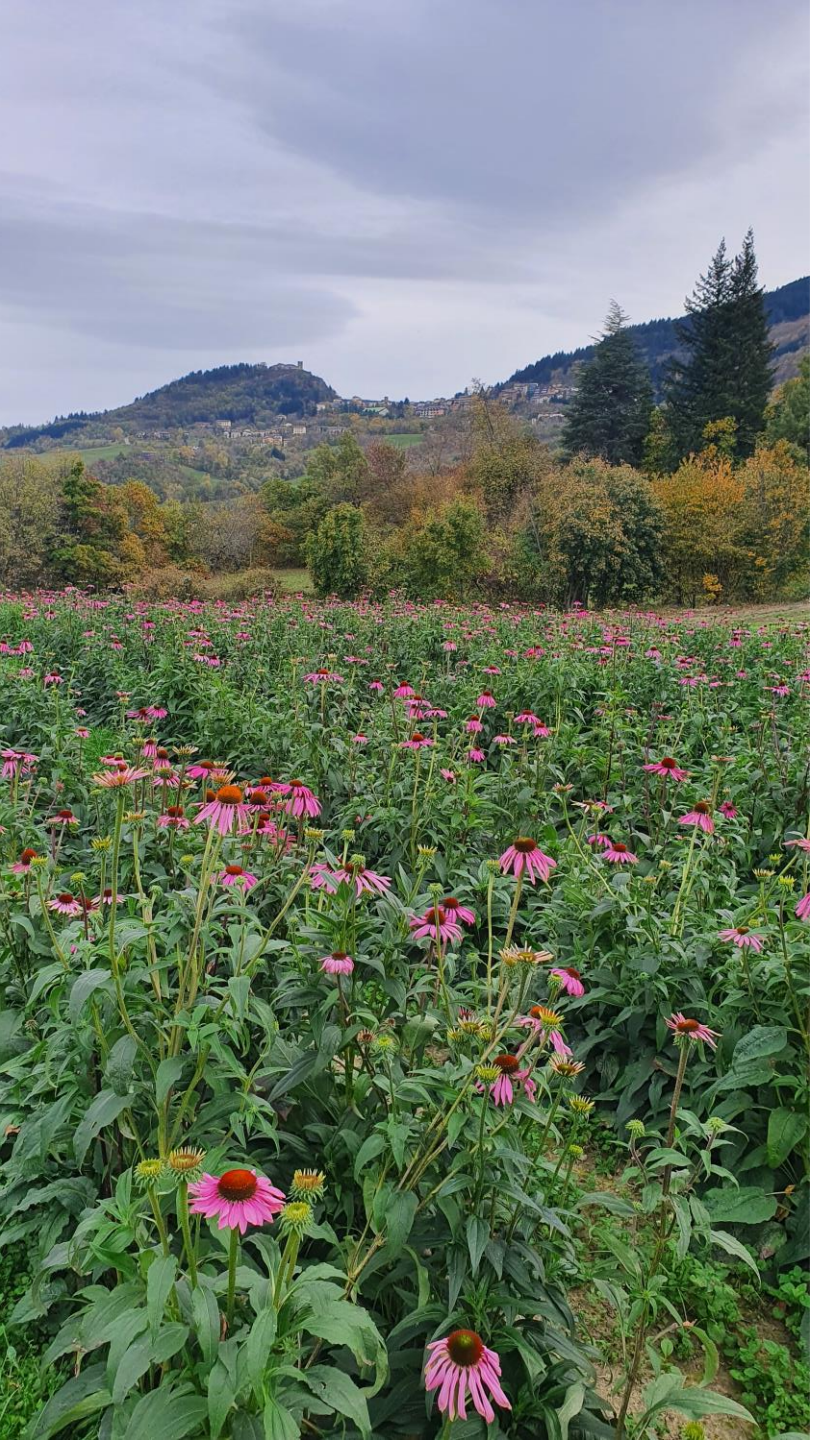
RegioneEmilia-Romagna

Macfrut 2023



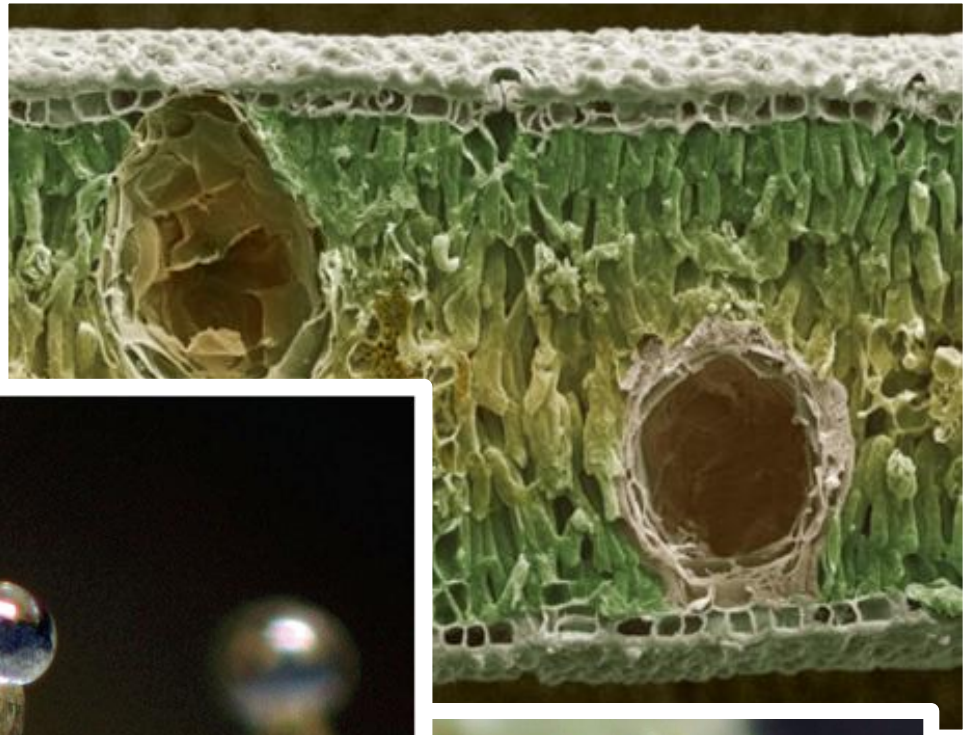
Piante officinali

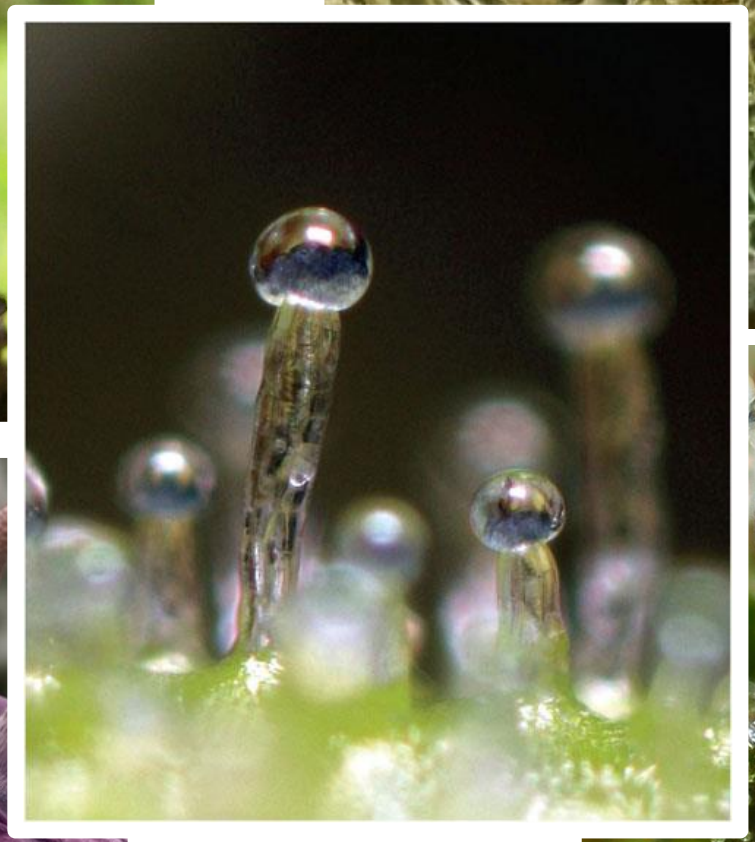
La definizione “**pianta officinale**” deriva da una tradizione culturale e storica del nostro paese ed è per la prima volta inserita nella legge n.99 del 6 gennaio 1931: *per piante officinali si intendono le piante medicinali, aromatiche e da profumo*.

