

# Comparative Investigations of the Essential Oil and Volatiles of Spearmint

by Leopold Jirovetz, Gerhard Buchbauer and Manochehr Shahabi, University of Vienna, Vienna, Austria, and Martin Benoit Ngassoum, University of Ngaoundere, Ngaoundere, Cameroon

Essential oils and extracts of *Mentha spicata* have long been of high importance in food-product, perfumery and cosmetic applications due to their intense, pleasant, clear-fresh odor. In addition to this species of the *Labiatae* family, *Mentha piperita* L. (peppermint) and *Mentha arvensis* L. (cornmint) are also used extensively.<sup>8</sup> Many analyses of the aroma compounds of *M. spicata* essential oils and extracts have been performed in the past, not only to identify these volatiles, which are responsible for the characteristic aroma, but also to study biological activities of spearmint samples.<sup>2-4,6,21,22,24-30</sup>

To the best of our knowledge no comparative studies on the volatiles of *M. spicata* oil from Cameroon and its corresponding solid-phase-microextraction (SPME)-headspace sample have been available until now. Therefore the objective of this work is to identify the fragrance compounds of this oil and its SPME-headspace by means of gas chromatographic-spectroscopic (GC and GC/MS using different polar and chiral phases) and olfactory methods (GC-sniffing technique and olfactory correlations) to find out the importance of each single constituent with their specific odor attributes, responsible for the characteristic and pleasant spearmint aroma for the first time.

## Results and Discussion

Professional perfumers olfactively evaluated the essential oil of *M. spicata* L. from Cameroon as follows: pleasant fresh spearmint (carvone, limonene, 1,8-cineole) aroma (top note), with green (leaf-like), floral (similar to rose and lavender), fruity (similar to citrus and peaches), and spicy (similar to pepper and caraway) side notes. Additionally, the perfumers generally identified a high olfactive quality.

For the trapping of the spearmint oil volatiles in the headspace, we used SPME, an effective method for concentrating odor-active constituents of complex aroma samples (e.g. essential oils) before identification by means of hyphenated systems (GC, GC-MS, etc.).<sup>11,12,14-18,23,30</sup>

As a result of GC and GC/MS analyses (using two achiral phase columns of different polarity and one chiral phase fused silica), more than 60 volatiles were detected; more than 50 were identified in the oil and SPME-headspace

sample of *M. spicata*. Main compounds (concentration higher than 1.0 percent, calculated as percentage-peak area of GC analysis using a polar [Carbowax] column) were found to be (essential oil/SPME headspace): (-)-carvone (69.34 percent/66.77 percent), (-)-limonene (6.55 percent/8.31 percent), 1,8-cineole (4.19 percent/7.12 percent), *cis*-carveol (2.14 percent/1.86 percent), germacrene D (1.78 percent/0.92 percent) and *trans*-1-hexen-3-ol (0.66 percent/1.72 percent). Further constituents, especially mono- and sesquiterpenes, hexane- and butane-derivatives were identified in concentrations from trace (< 0.01 percent) to 0.92 percent in the investigated samples (see Tables I and II). Difference in the concentrations of the essential oil and the corresponding SPME-headspace main constituents were only significant for limonene (6.55 percent/8.31 percent), 1,8-cineole (4.19 percent/7.12 percent) and *trans*-1-hexen-3-ol (0.66 percent/1.72 percent).

For a comparison of the odor impression of each single identified compound of the essential *M. spicata* oil from Cameroon to the above-described total aroma, a GC-sniffing technique was used. The obtained data of this analysis were correlated with published aroma attributes for each identified constituent in the essential spearmint oil published elsewhere.<sup>1,5,24,31</sup> As result of these combined data interpretation, we state the following (see Table III): the characteristic spearmint aroma with an intense fresh carvone-limonene-1,8-cineole top-note can be attributed to the identified main compounds of the essential oil and its corresponding SPME-headspace sample of *M. spicata* from Cameroon, namely (-)-carvone, (-)-limonene and 1,8-cineole, as well as further constituents with spearmint/mint aroma, like  $\alpha$ -phellandrene, *trans*- and *cis*-dihydrocarvone, (+)-menthol, (+)-pulegone, dihydrocarvyl acetate, *trans*- and *cis*-carvyl acetate, and *trans*- and *cis*-carveol; green (leaf-like) odor impressions were found to have hexanal, *cis*-3-hexen-1-ol ("leaf alcohol"), *trans*-1-hexen-3-ol, and partly 3-methyl butanal, 2-methyl butanal,  $\beta$ -bourbonene, and  $\beta$ -elemene; odor compounds with floral notes (direction of rose and lavender) are linalool, linalyl acetate, citronellyl acetate,  $\alpha$ -terpineol, geraniol and *cis*-jasmone, with fruity notes (similar to citrus and peaches), 3-methyl butanal, 2-methyl butanal, pentanal,  $\alpha$ -terpinene,  $\delta$ -3-

