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Multidimensional gas chromatography using a double cool-strand interface

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Abstract

Commercial interfaces for multidimensional gas chromatography (MDGC) are based either on a valve or a pneumatic switching between columns. Both exhibit significant drawbacks and only few suppliers exist. An extremely simple interface has been set up to overcome these limitations without requiring any pneumatic control or valves switching. This new MDGC design is based on the cryo-control of the analyte transfer from the first to the second column through two cool strands of a capillary. This technique is simple to implement and does not require any special column connections. Applications involve non-polar/polar phase combinations, as well as chiral analysis, hyphenation to a conventional mass spectrometer, and olfactometric detection. In contrast to conventional MDGC configuration, the present configuration allows the use of a single oven to operate both columns at different temperatures.

Keywords: Multidimensional gas chromatography; Interface; Chiral analysis; GC–olfactometry; GC/MS

1. Introduction

Multidimensional gas chromatography (MDGC) is a well-known technique that has been developed early after the invention of gas chromatography. The controlled transfer of analytes from the first to the second column is a critical issue. Most of the techniques achieve an on-line "heart-cut", i.e. only some peaks eluted from the first column are transferred to the second one, while others are vented [1]. Alternative off-line techniques such as the intermediate trapping of analytes in a sorbent and their subsequent desorption in the second column require much more time and sophisticated hardware and are not comparable with the present method [2,3].

The "heart-cut" can be achieved according two means: with a valve, or with a pneumatic switcher. A valve is the simplest interface as no pressure or flow control is required when two columns of similar diameters are used [4,5]. However, it exhibits serious drawbacks in regard to the analyte stability and adsorption [2]. Especially, a contact of labile compounds (e.g. sulfur derivatives) with a metal such as stainless steel promotes their degradation [6]. Other compounds (e.g. carboxylic acids, amines) are prone to adsorption on the stainless steel surface of valves. Such phenomena cause memory effects that are prejudicial to the analysis [7,8]. Moreover, because of its thermal inertia, the valve mass can also create a "cold spot" in the chromatographic oven when using a temperature program. To avoid altering the chromatogram quality, an independent heating of the valve has been proposed [2]. In addition, the internal dead volume of the valve may participate in the peak broadening when used with narrow bore capillary columns.

Pneumatic switching devices have been known for a long time. In 1968, D.R. Deans proposed an interface based on a pressure equilibrium, which avoids any contact of analytes with a valve [9,10]. The pneumatic switcher was miniaturized by G. Schomburg and called the "live-switching device" [11,12]. Siemens commercialized this technology ("Sichromat" gas chromatographs) [7], and later Gerstel proposed a similar design [13,14]. Pneumatic switching requires an accurate flow control of the pressure between both columns. This must be achieved using a make-up gas and e.g. electronic mass flow controllers. Such a sophistication makes the optimization of analytical parameters more complicated

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and increases the instrument cost [13]. Moreover, pneumatic switching may cause some peak broadening. The use of a cold trap has been proposed to re-focus the heart-cut peak [15]. Then an additional means to quickly heat and re-inject the trapped peak is required, which increases again the complexity of the system.

An alternative technique was proposed by Marriott et al. in 2000 [16]. This technique, called Targeted Mode GC, employed a longitudinally modulated cryogenic system (LMCS) usually used in comprehensive GC × GC to control the peak transfer between both dimensions. It allowed to sequentially block and release different parts of the chromatogram in a succession of "pulsing events". This elegant technique is easy to implement, and significant peak enhancement was reported. However, several limitations exists. As specified by the authors, a rapid analysis must be achieved in the second column, since it ensures that successive pulsing events do not result in overlapping elution zones and alter the peak resolution. This limits the choice of stationary phase as, for instance, no fast-GC chiral column is currently available. Moreover, due to the very rapid elution in the 2nd dimension, detectors with fast acquisition rate (>50 Hz), had to be used. Another limitation concerns the maximum trapping time of a target peak. To avoid recombination of the target peak with interfering peaks, the target has to be released from the trapping zone before the following peaks arrive. This limits the control of the target peak trapping length. Times of modulator event were defined according to a first GC run, after having corrected these time events to take into account the detection delay due to the elution (a few seconds) in the 2nd dimension.