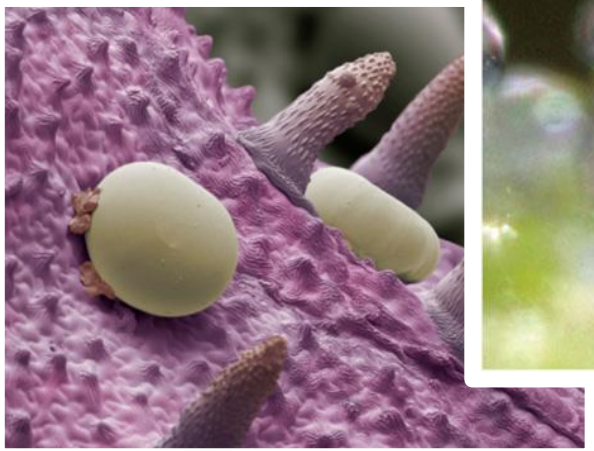
Il termine ”**officinale**” deriva dal latino *officina,* ossia il laboratorio dove le piante vengono sottoposte alle varie lavorazioni (essiccazione, triturazione, macerazione, estrazione di essenze, ecc.) in modo da renderle utilizzabili ai diversi scopi.

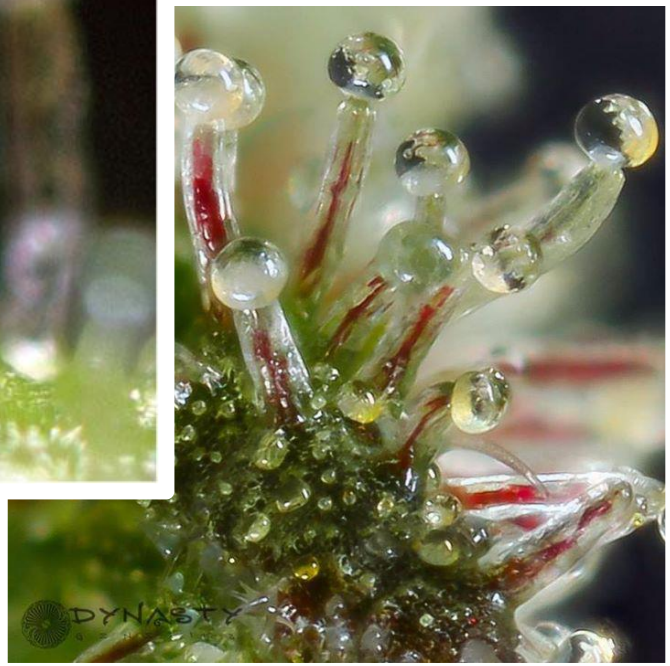
Secondo il DM 21 maggio 2018 n. 75, *Testo unico in materia di coltivazione, raccolta e prima trasformazione delle piante officinali* la tecnica di distillazione delle piante aromatiche è una procedura di prima trasformazione e quindi può essere condotta dal produttore in azienda agricola.

Origine









O.E. E PARTI DELLA PIANTA (droga)

sommità fiorite: achillea, camomilla, lavanda, rosmarino, timo…

foglie: eucalipto, verbena..

gemme: abete bianco, pino…

rametti: abete rosso, cipresso, pino…

frutti: anice, finocchio, ginepro

scorza dei frutti: arancio, bergamotto, limone….

corteccia: cannella, larice..

legno: canfora, sandalo…

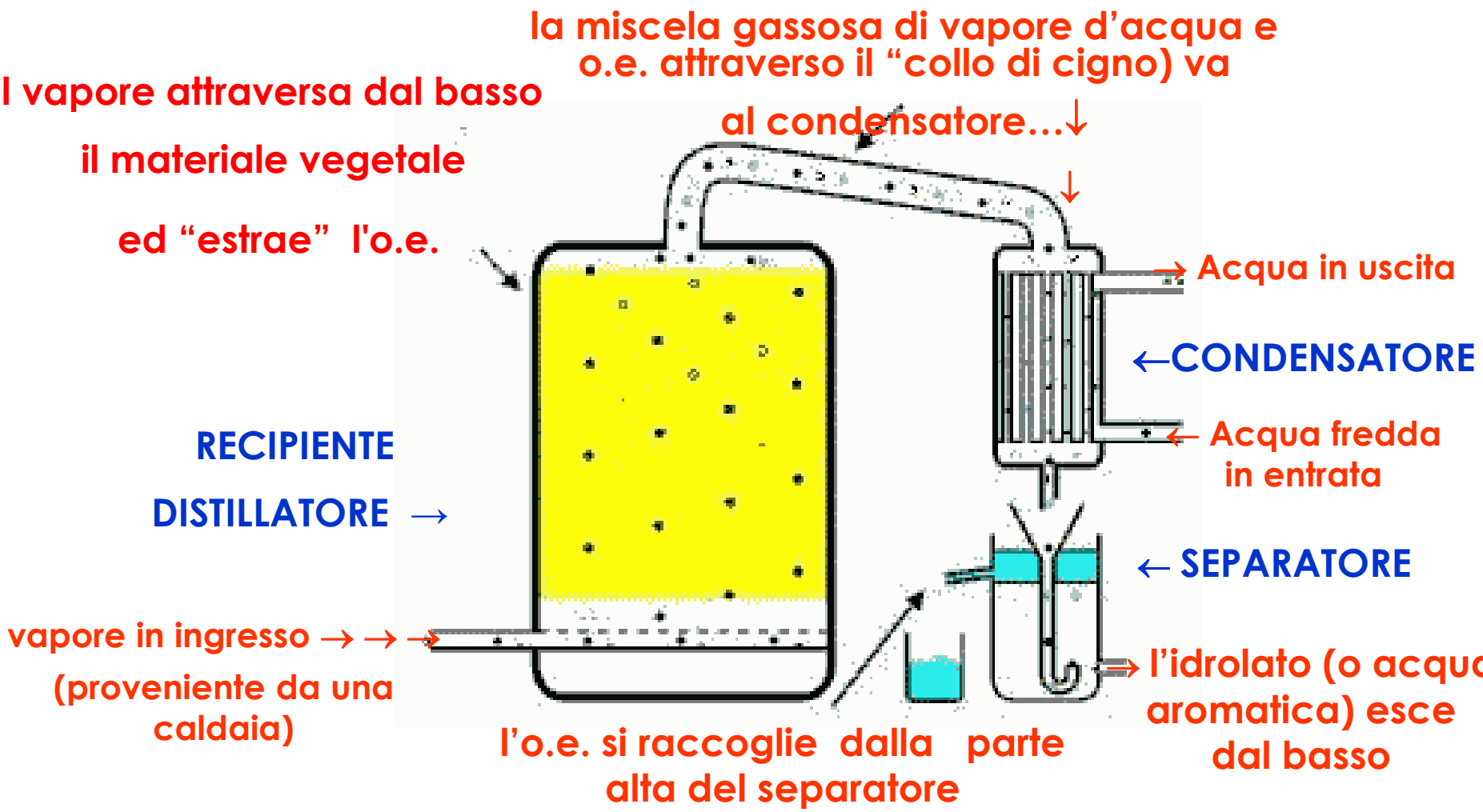
radici e rizomi: echinacea, iris, zenzero…