**Table I. Compounds of the essential oil (EO) of *Mentha spicata* L. from Cameroon and the corresponding SPME-headspace sample (SPME) in order of their retention indices (RI, using a polar column) and concentrations in percentage (calculated as percentage-peak area of GC analysis)**

Compound	EO1	SPME2	RI	Compound	EO1	SPME2	RI
3-methyl butanal	0.25	0.37	912	$\beta$ -bourbonene	0.19	0.12	1518
2-methyl butanal	0.10	0.22	917	linalyl acetate	0.11	0.08	1537
pentanal	nd	0.02	941	$\beta$ -elemene	0.45	0.37	1572
2-butanol	0.01	0.01	998	<i>trans</i> -dihydrocarvone	0.34	0.31	1601
$\alpha$ -pinene	0.17	0.45	1007	terpinen-4-ol	0.08	0.07	1608
camphene	0.41	0.92	1047	<i>cis</i> -dihydrocarvone	0.27	0.21	1610
$\beta$ -pinene	0.31	0.22	1072	$\beta$ -caryophyllene	0.42	0.16	1617
sabinene	0.14	0.05	1108	menthol	0.21	0.56	1623
$\beta$ -myrcene	0.88	0.36	1143	$\alpha$ -terpineol	0.53	0.36	1657
$\alpha$ -terpinene	0.15	0.09	1149	$\alpha$ -humulene	0.18	0.18	1659
$\alpha$ -phellandrene	0.44	0.24	1151	pulegone	0.24	0.13	1662
$\delta$ -3-carene	0.02	0.03	1154	<i>trans</i> - $\beta$ -farnesene	0.07	0.06	1666
limonene	6.55	8.31	1184	dihydrocarvyl acetate	0.22	0.23	1676
1,8-cineole	4.19	7.12	1219	borneol	0.26	0.18	1694
$\gamma$ -terpinene	0.12	0.18	1222	germacrene D	1.78	0.92	1711
<i>cis</i> - $\beta$ -ocimene	0.21	0.06	1225	<i>trans</i> -dihydrocarveol	0.19	0.17	1714
p-cymene	0.52	0.23	1244	bicyclogermacrene	0.06	0.03	1717
<i>cis</i> -3-hexenol	0.04	0.06	1248	carvone	69.34	66.77	1719
<i>trans</i> - $\beta$ -ocimene	0.56	0.38	1251	<i>cis</i> -dihydrocarveol	0.31	0.19	1726
terpinolene	0.05	0.44	1285	<i>trans</i> -carvyl acetate	1.23	0.99	1758
<i>trans</i> -1-hexen-3-ol	0.66	1.72	1302	<i>trans</i> -carveol	0.66	0.57	1789
hexanol	0.12	0.09	1329	<i>cis</i> -carvyl acetate	0.18	0.14	1796
3-octanol	0.17	0.14	1388	germacrene A	0.23	0.19	1802
<i>trans</i> -sabinene hydrate	0.02	t	1454	<i>cis</i> -carveol	2.14	1.86	1819
$\alpha$ -copaene	0.77	0.45	1475	geraniol	0.87	0.74	1822
linalool	0.48	0.87	1502	<i>cis</i> -jasnone	0.80	0.66	1871

nd=not detected; t=trace compound (< 0.01 percent)

carene, limonene,  $\gamma$ -terpinene, p-cymene, 3-octanol, citronellyl formate,  $\alpha$ -terpineol, geraniol and *cis*-jasnone as well as with spicy notes (direction of pepper and caraway) *cis*- $\beta$ -ocimene, *trans*- $\beta$ -ocimene, *trans*-sabinene hydrate, terpinen-4-ol,  $\beta$ -caryophyllene, germacrene D, *trans*-carveol, and *cis*-carveol.