Therefore, the present work aims at improving the technology of interfaces dedicated to MDGC with respect to the following criteria:

- Analytes only in contact with inert materials, i.e. no transfer through a valve.
- Simple or no control of the flow/pressure within the interface.
- No dead volume.
- Compatibility with usual capillary columns (0.15–0.53 mm), as well as with fast GC columns (0.1 mm or less).
- Simple remote control of the interface by the GC workstation.
- Low cost.

2. Experimental

2.1. Materials

Hyacinth oil was obtained from Quest International (Naarden, The Netherlands), jasmine oil from Danisco (Zug, Switzerland) and lavender oil from Firmenich SA (Geneva, Switzerland). Bergamot oil was a commercial test sample. All pure compounds were analytical grade (purity > 97%) except for α-isomethylionone (>95%). α-Ionone, β-ionone, (*R*/*S*)-linalool and (*R*)-linalool were purchased from Fluka (Buchs, Switzerland), α-isomethylionone from Bedoukian (Danbury, USA), tetrahydrolinalool was purchased from BASF AG (Frankfurt, Germany) and Zestover (2,4-dimethyl-3-cyclohexene-1-carbaldehyde) from Givaudan SA (Geneva, Switzerland). Nonanal, terpinolene and phenylethanol came from Firmenich SA. Compounds and mixtures were diluted 1:20 (v/v) using ethyl acetate (analytical grade, SDS, Peypin, France) prior to injection.

2.2. Gas chromatography

MDGC analyses were performed using a 6890N gas chromatograph (Agilent Technologies, Wilmington, DE, USA) equipped with two flame ionization detectors (FID, Agilent Technologies) and a longitudinally modulated cryogenic system (LMCS, Everest model unit, Chromatography Concepts, Doncaster, Australia). Two columns with different retention affinities were serially connected via a deactivated silica capillary (0.5 m × 0.25 mm i.d., Supelco, Buchs, Switzerland) passing through the LMCS cryo-trap (vide infra). Helium purified with filters for water, oxygen and hydrocarbons was used as carrier gas and delivered at constant pressure. Samples (1 μl) were injected with a 1/50 split. Chromatographic configurations and conditions are summarized in Table 1. All instrumental parameters and data acquisition were controlled via the Galaxie Chromatography Data System software (Varian-JMBS, Fontaine, France).

2.3. Double cool-strand interface

The capillary connecting the two columns was wound to form a loop, whose strands were passed trough the cryotrap of the LMCS (Fig. 1). The flow exiting the first column was splitted in two parts owing to a deactivated fused silica "Y" connector. One part was hyphenated to the capillary loop whereas the second one was connected to the first flame ionization detector. The loop outlet was connected to the second column inlet via a glass-coated mini-union capillary connector (SGE, Courtaboeuf, France). The second-column outlet was directly connected to the second FID. When required, this second FID was connected in parallel with an MS or a sniff port (vide infra). The LMCS was monitored by the external events of the workstation, as a function of the retention time. The cryotrap was cooled by liquid carbon dioxide, which allowed the efficient trapping of compounds heavier than heptane [17]. For more volatile analytes, liquid nitrogen should be used.

2.4. Mass spectrometry

For mass spectrometric detection, the second column outlet flow was splitted between the second FID and an Ion

Table 1
Analytical conditions used for the different examples

Product	1st column/2nd column	Detectors after 1st column/2nd column	Inlet temperature/pressure	Oven temperature program
Ionones Jasmine and hyacinth essential oils	SPB1, 30 m × 0.25 mm × 1.0 μm, Supelco/DB-WAX 30 m × 0.25 mm × 0.25 μm, J&W Scientific	FID[a]/FID[a]	250 °C/280 kPa	5 min at 40 °C then 5 °C/min to 220 °C 5 min
Enantiomers of linalool in bergamot oil	SPB1, 30 m × 0.25 mm × 1.0 μm, Supelco/Megadex DMPβ 10 m × 0.25 mm × 0.25 μm, Mega	FID[a]/FID[a]	250 °C/280 kPa	90 min at 85 °C then 10 °C/min to l50 °C
Chiral resolution of linalool and linalyl acetate in lavander oil	CP-Sil5CB30 m × 0.32 mm × 1.0 μm, Chrompack/Megadex DETTBSβ 25 m × 0.25 mm × 0.25 μm, Mega	FID[a]/FID[a]	250 °C/280 kPa	2 min at 50 °C then 8 °C/min to 165 °C 1 min then 25 °C/min to 120 °C 8 min then 25 °C/min to 50 °C 1 min then 2 °C/min to 150 °C 1 min
Fragrance model mixture	SPB1, 30 m × 0.25 mm × 1.0 μm, Supelco/DB-WAX 30 m × 0.25 mm × 0.25 μm, J&W Scientific	FID[a]/FID[a] + ion trap detector[b] and FID[a]/FID[a] + sniffing port[c]	250 °C/280 kPa	5 min at 40 °C then 5 °C/min to 220 °C 5 min

[a] Supplied by Agilent Technologies.
[b] Supplied by Finnigan Mat.
[c] Home made.