UNIMORE

METODI DI ESTRAZIONE DEGLI OLI ESSENZIALI

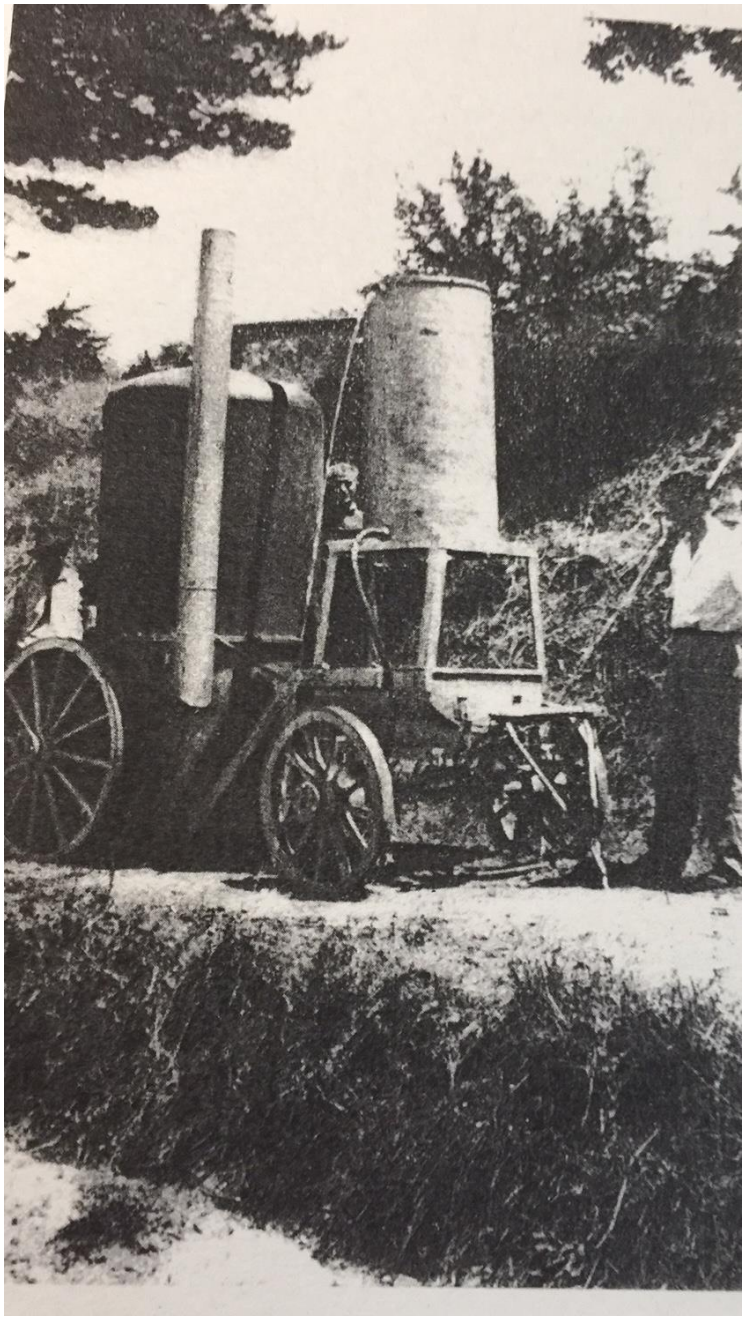
1) MECCANICI	2) FISICI	3) CHIMICI
"Abrasione","sfumatura""pelatura", direttamente su scorze o sul frutto intero. Di norma "essenze agrumarie", o.e. di piante di *Citrus*. Un tempo con metodi manuali, ora si usano apposite macchine	Distillazione passaggio di stato: liquido- aeriforme - liquido 2_{1a} in immersione 2_{1b} tecniche speciali (IDA, idrodistill.ne accelerata) 2_2 corrente di vapore(c.v.): 2_{2a} - a P ambiente (a fuoco diretto o altro) 2_{2b} - a P > ambiente (a produz. est. del vapore, da caldaia e diffusione): - impianto tradizionale - in "caisson" 2_3 Altri metodi: distill.ne in continuo	Estrazione con solventi 3_1 diretta sulla droga (- concreta - assoluta) 3_2 - con grassi e con solventi (vedi sopra): è l' enfleurage (a T amb. o a caldo) 3_3- con fluidi supercritici (S.F.E.), es. CO_2

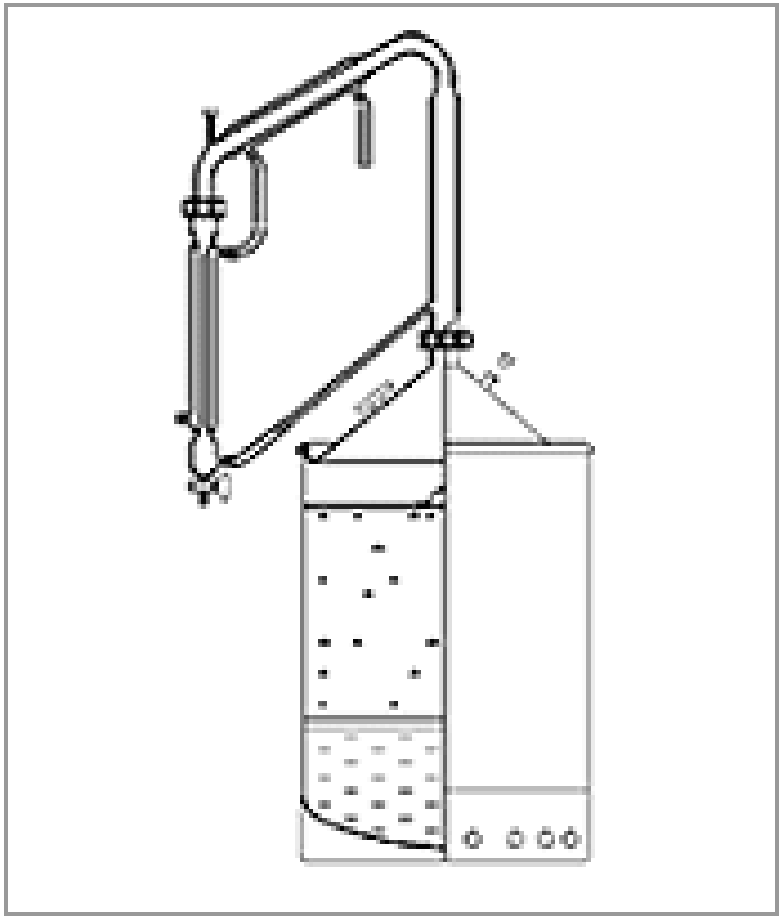
Distillazione

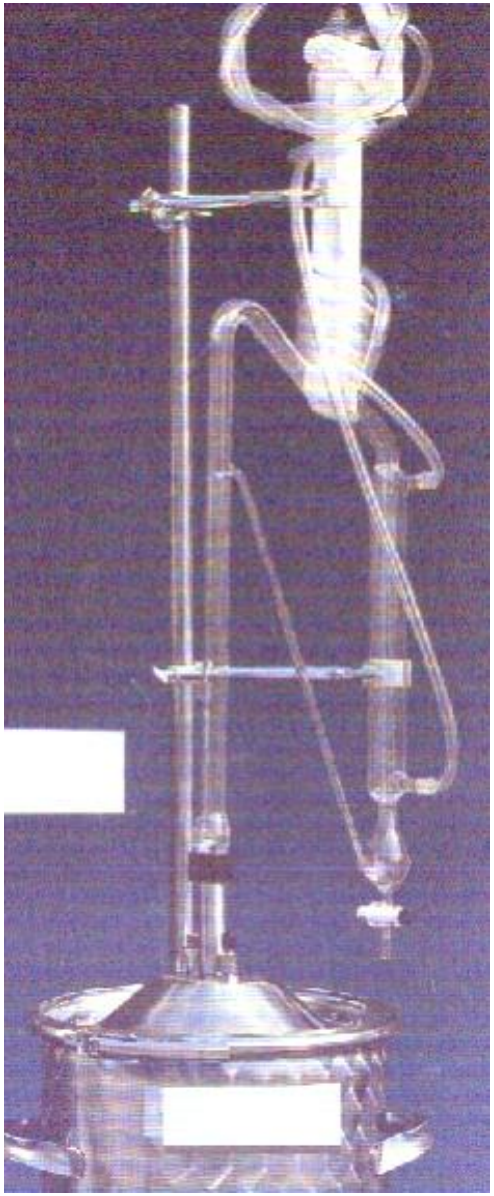


Schema del processo di distillazione in corrente di vapore

(impianto a produzione esterna di vapore)







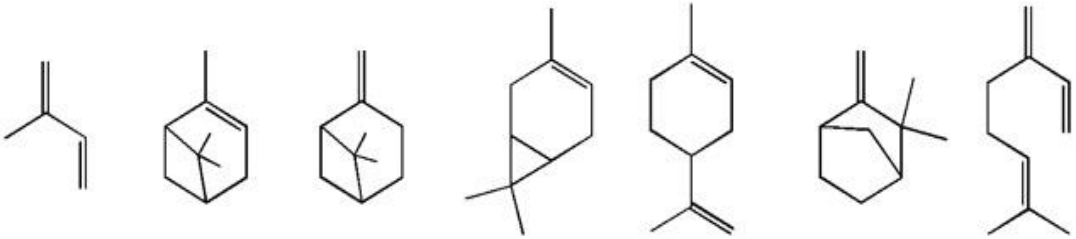
Sottoprodotti

Chimica

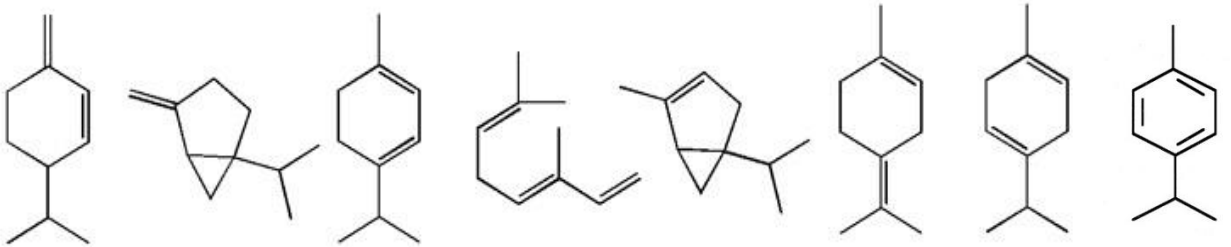
-Terpeni e terpenoidi

-2 o 3 componenti principali ad alte concentrazioni (20-95%)

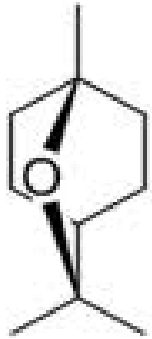
Monoterpeni (C_{10}) monoterpenoidi ($C_{10}O$)