To summarize this aroma compound analysis of an essential oil of *M. spicata* L. from Cameroon, we can state that the odor impression was found to be a very pleasant spearmint one, with green, floral, fruity and spicy side-notes. The composition of the essential spearmint oil and its corresponding SPME-headspace sample is very similar and differs only (but significantly) in the concentrations of the main compounds (-)-limonene (essential oil: 6.55 percent, SPME: 8.31 percent), 1,8-cineole (4.19 percent, 7.12 percent) and *trans*-1-hexen-3-ol (0.66 percent, 1.72 percent). In addition to the composition of both samples, the olfactive evaluations certify a high quality of the investigated essential spearmint oil from Cameroon and a possible use in food, perfumery and cosmetic products requiring a fresh-spearmint odor.

**Table II. Optical purity of target compounds of the essential spearmint oil from Cameroon with fresh and/or spearmint aroma by means GC and GC/MS with chiral phase columns<sup>10,13,27,30</sup>**

Compound	(+)	(-)
limonene		#
<i>trans</i> -dihydrocarvone	ns	ns
<i>cis</i> -dihydrocarvone	ns	ns
menthol	#	
pulegone	#	
dihydrocarvyl acetate	#	#
<i>trans</i> -dihydrocarveol	ns	ns
carvone		#
<i>trans</i> -carvyl acetate		#
<i>trans</i> -carveol	#	#
<i>cis</i> -carvyl acetate		#
<i>cis</i> -carveol	#	#

ns=no chiral separation found

**Table III. Compounds of the essential oil and SPME-headspace sample of *Mentha spicata* from Cameroon and their corresponding aroma impressions in accordance to published data and obtained by the use of a GC-sniffing-technique (GCST)<sup>1,5,24,31</sup>**

Compound	Aroma impressions	GCST <sup>*</sup>
3-methyl butanal	fruity, green, weak woody	fruity
2-methyl butanal	fruity, green	fruity
pentanal	woody, vanilla, fruity, nutty on dilution	warm
2-butanol	medicinal, ethereal	ethereal
$\alpha$ -pinene	woody, pine-like	piney
camphene	camphoraceous, fresh	fresh
$\beta$ -pinene	woody, pine-like	piney
sabinene	weak turpentine-like, spicy, warm-woody	spicy
$\beta$ -myrcene	sweet-balsamic, spicy	sweet
$\alpha$ -terpinene	spicy, citrus-note	spicy
$\alpha$ -phellandrene	minty, herbaceous	herbal
$\delta$ -3-carene	sweet, refined limonene-note, penetrating	sweet
limonene	fresh, citrus-, lemon- and orange-note	citrus-like
$\gamma$ -terpinene	herbaceous, citrus-like	citrus-like
1,8-cineole	fresh, eucalyptus-like	eucalyptus
<i>cis</i> - $\beta$ -ocimene	spicy (estragon- and basil-notes)	spicy
p-cymene	weak citrus-note	citrus-like
<i>cis</i> -3-hexen-1-ol	green ("leaf alcohol"), fresh-grass-like	green
<i>trans</i> - $\beta$ -ocimene	spicy (estragon- and basil-notes)	spicy
terpinolene	sweet-piney, slightly sweet-anisic	terpenic
<i>trans</i> -1-hexen-3-ol	intense green with bitter and fatty notes	green
hexanol	alcoholic, ethereal, medicinal	alcoholic
3-octanol	oily, nutty, melon- and citrus-note	oily
<i>trans</i> -sabinene hydrate	spicy, warm, weak woody	warm
$\alpha$ -copaene	woody, spicy	spicy
linalool	clean-floral, citrus-lemon-orange notes	floral
$\beta$ -bourbonene	vetiver-notes, fresh-green	vetiver
linalyl acetate	floral, fresh, lavender-like	floral
$\beta$ -elemene	weak woody, green-herbal	woody
citronellyl formate	rose-like, fruity	floral
<i>trans</i> -dihydrocarvone	spearmint, herbaceous	spearmint
terpinen-4-ol	spicy, woody, nutmeg- and lilac-notes	terpenic
<i>cis</i> -dihydrocarvone	spearmint, herbaceous, minty	minty
$\beta$ -caryophyllene	terpene-odor, woody, spicy	terpenic
menthol	minty, fresh	minty
$\alpha$ -terpineol	lilac odor, floral, fruity	fruity
$\alpha$ -humulene	weak woody	woody
pulegone	minty, herbaceous, camphor-like	minty
<i>trans</i> - $\beta$ -farnesene	mild, sweet, warm	warm
dihydrocarvyl acetate	minty, camphoraceous, mild medicinal	minty
borneol	herbaceous, sweet	herbal
germacrene D	woody, spicy	spicy
<i>trans</i> -dihydrocarveol	woody-floral, sweet, minty	sweet
bicyclogermacrene	herbaceous, woody	herbal