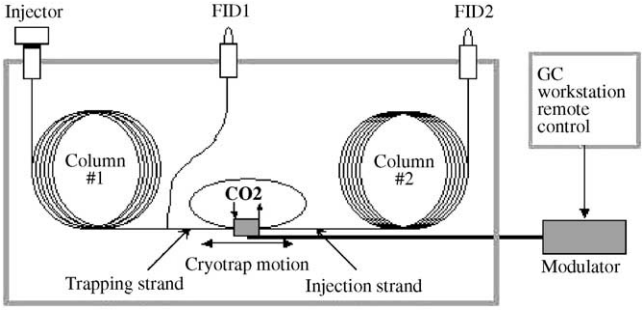

Fig. 1. Scheme of the MDGC with the double cool-strand interface.

Trap Detector (ITD 800, Finnigan MAT, San Jose, USA), owing to a Gerstel cross [18]. The ITD transfer line (deactivated capillary, 0.45 m × 0.25 mm) was heated to 250 °C (flow rate = 1 mL min^{-1}). A make-up of helium (1 mL min^{-1}) was added via this cross. The mass spectra were acquired under a 70 eV ionization potential. The range of masses acquired was $33 < m/z < 320$ atomic mass units (amu). Compounds were identified by comparison of experimental data with those of Firmenich's data bank and by comparison of their retention time with those of authentic samples.

2.5. Sniffing procedure

For the olfactometric detection, the second column outlet flow was splitted into two parts via a fused silica "Y" connector and directed to the second FID and to a thermostated transfer line (1 m × 0.25 mm, Supelco) maintained at 200 °C. The line was ended by a Dewar-type sniffing port (silver-coated double jacketed glass port under vacuum) to isolate the panelist's nose from the hot parts of the transfer line [19]. A panel of seven assessors was selected (four men and three women aged from 25 to 55 years). All of them regularly participated in sniffing sessions. During the 10 min session, they were asked to freely describe the aroma eluted from the second column. The start- and end-times of olfactive peaks and odor descriptions were noted. The seven individual aromagrams were averaged according to the detection frequency method [20].

2.6. Data processing

Height, half-height width and resolution of peaks were calculated by the Galaxie Chromatography Data System software (Varian-JMBS). The resolution between two successive peaks was calculated according to the following formula:

$$R_s = 1.18 \times \frac{Rt_2 - Rt_1}{\omega_2 + \omega_1}$$

Rt_1 and Rt_2 represent the first and second peak retention time, respectively; ω_1 and ω_2 are the half height widths of the first and the second peaks, respectively.

3. Results and discussion

3.1. Principle

The role of all MDGC interfaces described in the introduction consists in transferring some selected peaks from the first to the second column. Other peaks, which would have overlapped with target ones, are vented. In fact, controlling the transfer rate of peaks through the interface would be sufficient to achieve the same objective, i.e. eluting the target peaks in the second column, in a retention zone free of any interference. In other terms, the target peak(s) must be retained in the interface until preceding ones elute from the second column. Then, the target peak(s) can be transferred into the second column while the following ones are still retained for a while in the interface.

Achieving such a rate-controlled transfer requires two simultaneous trapping zones in the transfer capillary between both columns, and thus avoids mixing the target peak with following ones when maintaining the target in the interface for a long time. To simplify the system, these two zones may be controlled by the same cryo-trap through which the transfer capillary passes twice (Fig. 1). The cryogenic control of these two loop strands is performed with a commercially available cryogenic trap (LMCS [17]). The latter allows to practically stop the elution of analytes in the cold zones. Then, when moving the trap, the cold zones are re-heated by the GC-oven and analytes are re-injected as very narrow peaks. The trap motion is remote-controlled by the GC workstation, according to the following sequence:

(a) Waiting position (Fig. 2.1): peaks preceding the target peaks are trapped in the trapping strand.
(b) Trapping of target peaks (Fig. 2.2): the target zone is stopped in the trapping strand while the preceding peaks are transferred in the injection strand.
(c) Back to the waiting position (Fig. 2.3): target peaks are still stopped in the trapping strand. Preceding peaks are ready to be injected in the second column.
(d) Pre-injection (Fig. 2.4): the target zone is transferred from the trapping strand to the injection strand. Preceding peaks are injected in the second column. Following peaks are blocked in the trapping strand.
(e) Back to the waiting position (Fig. 2.5): target peaks are ready to be injected. Following peaks are still blocked in the trapping strand.
(f) Injection (Fig. 2.6): the target zone is injected into the second column. Following peaks are transferred from the trapping strand to the injection strand. They are retained in the injection strand.

Time delays between those steps depends on the retention time of the targeted peaks, on their duration and on the presence of interfering peaks before and after the target zone. Delays are adjustable by the analyst, as a function of the retention time, owing to the external events of the GC workstation.

This new interface does not require any flow or pressure control and is compatible with usual capillary columns as well as with fast GC columns. Owing to zero-dead volume connecting parts used (fused capillary connector), analytes are never in contact with potentially reactive materials such as metals.

3.2. Hyphenation of a non-polar column to a polar column

α-Ionone and β-ionone are often used in a same perfumery formula, such as violet-like fragrances, but they sometimes contain traces of α-isomethylionone, a suspected allergen. The separation of α-isomethylionone, α-ionone and β-ionone by either a single non-polar column (PDMS-type), or a single polar column (carbowax-type) typically exemplifies the limitation of monodimensional-GC. α-Isomethylionone coelutes with β- or α-ionone on non-polar column, and on polar column, respectively. Since all three compounds are structurally related, abundant MS ions for α-isomethylionone are not strictly characteristic of this compound to allow a de-

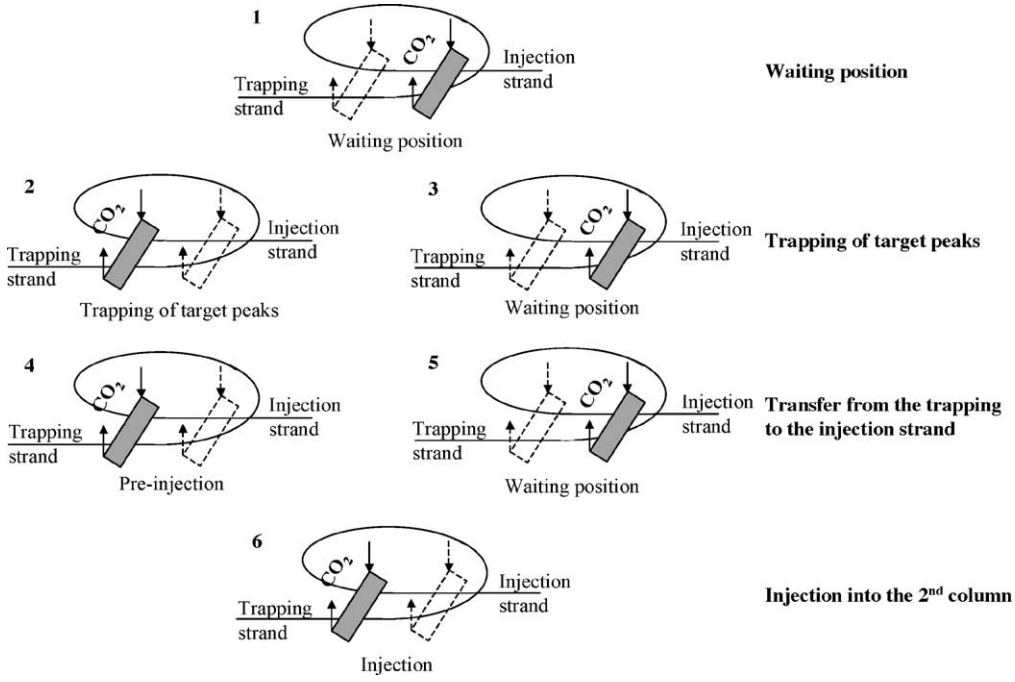

Fig. 2. Working principle of the double cool-strand interface (see explanations (a–f) under "Section 3.1". Grey rectangle corresponds to the position of the cold cryotrap.