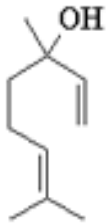
isoprene α-pinene β-pinene Δ^3-carene d-limonene camphene myrcene



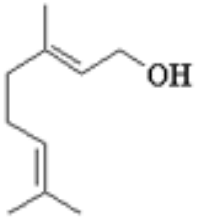
β-phellandrene sabinene α-terpinene ocimene α-thujene terpinolene γ-terpinene p-cymene



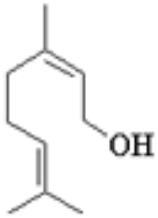
eucaliptolo



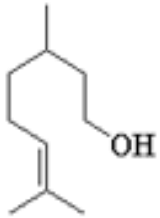
Linalolo



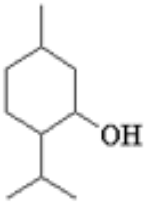
Geraniolo



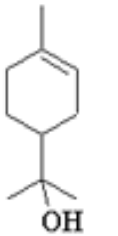
Nerolo



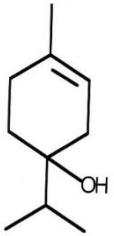
Citronellolo



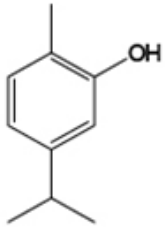
Mentolo



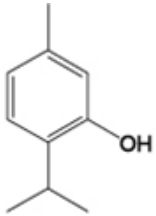
α-Terpineolo



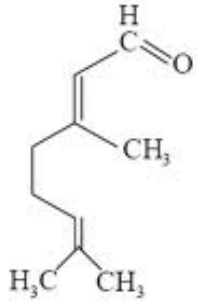
Terpinen-4-olo



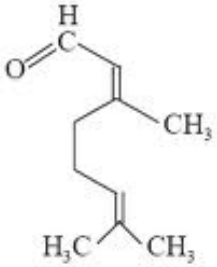
Carvacrolo



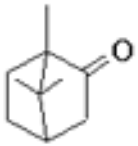
Timolo



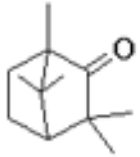
Geranial (Citral-a)



Neral (Citral-b)

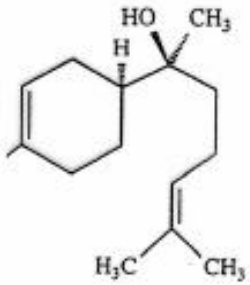


Canfora

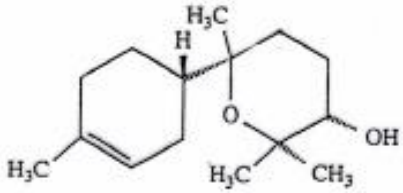


Carvone

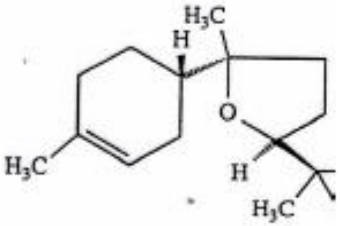
Sesquiterpeni



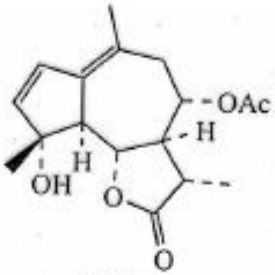
(-)-α-Bisabololo



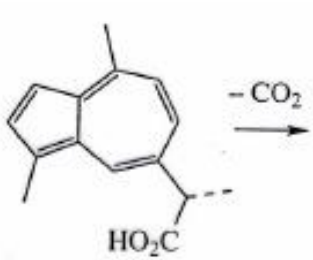
(-)-α-Bisabololossido A



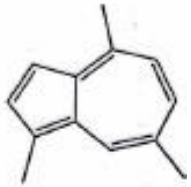
(-)-α-Bisabololossido



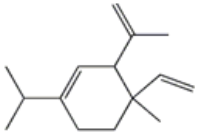
matricina



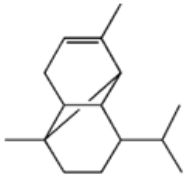
acido camazulen carbossilico



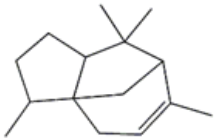
camazulene



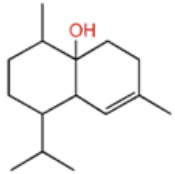
δ-elemene



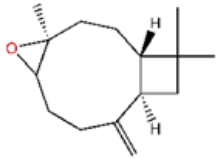
α-copaene



α-cedrene

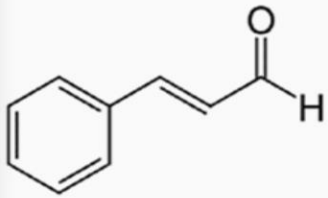


Cubenol

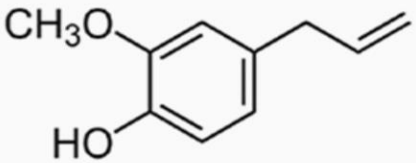


Caryophyllene oxide

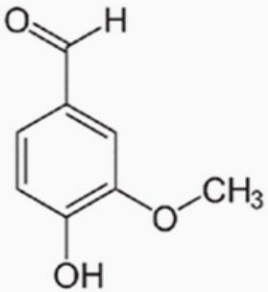
Phenylpropanoids



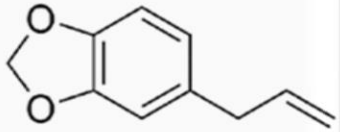
Cinnamaldehyde



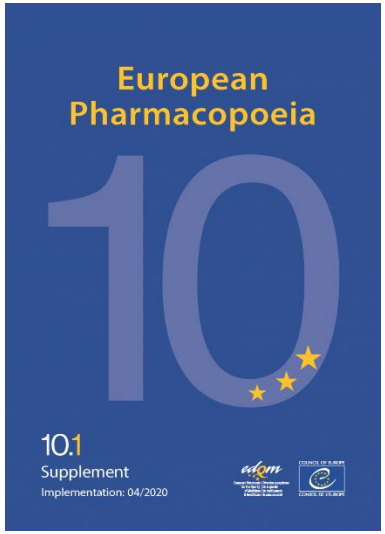
Eugenol



Vanillin



Safrole



ESSENTIAL OILS

Aetherolea

DEFINITION

Odorous product, usually of complex composition, obtained from a botanically defined plant raw material by steam distillation, dry distillation, or a suitable mechanical process without heating. Essential oils are usually separated from the aqueous phase by a physical process that does not significantly affect their composition.

Essential oils may be subjected to a suitable subsequent treatment. Thus an essential oil may be commercially known as being deterpenated, desesquiterpenated, rectified or 'x'-free.

LEMON OIL

Limonis aetheroleum

DEFINITION

Essential oil obtained by suitable mechanical means, without the aid of heat, from the fresh peel of *Citrus limon* (L.) Burman fil.

CHARACTERS

Appearance: clear, mobile, pale yellow or greenish-yellow liquid. It may become cloudy at low temperatures.

Characteristic odour.

TEA TREE OIL

Melaleucae aetheroleum

DEFINITION

Essential oil obtained by steam distillation from the foliage and terminal branchlets of *Melaleuca alternifolia* (Maiden and Betch) Cheel, *M. linariifolia* Smith, *M. dissitiflora* F. Mueller and/or other species of *Melaleuca*.

CHARACTERS

Appearance: clear, mobile, colourless or pale yellow liquid.

Characteristic odour.

LAVENDER OIL

Lavandulae aetheroleum

DEFINITION

Essential oil obtained by steam distillation from the flowering tops of *Lavandula angustifolia* Mill. (*Lavandula officinalis* Chaix).

CHARACTERS

Appearance: colourless or pale yellow, clear liquid.

Odour: complex, reminiscent of linalyl acetate.

LAVENDER OIL

Lavandulae aetheroleum

DEFINITION

Essential oil obtained by steam distillation from the flowering tops of *Lavandula angustifolia* Mill. (*Lavandula officinalis* Chaix).

CHARACTERS

Appearance: colourless or pale yellow, clear liquid.

Odour: complex, reminiscent of linalyl acetate.

IDENTIFICATION

First identification: B.

Second identification: A.

A. Thin-layer chromatography (2.2.27).

Test solution. Dissolve 20 µL of the oil to be examined in 1 mL of *toluene R*.

Reference solution. Dissolve 10 µL of *linalol R*, 10 µL of *cineole R* and 10 µL of *linalyl acetate R* in 1 mL of *toluene R*.

Plate: *TLC silica gel plate R* (5-40 µm) [or *TLC silica gel plate R* (2-10 µm)].

Mobile phase: *ethyl acetate R, toluene R* (5:95 *v/v*).

Application: 10 µL [or 2 µL] as bands of 10 mm [or 6 mm].