Table III. Continued

Compound	Aroma impressions	GCST <sup>a</sup>
carvone	spearmint and minty (-), caraway (+)	spearmint
<i>cis</i> -dihydrocarveol	woody-floral, sweet-animalic, minty	woody
<i>trans</i> -carvyl acetate	green, spearmint, fresh	fresh
<i>trans</i> -carveol	spearmint, caraway	spearmint
<i>cis</i> -carvyl acetate	spearmint, fresh, green	fresh
germacrene A	woody, spicy, herbaceous	spicy
<i>cis</i> -carveol	spearmint, caraway	spearmint
geraniol	rose-like, sweet-floral, fruity, mild	rose
<i>cis</i> -jasmone	floral, jasmine-like, fruity, warm, spicy	floral

<sup>a</sup>odor impressions given by professional perfumers by means of GC-sniffing technique analyses of the essential *Mentha spicata* oil

## Experimental

**Sample preparation:** *M. spicata* plants were collected in the area of Ngaoundere (Cameroon), the species identified by a local botanist (S. Yonkeu, University of Ngaoundere) and a voucher specimen (No. 25745 SRFK/CM) deposited at the National Herbarium of Yaounde (Cameroon). About 0.2 kg of fresh leaves were harvested during March 1999 early in the morning (before sunrise); the essential oil was

obtained by steam-distillation for 4 h using a Clevenger apparatus. The oil was separated from the water phase by extraction with dichloromethane (100 mL) and the solvent eliminated by evaporation. A yield of 0.1 percent was obtained.

**Olfactive evaluations:** The essential spearmint oil was olfactively evaluated by perfumers from Dragoco Co., Vienna, Austria; a characteristic *M. spicata* aroma was

identified, with an intense, pleasant fresh carvone-limonene-1,8-cineole top-note and with green (leaf-like), floral (direction of rose and lavender), fruity (citrus- and peach-like) and spicy (direction of pepper and caraway) side-notes.

**Solid phase micro-extraction sampling:** The essential *M. spicata* oil was stored in a dark-brown 5 ml flask, and extracted with a 2 cm-50/30  $\mu$ m DVB/Carboxen/PDMS/StableFlex coated glass fiber (Supelco, USA, Cat-No. 5-7348) for 30 min at room temperature. Immediately afterwards the trapped volatiles on the fiber were directly analyzed by GC and GC/MS.

**GC:** GC analyses were carried out using a GC-14A with SPME sleeve adapted to injector, FID and C-R6A-Chromatopac integrator (Shimadzu, Japan), a GC-3700 with FID (Varian, Germany) and C-R1B-Chromatopac integrator (Shimadzu). The carrier gas was hydrogen; injector temperature, 250°C; detector temperature, 320°C. The temperature program was: 40°C/5 min to 280°C/5 min, with a heating rate of 6°C/min. The columns were 30 m x 0.32 mm bonded FSOT-RSL-200 fused silica, with a film thickness of 0.25  $\mu$ m (Biorad, Germany) and 30 m x 0.32 mm bonded Stabilwax, with a film thickness of 0.50  $\mu$ m (Restek, USA) as well as for chiral separations 25 m x 0.25 mm FS-HYDRODEX- $\beta$ -PM fused silica (film thickness: 0.25  $\mu$ m, Restek, USA). Quantification was achieved using peak area calculations, and compound identification was partly carried out using correlations between retention times.<sup>7,9,20,31</sup>