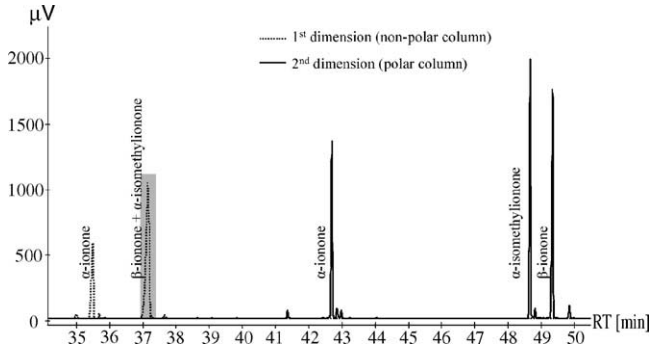

Fig. 3. MDGC separation of ionones (SPBl + DB-WAX with FID as 1st and 2nd detector). The grey tint zone corresponds to the transfer of the corresponding peak into the second column owing to the double cool-strand interface.

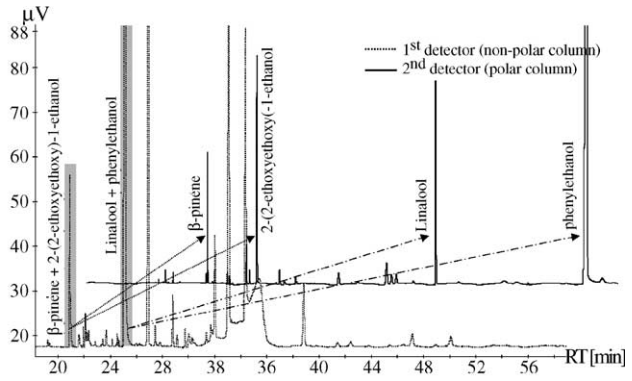

Fig. 4. MDGC analysis of a blend of jasmine and hyacinth essential oils (SPBl + DB-WAX with FID as 1st and 2nd detectors). The grey tint zones correspond to the cryotrapping zones.

convolution. The MDGC however was able to resolve the target component from both peaks that were observed to interfere with a single-column analysis. A commercial blend of ionones was injected into the MDGC equipped with a non-polar first column coupled to a polar second column (Table 1). After the first column, α-isomethylionone was separated from α-ionone, but co-eluted with β-ionone (dotted line, Fig. 3). After a controlled transfer of the α-isomethylionone plus β-ionone peak owing to the double cool-strand interface, α-isomethylionone was clearly separated from α- and β-ionone (solid line, Fig. 3). Table 2 summarizes the different trapping events. The peak height increase observed in Fig. 3 will be discussed in Section 3.7.

Usual MDGC instruments, based on a valve or a pneumatic switching, allow to perform several heart-cuts during a single run, to further separate in the second column several groups of peaks co-eluted in the first column. The following example demonstrates that such a multiple transfer is also feasible with a double cool-strand interface.

A blend of jasmine and hyacinth essential oils was injected in the same chromatographic conditions as for the ionones (Table 1). This blend exhibited co-elutions and overlaps using a non-polar column (β-pinene and 2-(2-ethoxyethoxy)-l-ethanol at 20.91 min, linalool and phenylethanol at 25.19 min, see Fig. 4). Two trapping/release sequences were achieved in the same run, according to Table 3. These four peaks were eluted in a free zone of the second column, with an excellent resolution.

Table 2
Analysis of ionones[a]

Position of the cryotrap	Time (min)
Waiting position (see Fig. 2.1)	36.00
Trapping of target peaks (see Fig. 2.2)	36.80
Waiting position (see Fig. 2.3)	37.20
Pre-injection (see Fig. 2.4)	38.00
Waiting position (see Fig. 2.5)	38.50
Injection (see Fig. 2.6)	44.00

[a] Cryotrapping events.

In such case, the interface ability to trap separately two closely eluted peaks is only limited by the rapidity of the peak transfer from the trapping to the injection strand. This depends principally on the analyte velocity in the loop, which is approximately the same as the velocity of the carrier gas. In the previous experiment for instance, the average velocity of the carrier gas was 30 cm s^{-1}, meaning that the 25 cm of the capillary between both cool strands were crossed in 830 ms. The cryogenic trap motion and the column re-heating time were negligible compared to this step, because the cryogenic trap can move away from the cooled zone in less than 10 ms, while the cooled column heats up to the prevailing oven temperature within 10–15 ms [17]. Even if these observations correspond to a single strand passing through the cryotrap, the order of magnitude of these times were assumed to remain identical for a double strand in the cryotrap. Experimentally, we observed that the retention time difference of two target peaks must exceed 1 s to allow their separate trapping.