Development: over a path of 10 cm [or 8 cm].

Drying: in air.

Detection: spray with *anisaldehyde solution R* and heat at 100-105 °C for 5-10 min; examine immediately in daylight.

Results: see below the sequence of zones present in the chromatograms obtained with the reference solution and the test solution. Furthermore, other violet-red or greenish-brown zones are present in the chromatogram obtained with the test solution above the zone of linalyl acetate up to the solvent front.

Top of the plate	
	A violet-red or greenish-brown zone
Linalyl acetate: a violet or brown zone	A violet or brown zone (linalyl acetate) A violet-red zone
1,8-Cineole: a violet-brown zone Linalol: a violet or brown zone	Possibly a weak violet-brown zone (1,8-cineole) A violet or brown zone (linalol) A weak yellowish-brown zone Several unnamed zones
Reference solution	**Test solution**

B. Examine the chromatograms obtained in the test for chromatographic profile.

Results: the characteristic peaks in the chromatogram obtained with the test solution are similar in retention time to those in the chromatogram obtained with reference solution (a).

TESTS

Relative density (2.2.5): 0.878 to 0.892.

Refractive index (2.2.6): 1.455 to 1.466.

Optical rotation (2.2.7): −12.0° to 0.0°.

Acid value (2.5.1): maximum 1.0, determined on 5.0 g of the substance to be examined dissolved in 50 mL of the prescribed mixture of solvents.

Chromatographic profile. Gas chromatography (2.2.28): use the normalisation procedure.

Test solution. Dissolve 200 µL of the oil to be examined in *heptane R* and dilute to 10.0 mL with the same solvent.

Reference solution (a). Dissolve 5 µL of *limonene R*, 5 µL of *cineole R*, 5 µL of *3-octanone R*, 5 mg of *camphor R*, 40 µL of *linalol R*, 50 µL of *linalyl acetate R*, 30 µL of *terpinen-4-ol R*, 5 µL of *lavandulyl acetate R*, 5 µL of *lavandulol R* and 5 mg of *α-terpineol R* in *heptane R* and dilute to 10 mL with the same solvent.

Reference solution (b). Dissolve 5 µL of *limonene R* in *heptane R* and dilute to 50.0 mL with the same solvent. Dilute 0.5 mL of the solution to 5.0 mL with *heptane R*.

Column:

- *material*: fused silica;
- *size*: *l* = 60 m, Ø = 0.25 mm;
- *stationary phase*: *macrogol 20 000 R* (film thickness 0.25 µm).

Carrier gas: *helium for chromatography R*.

Flow rate: 1.5 mL/min.

Split ratio: 1:50.

Temperature:

	Time (min)	Temperature (°C)
Column	0 - 18	70
	18 - 70	70 → 180
Injection port		220
Detector		220

Detection: flame ionisation.

Injection: 1 µL.

Elution order: order indicated in the composition of reference solution (a). Record the retention times of these substances.

System suitability: reference solution (a):

- *resolution*: minimum 1.4 between the peaks due to terpinen-4-ol and lavandulyl acetate.

Using the retention times determined from the chromatogram obtained with reference solution (a), locate the components of reference solution (a) in the chromatogram obtained with the test solution.

Determine the percentage content of each of these components. The percentages are within the following ranges:

- *limonene*: maximum 1.0 per cent;
- *1,8-cineole*: maximum 2.5 per cent;
- *3-octanone*: 0.1 per cent to 5.0 per cent;
- *camphor*: maximum 1.2 per cent;
- *linalol*: 20.0 per cent to 45.0 per cent;
- *linalyl acetate*: 25.0 per cent to 47.0 per cent;
- *terpinen-4-ol*: 0.1 per cent to 8.0 per cent;
- *lavandulyl acetate*: minimum 0.2 per cent;
- *lavandulol*: minimum 0.1 per cent;
- *α-terpineol*: maximum 2.0 per cent;
- *disregard limit*: the area of the principal peak in the chromatogram obtained with reference solution (b) (0.05 per cent).

Chiral purity. Gas chromatography (2.2.28).

Test solution. Dissolve 0.02 g of the oil to be examined in *pentane R* and dilute to 10 mL with the same solvent.

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Etichetta della confezione

- Informazioni generali e obbligatorie:

NOME BOTANICO (genere, specie, varietà), lotto, produttore, scadenza, peso/volumi

- Indicazioni di pericolo
- Consigli di prudenza
- Nome e recapito del produttore
- Pittogrammi

Scheda di sicurezza

- Se la sostanza e/o miscela è classificata come pericolosa (dir.1967/548 CEE; 1999/45/CE; CLP)
- Ha un limite di esposizione comunitario
- E' un PBT (Sostanze Persistenti – Bioaccumulabili – Tossiche)
- Ha lo scopo di ridurre i pericoli per i lavoratori e per l'ambiente;
- È un'autodichiarazione;
- Corredata di dati analitici essenziali;
- Documento che deve riportare tutte le informazioni all'operatore affinchè possa utilizzare il prodotto in modo appropriato e sicuro.

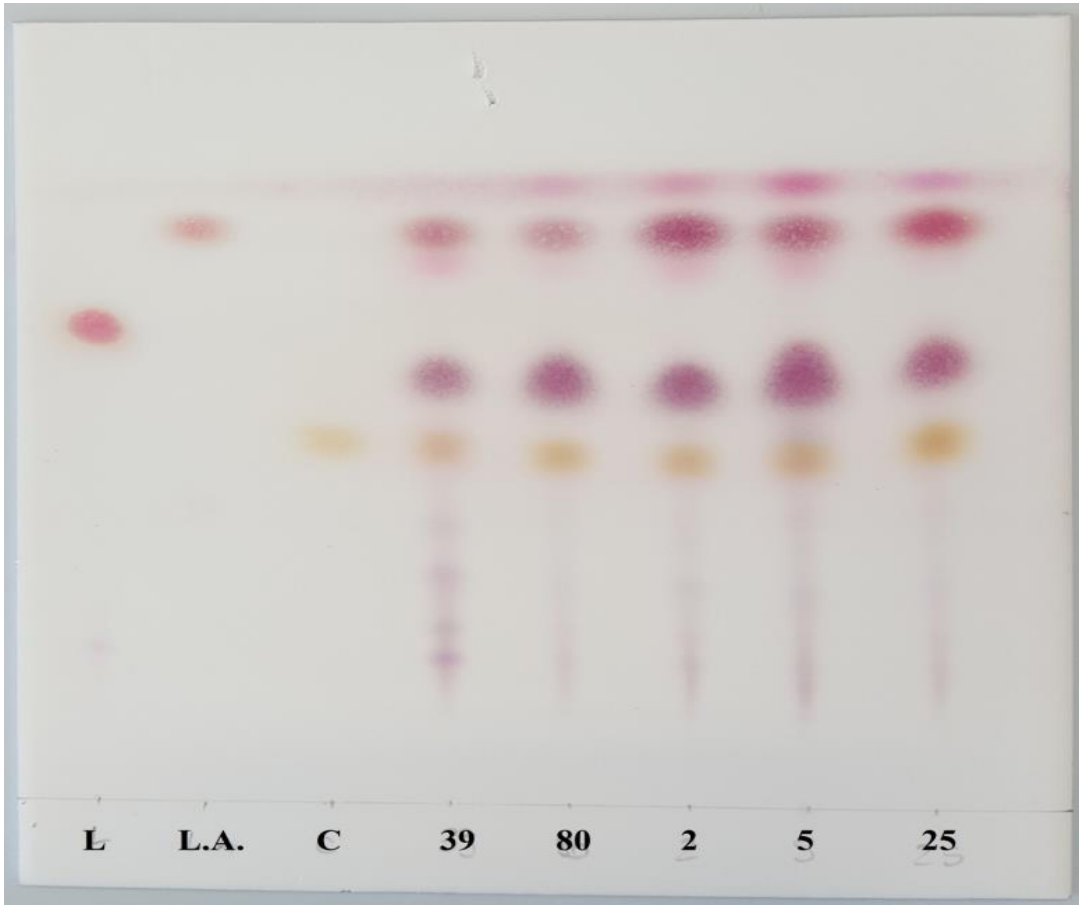
Scheda tecnica

- Ha fini commerciali
- Funge da dichiarazione per il prodotto è per uso alimentare o non
- Può dare indicazioni circa le modalità di manipolazione e stoccaggio
- È una «autocertificazione»»

Controllo di qualità

- cromatografia su strato sottile (TLC e HPTLC),
- gascromatografia (GC-FID, GC-MS),
- cromatografia liquida (HPLC),
- tecniche spettroscopiche (tecniche vibrazionali e risonanza magnetica nucleare).