**GC/MS:** For GC/MS measurements a GC-17A with QP5000 (Shimadzu), SPME sleeve adapted to injector and Compag-ProLinea data system (class 5k-software), a GC-HP5890 with HP5970-MSD (Hewlett-Packard, USA) and ChemStation software on a Pentium PC (Böhm, Austria), a GCQ (Finnigan-Spectronex, Germany-USA) and Gateway-2000-PS75 data system (Siemens-Nixdorf, Germany, GCQ-software) were used. The carrier gas was helium; injector temperature, 250°C; interface-heating at 300°C, ion-source-heating at 200°C, EI-mode was 70 eV, and the scan-range was 41-450 amu. For other parameters, see description of GC, above. Mass spectra correlations were done using Wiley, NBS, NIST and our own library as well as published data.<sup>9,19</sup>

## Acknowledgements

We acknowledge the olfactory evaluations by W. Höppner and V. Hausmann, chief-perfumers of Dragoco Co., Vienna, Austria and the grants for M.S. and M.B.N. from the University of Vienna.

Address correspondence to Leopold Jirovetz, Institute of Pharmaceutical Chemistry, University of Vienna, Althanstrasse 14, A-1090 Vienna, Austria.



## References

- Arctander, St. 1969 *Perfume and Flavor Chemicals*, Published by the editor, Montclair.
- Aggarwal, K.K., Khanuja, S.P.S., Ahmad, A., Kumar, T.R.S., Gupta, V.K., Kumar, S. 2002 *Antimicrobial activity profiles of the two enantiomers of limonene and carvone isolated from the oils of Mentha spicata and Anethum soua* Flav.Fragr.J. 17: 59-63.
- BACIS-Boelens *Aroma Chemical Information Service* 1999 *Volatile Compounds in Food Database*, TNO Nutrition and Food Research, Zeist, The Netherlands.
- Baser, K.H.C., Kürcüoğlu, M., Tarımcılar, G., Kaynak, G. 1999 *Essential Oils of Mentha Species from Northern Turkey* J.Essent.Oil Res. 11: 579-588.
- Bauer, K., Garbe, D. 1985 *Common Fragrance and Flavor Materials*, VCH Verlagsgesellschaft mbH, Weinheim.
- Chao, S.C., Young, D.G., Oberg, C.J. 2000 *Screening for Inhibitory Activity of Essential Oils on Selected Bacteria, Fungi and Viruses* J.Essent.Oil Res. 12: 639-649.
- Davis, N.W. 1990 *Gas chromatographic retention indices of monoterpenes and sesquiterpenes on methyl silicone and Carbowax 20M phases* J.Chromatogr. 503: 1-24.
- Günter, M., Krammer, G., Lambrecht, St., Sommer, H., Surburg, H., Werkhoff, P. 2001 *Flavor Chemistry of Peppermint Oil (Mentha piperita L.)* ACS Symposium Series 794 (Aroma Active Compounds in Foods), Chapter 10, American Chemical Society.
- Jennings, W., Shibamoto, T. 1980 *Qualitative Analysis of Flavour and Fragrance Volatiles by Glass Capillary Gas Chromatography*. Academic Press, New York.
- Jirovetz, L., Buchbauer, G., Nikiforov, A. 1994 *Vergleichende Inhaltsstoffanalyse verschiedener Dillkraut- und Dillsamenöle mittels GC/FID und GC/MS* Ernährung/Nutrition 18: 534-536.
- Jirovetz, L., Buchbauer, G., Ngassoum, M.B. 1999 *Analysis of the aroma compounds of "Baton", the fermented root-crop Cassava from Cameroon, using SPME* Ernährung/Nutrition 23: 461-464.
- Jirovetz, L., Buchbauer, G., Ngassoum, M.B. 2000 *Analysis of the aroma compounds of the pulp of Borassus aethiopum L. (Palmaceae) from Cameroon using GC-MS, SPME-GC-MS and Olfactometry* Ernährung/Nutrition 24: 159-161.
- Jirovetz, L., Buchbauer, G., Stoyanova, A., Balinova, A. 2001 *Analysis, Chemotype and Quality Control of the Essential Oil of a New Cultivated Basil (Ocimum basilicum L.) Plant from Bulgaria* Sci.Pharm. 69: 85-89.