3.3. Chiral resolution in a complex mixture

Natural products often contain chiral compounds whose enantiomeric purity depends on their origin. Determining the enantiomeric ratios is a useful way to characterise sample origin and a possible adulteration. Because of the relative complexity of natural oils, chiral compounds often require to be isolated from the others via a first non-chiral column, prior to their resolution in the chiral column of a MDGC system. A bergamot essential oil was injected in the MDGC with a non-polar column as first dimension connected to a chiral column as the second dimension (2,6-di-*O*-methyl-β-cyclodextrin, Table 1). The linalool re-injection in a peak-free zone of the second column allowed a baseline resolution ($\alpha = 1.94$, Fig. 5), and an accurate determination of enantiomeric proportions (15/100, *S*/*R*). These values are far from the expected proportions in natural bergamot (0.5/100, *S*/*R*) [21]. This suggests an adulteration with some racemate addition.

Table 3
Analysis of a blend of jasmine and hyacinth essential oils

Time (min)	1st strand	2nd strand
19.00	Retention of peaks preceding the 1st target zone	
20.75–21.70	Retention of the 1st target zone (β-pinene and 2-(2-ethoxyethoxy)-l-ethanol) then transfer into the second strand	Retention of peaks eluting before the 1st target zone then injection into the second column
21.70–24.75	Retention of peaks eluting between both target zones then transfer into the second strand	Retention of β-pinene and 2-(2-ethoxyethoxy)-l-ethanol then injection into the second column
24.75–26.20	Retention of the 2nd target zone (linalool and phenylethanol) then transfer into the second strand	Retention of peaks eluting between both target zones then injection into the second column
26.20–40.00	Retention of peaks following the 2nd target zone then transfer into the second strand	Retention of linalool and phenylethanol then injection into the second column

Table of time-events controlling the transfer of analytes in the two cool strands.

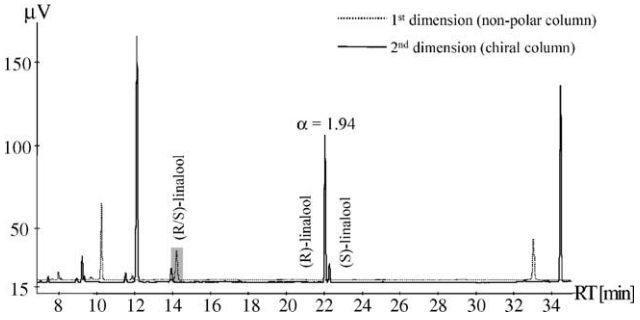

Fig. 5. MDGC-chiral analysis of linalool in adulterated bergamot oil (SPBl + DMPβ with FID as 1st and 2nd detectors). The grey tint zone corresponds to the cryotrapping zone.

3.4. Use of a single oven to operate both columns at different temperatures

Sometimes the elution temperature of a given compound from the first dimension is too high for its resolution in the second dimension. This is especially the case with a chiral second dimension in which the enantiomer resolution is very temperature-dependent [22]. This situation requires two separate ovens to operate both columns at different temperature. Using the apparatus of the invention, due to the controlled trapping of the target compound in the interface, the oven can be rapidly cooled down to the initial temperature required by the second column before re-injecting the analytes in the second column. To illustrate this capability, the two main chiral constituents of lavender oil, linalool and linalyl acetate, were resolved within the same analysis. The controlled cryotrapping of these compounds allowed the adaptation of the chromatographic conditions (cooling of the oven before re-injection in the second dimension) to achieve the best resolution of those racemates. Both compounds were resolved independently from each other during the same analysis: linalool was resolved using isothermal conditions whereas linalyl acetate has been resolved under slow temperature ramp (Table 1 and Fig. 6A). The enantiomeric ratios for both compounds suggest that this lavender oil was an authentic natural essential oil (Fig. 6B).