Cromatografia su strato sottile



L = standard di linalolo,

L.A. = standard di acetato di linalile,

C = standard di canfora,

39 e 80 = oli essenziali di *Lavandula x intermedia*,

2-5-25 : oli essenziali di *Lavandula angustifolia*

Ylang-Ylang oil (*Cananga odorata*)

1. Scope

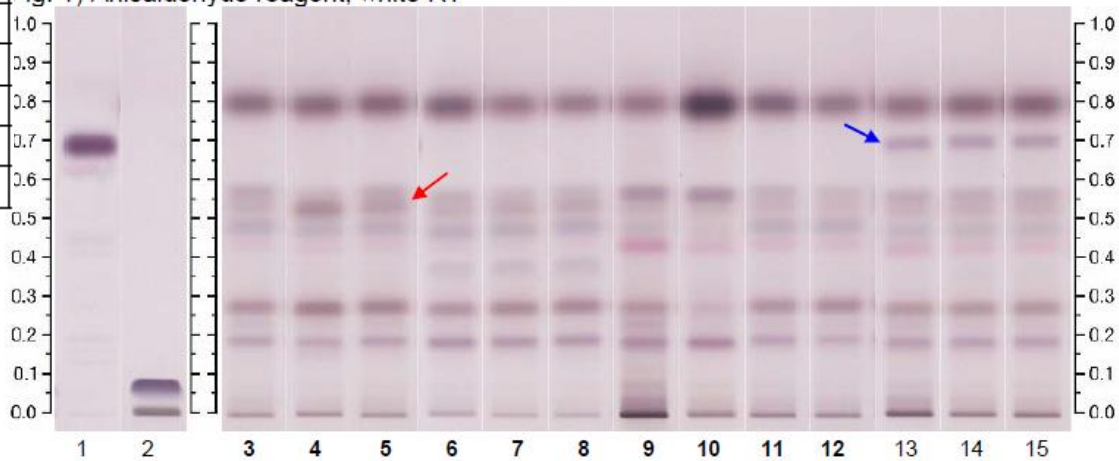
This method identifies Ylang-Ylang essential oil (*Cananga odorata* Hook. f. & Thomson) by HPTLC fingerprint and detects adulteration with fatty oils.

Track	Volume	Sample
1	2 µL	Sunflower oil
2	2 µL	Linoleic acid
3	**2 µL**	**Ylang-Ylang oil 1**
4	**2 µL**	**Ylang-Ylang oil 2**
5	**2 µL**	**Ylang-Ylang oil 3**
6	**2 µL**	**Ylang-Ylang oil 4**
7	**2 µL**	**Ylang-Ylang oil 5**
8	**2 µL**	**Ylang-Ylang oil 6**
9	**2 µL**	**Ylang-Ylang oil 7**
10	**2 µL**	**Ylang-Ylang oil 8**
11	**2 µL**	**Ylang-Ylang oil 9**
12	**2 µL**	**Ylang-Ylang oil 10**
13	2 µL	Ylang-Ylang oil, with adulteration
14	2 µL	Ylang-Ylang oil, with adulteration
15	2 µL	Ylang-Ylang oil, with adulteration