- Jirovetz, L., Buchbauer, G., Ngassoum, M.B. 2001 *Analysis of the Aroma Compounds of Two Different Palm Wine Species ("Matango" and "Raffia") from Cameroon Using SPME-GC-FID, SPME-GC-MS and Olfactometry* Ernährung/Nutrition 25: 67-71.
- Jirovetz, L., Buchbauer, G., Ngassoum, M.B. 2001 *Analysis of the Aroma Compounds of the Seeds of the Cameroonian "Garlic Tree" Scorodophloeus zenkeri Harms. Using GC-MS, SPME-GC-MS and Olfactometry* Ernährung/Nutrition 25: 354-356.
- Jirovetz, L., Leitner, E., Buchbauer, G. 2001 *Applications of solid phase micro extraction [SPME] in aroma compound analysis of agriculturare and food products* Rec.Res.Develop.Agric.Food Chem., Vol. 5, 143-166.
- Jirovetz, L., Buchbauer, G., Ngassoum, M.B., Geissler, M. 2002 *Analysis of the headspace aroma compounds of the seeds of the Cameroonian "garlic plant" Hua gabonii using SPME/GC/FID, SPME/GC/MS and olfactometry* Eur.Food Res.Technol. 214: 212-215.
- Jirovetz, L., Smith, D., Buchbauer, G. 2002 *Aroma Compound Analysis of Eruca sativa (Brassicaceae) SPME Headspace Leaf Samples Using GC, GC-MS and Olfactometry* J.Agric.Food Chem. 50: 4643-4646.
- Joulain, D., König, W.A. 1998 *The Atlas of Spectral Data of Sesquiterpene Hydrocarbons*, E.B.-Verlag, Hamburg.
- Kondioya, N. and Berdaque, J.-L. 1996 *A Compilation of Relative Retention Indices for the Analysis of Aromatic Compounds*, Edition du Laboratoire Flaveur, Saint Genes Champelle.
- Maffei, M., Codignola, A., Fieschi, M. 1986 *Essential Oil from Mentha spicata L. (Spearment) Cultivated in Italy* Flav.Fragr.J. 1: 105-109.
- Maffei, M. 1990 *F1 and F2 Hybrids from Mentha x verticillata 7303 x Mentha spicata L.A. Chemogenetic Study* Flav.Fragr.J. 5: 211-217.
- Ngassoum, M.B., Jirovetz, L., Buchbauer, G. 2001 *SPME/GC/MS analysis of headspace aroma compounds of the Cameroonian fruit Tetrapleura tetraptera (Thonn.) Taub.* Eur.Food Res. Technol. 213: 18-21.
- Ohloff, G. 1994 *Scent and Fragrances*, Springer-Verlag, Berlin.
- Pino, J.A., Garcia, J., Martinez, M.A. 1999 *Comparison of Solvent Extract and Supercritical Carbon Dioxide Extract of Spearment Leaf* J.Essent.Oil Res. 11: 191-193.
- Pino, J., Borges, P., Martinez, M., Vargas, M., Flores, H., Estarrón, M. 2001 *Essential Oil of Mentha spicata from Jalisco* J.Essent.Oil Res. 13: 409-410.
- Ravid, U., Bassat, M., Putievsky, E., Weinstein, V., Ikan, R. 1987 *Isolation and Determination of Optically Pure Carvone Enantiomers from Caraway (Carum carvi L.), Dill (Anethum graveolens L.), Spearmint (Mentha spicata L.) and Mentha longifolia (L.)* Huds. Flav.Fragr.J. 2: 95-97.
- Ravid, U., Putievsky, E., Katzir, I., Weinstein, V., Ikan, R. 1992 *Chiral GC Analysis of (S)(+)- and (R)(-)-Carvone with High Enantiomeric Purity in Caraway, Dill and Spearmint Oils* Flav.Fragr.J. 7: 289-292.
- Schulz, H., Drews, H.-H., Krüger, H. 1999 *Rapid NIRS Determination of Quality Parameters in Leaves and Isolated Essential Oils of Mentha Species* J.Essent.Oil Res. 11:185-199.
- Sigma-Aldrich 1998 *Solid Phase Microextraction: Solventless Sample Preparation for Monitoring Flavor Compounds by Capillary Gas Chromatography* Supelco Bulletin 869A, Bellefont.
- Sigma-Aldrich 2002 *Flavors & Fragrances – The Essence of Excellence*, Milwaukee.
- Tudor, E. 1997 *Temperature dependence of the retention index for perfumery compounds on a SE-30 glass capillary column, I. Linear equations* J.Chromatogr. A 779: 287-297. ■