3.5. Hyphenation to slow detectors: MDGC/MS

During the last decade, the development of comprehensive two-dimensional gas chromatography (GC × GC) has become a major improvement in the analytical field. It achieves

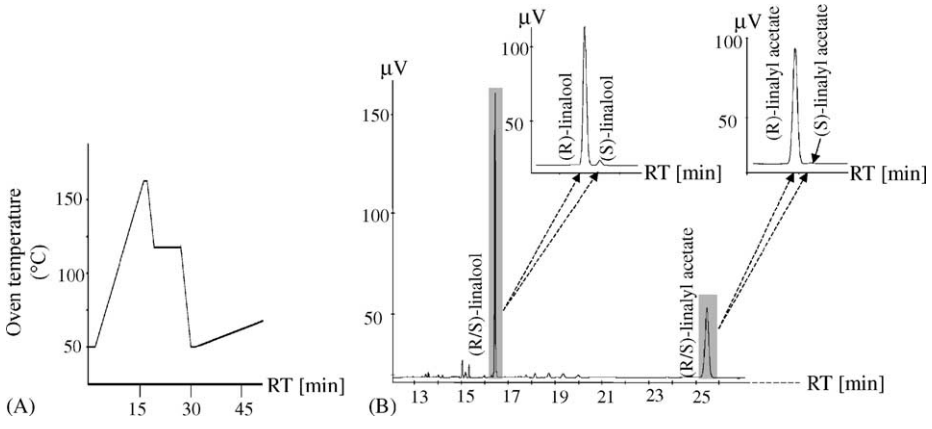

Fig. 6. MDGC-chiral analysis of linalool in natural lavender oil (CP-Sil5CB + DETTBSβ with FID as 1st and 2nd detectors). Both columns have been operated at different temperatures with a single oven. (A) Temperature program of the oven. (B) Chromatogram. The grey tint zones correspond to cryotrapping zones. The enantiomeric ratios were 4.3/100 ((*R*/*S*)-linalool, expected values = 4.2/100) and 0.8/100 (*S*/*R* linalyl acetate, expected values = 0.8/100).

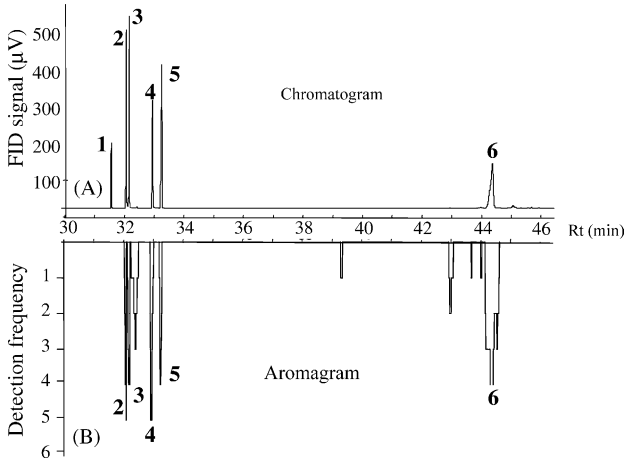

Fig. 7. MDGC analysis of the fragrance model (SPB1 + DB-WAX with an FID (A) and an olfactometric detector (B) as 2nd detector).

a true continuous multidimensional (dual column) gas chromatography over the whole chromatographic run, without venting any peak [17]. A major difference exists between this work and comprehensive GC × GC. In this latter, the carrier gas velocity is normally much higher in the second column than in the first one, because peaks pulsated by the modulator in the second dimension must elute within the time interval between two modulations [23]. In the case of MDGC, the analyst only focuses on a few peaks, which does not require a rapid elution in the second dimension. As the carrier gas velocity is the same as in a usual capillary column, acquisition detectors with a slow sampling rate can be used (e.g. quadrupole or ion-trap MS, human nose, etc.).

An ion trap mass spectrometer was coupled to the MDGC system, after the second column. To test this configuration, a fragrance model mixture containing six compounds (terpinolene, nonanal, tetrahydrolinalool, Zestover, linalool and phenylethanol) eluting at the same time on the first dimension was injected in the MDGC–MS system equipped with the double cool-strand interface. Co-eluted compounds (20.90–21.50 min) were cryotrapped and re-injected in the second column in a peak-free zone. All constituents were fully resolved (Fig. 7), and unambiguously identified by the ion-trap MS (Table 4).