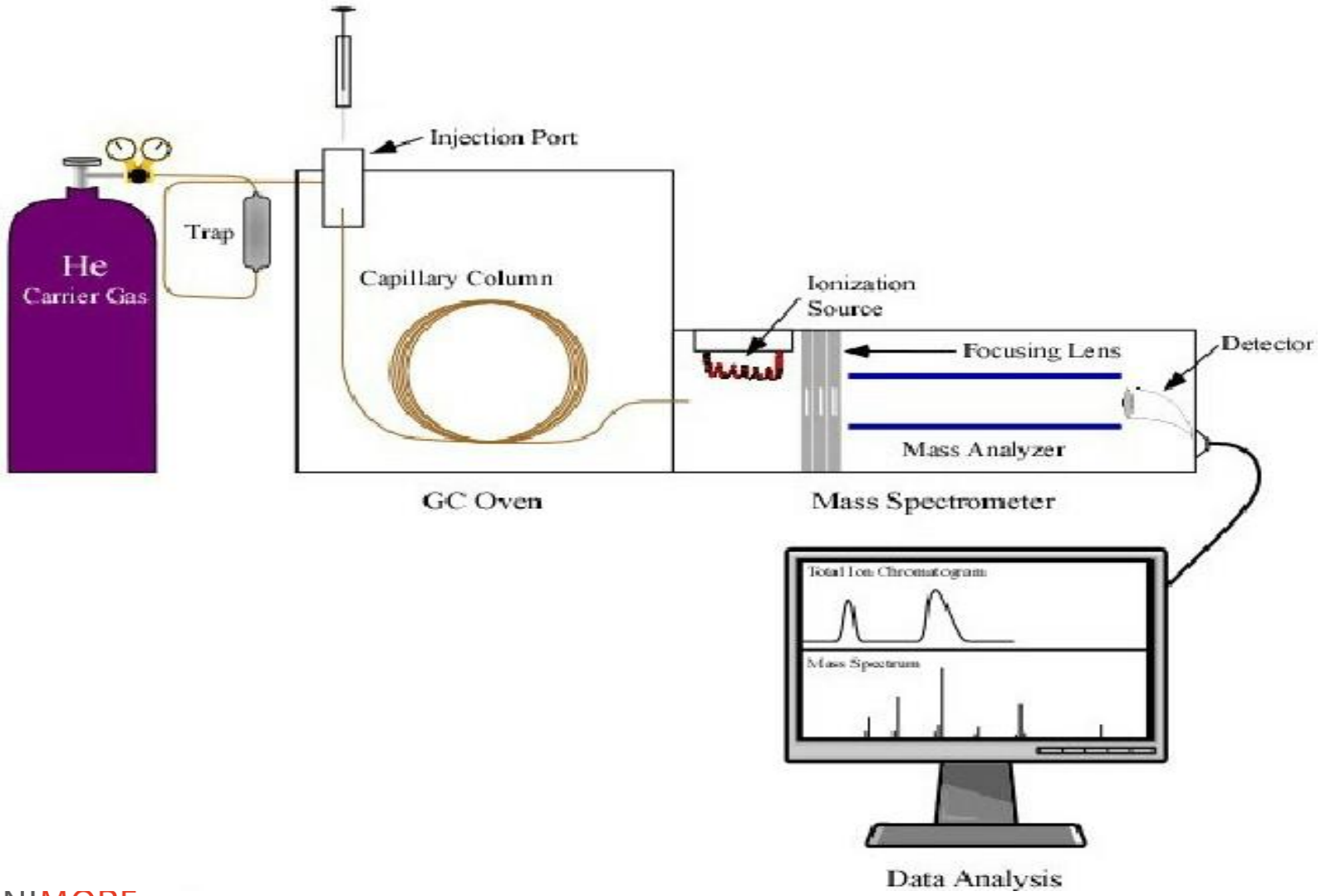
→ Fatty acids

→ EO components

Fig. 1) Anisaldehyde reagent, white RT



Gascromatografia



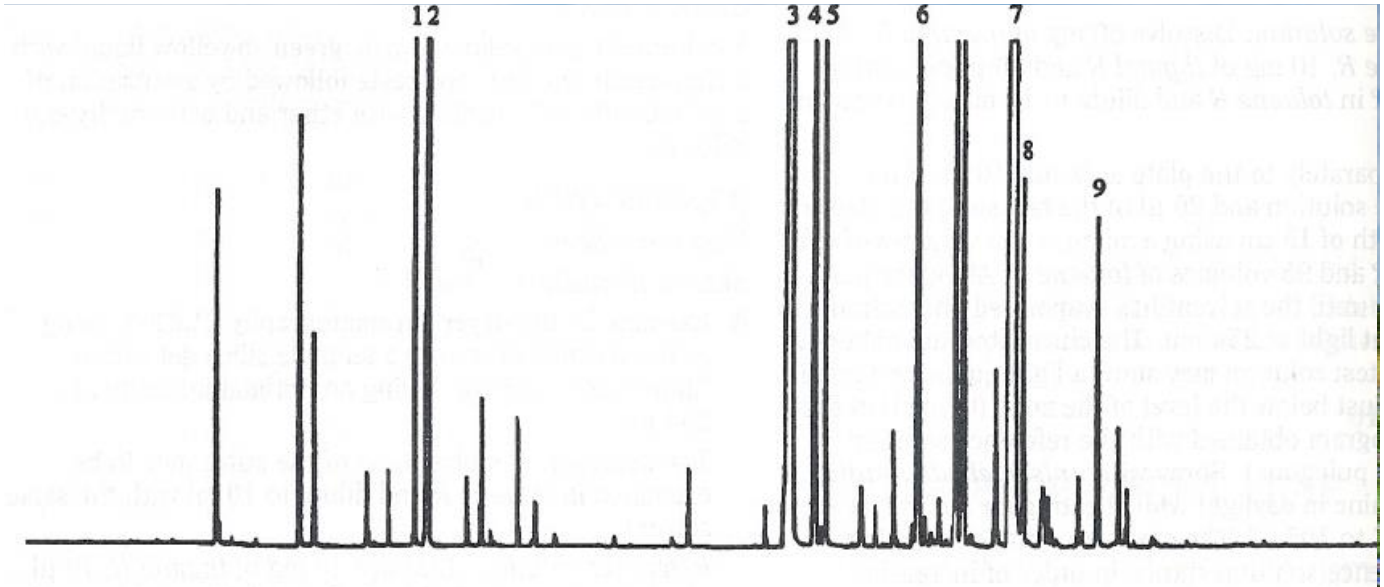
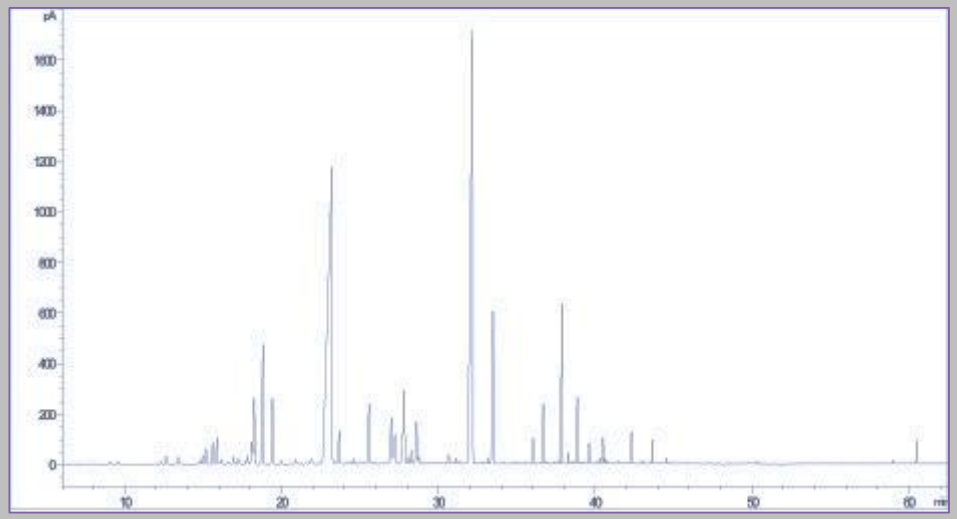


Figure 0405-1. – *Type chromatogram for peppermint oil*

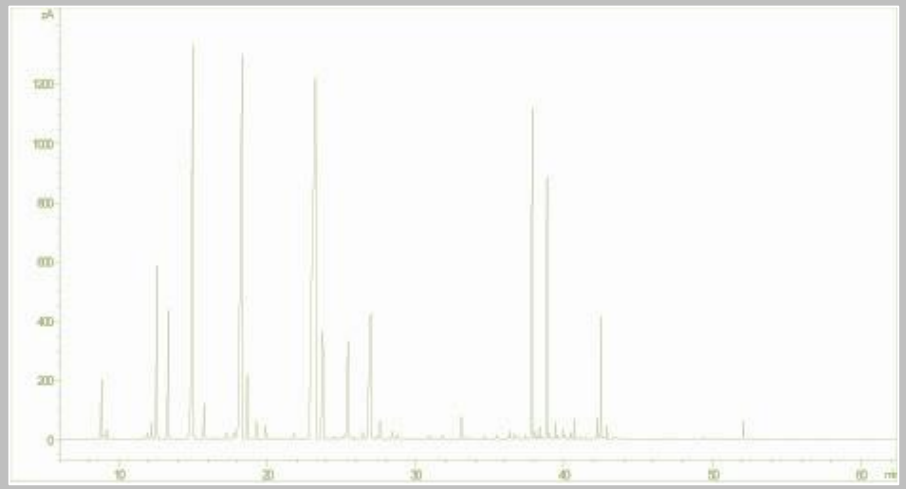
1. limonene
2. cineole
3. menthone
4. menthofuran
5. isomenthone
6. menthyl acetate
7. menthol
8. pulegone
9. carvone

Analisi semiquantitativa

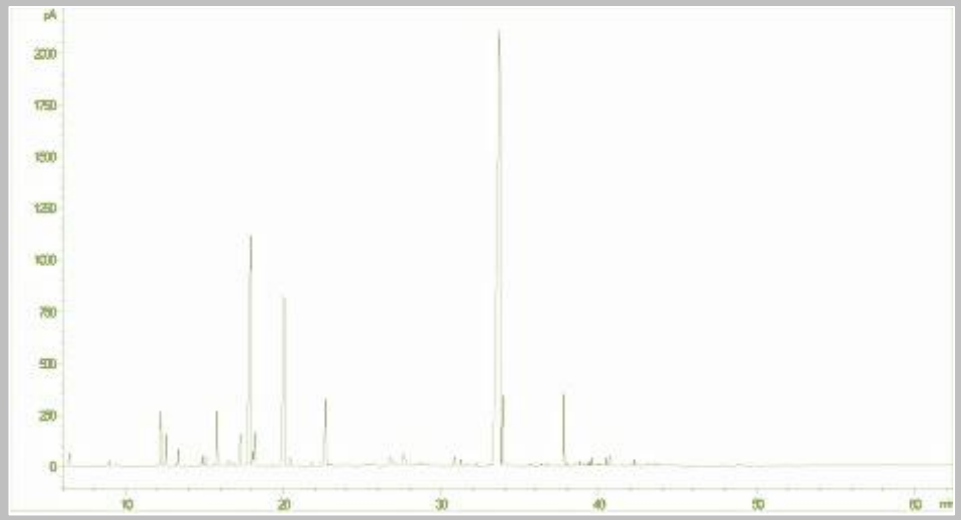
$$a_1' = (a_1/a_1 + a_2 + a_3 + \dots a_n) \times 100$$