Table 4
Comparison of the MS identification quality of the constituents of a fragrance model mixture injected in a MDGC, with and without cryofocusing

	Match quality without cryofocusing	Match quality with cryofocusing
Terpinolene	97	98
Zestover	Wrong identification	98
Nonanal	Wrong identification	98
Phenylethanol	46	96
Linalool	87	94
Tetrahydrolinalool	78	96

3.6. Hyphenation to slow detectors: MDGC/olfactometry

Another important and powerful slow-rate detector is the human nose. It is particularly employed in the field of flavor, fragrance and off-odors analysis. Eluting peaks have to be intense enough to be well perceived, and pure enough to allow panelist assigning them the appropriate descriptors. A sniffing port was connected at the end of the second column, instead of the mass spectrometer (chromatographic conditions are summarized in Table 1). The same model fragrance mixture was injected, and seven different assessors smelt the odorants eluting from the second column and described them using a free vocabulary. The seven individual aromagrams were averaged and the resulting profile was compared to the FID signal (Fig. 7). All peaks were baseline separated and were significantly detected. Their descriptors perfectly fitted those from the literature (Table 5). No panelist perceived terpinolene, which is consistent with its high detection threshold (1–2 order of magnitude higher than for the other compounds, according to personal data).

3.7. Efficiency and sensitivity improvement

Cryo-trapping analytes in a capillary is known to re-focus them. This improves the resolution and height of chromatographic peaks [15], which increases the sensitivity towards traces in complex mixtures. When using a valve or a Deans' interface without additional refocusing, the peak width of analytes entering the second column is at least the width of peaks leaving the first dimension. In practice, this width is often increased due to the inner dead volume of the interface. Therefore, shape and resolution of target peaks were compared under our configuration with and without cryofocusing (i.e. either the two column in series, without trapping, or with a trapping/re-injection sequence, see Table 6). The poor chiral resolution of linalool in the absence of cryofocusing did not allow the calculation of its areas, heights and half-height widths under these conditions. As illustrated by

Table 5
Comparison between literature descriptors [25] and the panelists' descriptors of eluting odorants of a fragrance model mixture

Peak	Compound	Literature descriptors	Panel descriptors
1	Terpinolene	Sweet-piney, oily, pleasant	Not perceived
2	Nonanal	Fatty-floral, waxy	Aldehydes, fatty
3	Tetrahydrolinalool	Floral, linalool, green	Linalool, floral
4	Zestover	Sweet-green, leafy	Green, grassy
5	Linalool	Fresh, floral-woody, faintly citrus	Linalool, floral
6	Phenylethanol	Rosy, floral, green	Rosy, phenylethanol

Table 6
Comparison of areas heights, half height widths and resolutions of peaks using MDGC without and with cryotrapping (plain and bold characters, respectively)

Compound	Area (μV min)	Height (μV)	Half height width	Resolution[a]
Ionones				
α-Ionone	55.2/**51.2**	568.8/**1348.2**	0.07/**0.03**	–/–
α-Isomethylionone	83.3/**79.1**	1003.4/**1967.9**	0.07/**0.04**	–/–
β-Ionone	88.6/**80.2**	926.6/**1740.7**	0.08/**0.04**	3.09/**10.13**
Resolution of linalool				
(*R*)-linalool	–/**9.4**	–/**122.1**	–/**0.07**	–/–
(*S*)-linalool	–/**4.1**	–/**50.7**	–/**0.09**	0.80/**1.38**
Fragrance model mixture				
Terpinolene	1.2/**0.9**	15.5/**56.3**	0.07/**0.01**	–/–
Nonanal	2.7/**2.5**	43.7/**111.9**	0.06/**0.02**	4.66/**19.92**
Tetrahydrolinalool	2.8/**2.6**	48.5/**109.9**	0.05/**0.02**	2.11/**4.73**
Zestover	2.4/**2.2**	40.8/**76.7**	0.05/**0.03**	5.13/**19.02**
Linalool	3.1/**2.9**	59.6/**84.4**	0.05/**0.03**	3.43/**10.34**
Phenylethanol	3.0/**2.9**	59.6/**42.8**	0.05/**0.06**	–/–

[a] Resolution between the previous and the target peak.

the identical peak areas with or without trapping, cryofocusing and reinjection did not alter the quantity of detected analytes. As a general observation, the heights and the resolution of targeted peaks were increased by a factor of 2–4, while the half-height width was reduced by a factor of 2–7, leading to an improved sensitivity for these compounds. As a consequence, this improvement in peak shape resulted in a better identification when using an MS detector (Table 4).

4. Conclusion

The simplicity of this new MDGC interface design makes it a very flexible analytical tool, as shown by the various configurations used in this study and in the related patent [24]. The technique is easy to implement and does not require any special equipment, except the commercially available cryogenic trap.

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