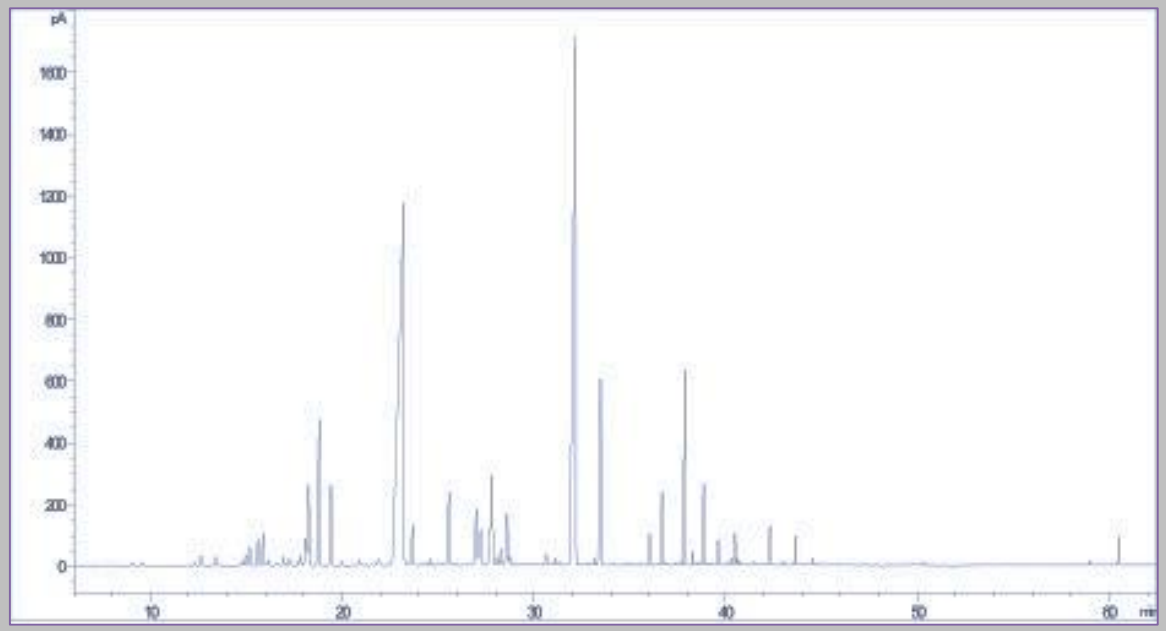
Lavandula angustifolia



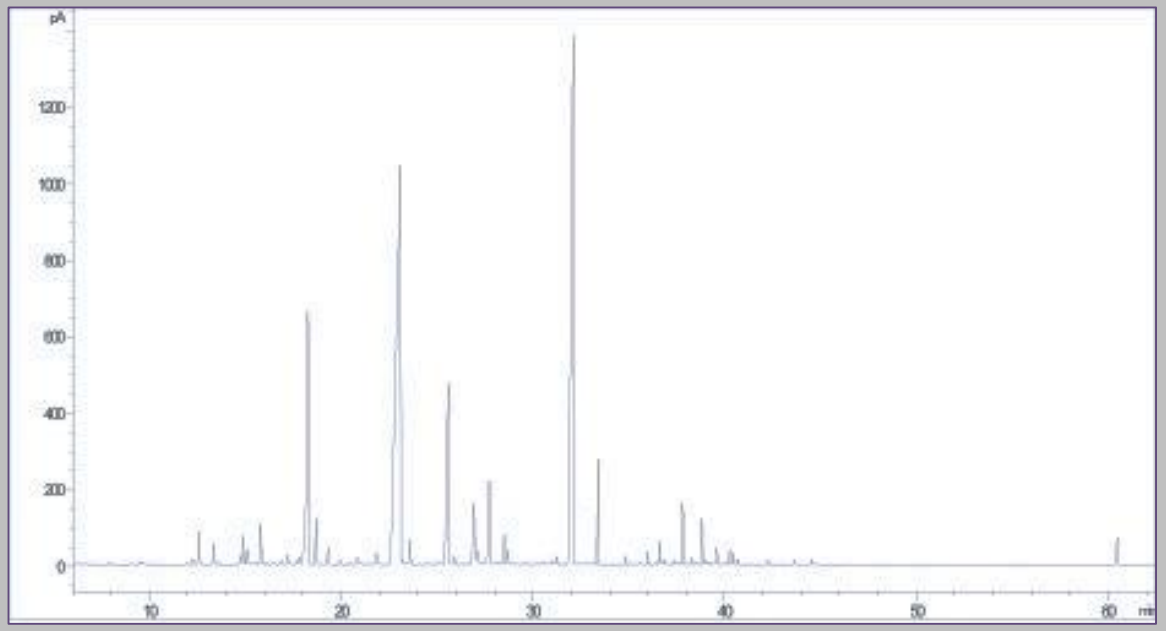
Salvia officinalis



Thymus vulgaris



Lavandula angustifolia



Lavandula x intermedia

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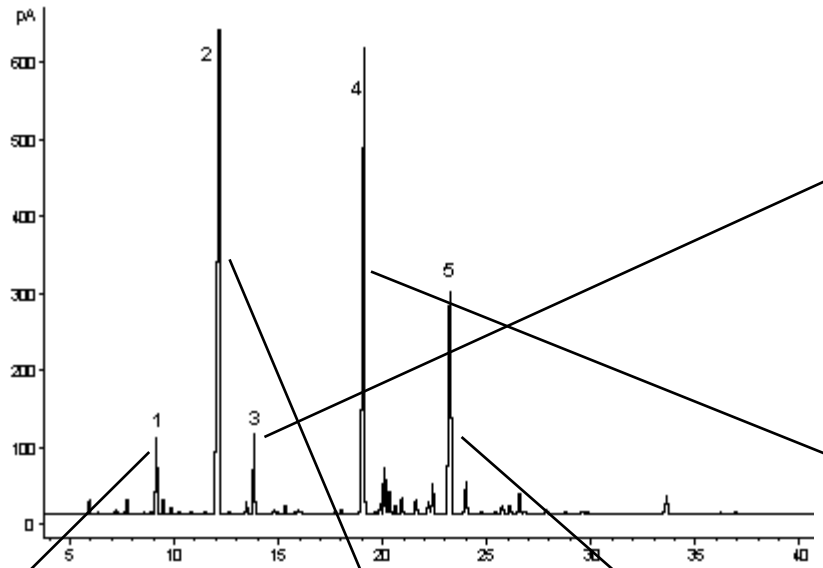
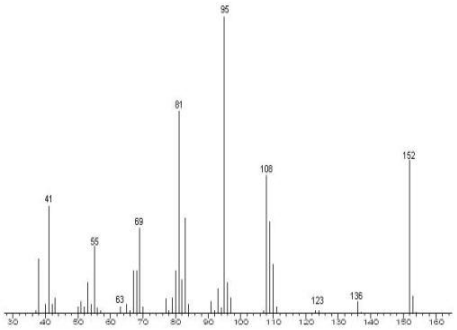
	LRI	Area%	Area%	Area%
alpha thujene	924	0,13	0,18	0,13
alpha pinene	930	0,32	0,45	0,33
camphene	945	0,24	0,25	0,25
beta pinene	977	0,16	0,18	0,16
3-octanone	985	0,85	1,02	0,89
myrcene	990	0,99	1,68	0,93
3-octanol	995	0,18	0,21	0,17
3-carene	1008	0,37	0,29	0,38
alpha terpinene	1013	0,42	0,48	0,43
o-cymene	1022	0,16	0,14	0,14
p cymene	1026	1,31	1,40	1,67
limonene	1029	1,27	0,84	1,08
cis ocimene	1038	8,61	11,75	8,99
trans-ocimene	1047	2,99	4,46	3,07
gamma terpinene	1057	0,18	0,30	0,18
trans linalool oxide	1087	0,14	0,31	0,14
linalool	1104	29,53	32,73	28,58
cis rose oxide	1113	0,74	0,91	0,72
trans rose oxide	1129	0,36	0,53	0,36
camphor	1143	0,53	0,26	0,54
isomenthone	1165	0,85	0,54	0,81
neomenthol	1168	0,65	0,69	0,62
terpinen-4-ol	1178	4,23	4,44	4,08
alpha terpineol	1190	0,65	1,77	0,63
lynalil acetate	1264	29,93	19,60	29,30
lavandulyl acetate	1293	3,62	3,59	3,46
neryl acetate	1367	0,25	0,52	0,24
beta cubebene	1385	0,41	0,90	0,42
beta caryophyllene	1424	3,94	3,77	3,78
alpha bergamotene	1439	0,14	0,13	0,13
alpha humulene	1460	2,99	2,79	2,91
ar curcumene	1486	0,39	0,30	0,41
gamma cadinene	1519	0,16	0,10	0,17
caryophyllene oxide	1591	0,17	0,11	0,16
		97,87	97,58	96,23

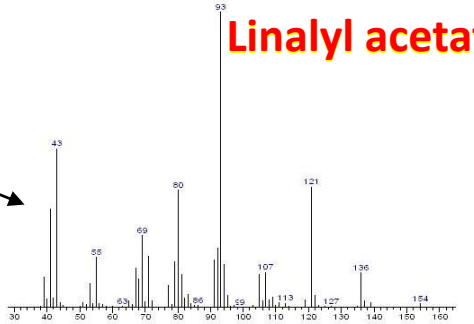
GC-MS



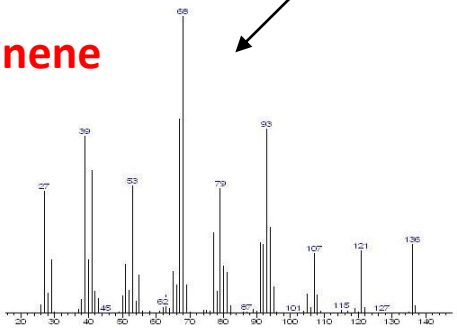
Camphor

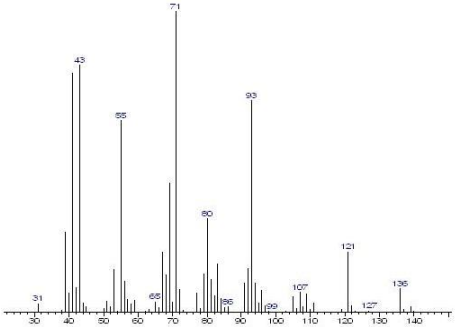


Linalyl acetate

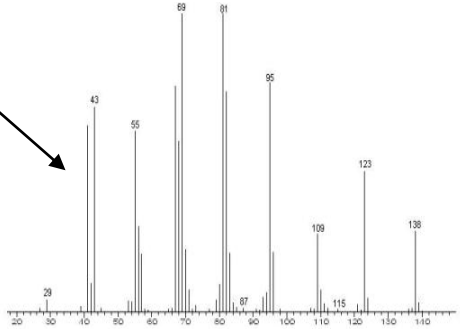


Limonene





Linalool



Citronellyl acetate

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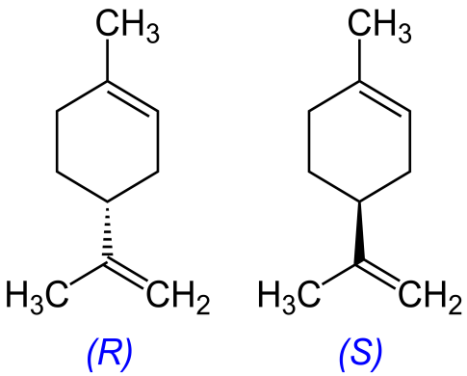
Analisi chirale

I composti chirali si presentano in natura in specifici rapporti enantiomerici (stereospecificità), mentre quelli sintetici sono in forma racemica (1:1).

Gli enantiomeri possono esibire attività biologiche anche divergenti; un'eventuale adulterazione può essere riconosciuta per la presenza del racemo.

Limonene



orange odour **lemon odour**

Grazie per la vostra attenzione

stefania.benvenuti@unimore.it

RegioneEmilia-Romagna

Macfrut 2023