Enantiomer separation of α-campholene and fencholene derivatives by capillary gas chromatography on permethylated cyclodextrin phases
I [☆]. Compounds separable with single columns

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Abstract

α-Campholene and fencholene derivatives are compounds with interesting odour properties, e.g. sandalwood and woody notes. They have one (derived from the initial product, α-pinene) or several stereogenic centres. The stereoisomers of these compounds may have different odour properties; therefore, analysis methods are developed for the complete separation of the diastereomers and enantiomers. It is shown that the alcohols with one stereogenic centre as well as of the esters and ethers of α-campholene and fencholene derivatives with two stereogenic centres can be separated on permethylated α- und β-cyclodextrins dissolved in polysiloxanes. Factors influencing the separation are discussed.

1. Introduction

Apart from the characteristic functional groups, the typical odour note of a compound is determined especially by the molecular size and form [1]. Accordingly, different diastereomers as well as enantiomers may have different odour notes or even completely different types of odour [2–4].

One of the odorous substances which have been popular for more than 4000 years is the East Indian sandalwood oil. Originally, for the main substances α- and β-santalol, equal odour tones were given for all the diastereomers and enantiomers [5], but more recent studies deny this [6].

As the demand for sandalwood fragrances for a long time could not be covered by natural resources, synthetic sandal perfumes were introduced in 1940. The α-campholene [6] and fencholene compounds [7,8] found in Leipzig, which can be easily obtained from α-pineneoxide via α-campholene (1) or fencholene aldehyde (2), are suitable sandal and woody odourants. Also other substances which can be synthesized from 1 and 2, for example 3–28, have typical flowery, earthy, woody notes (e.g. 28) as well as sandal

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[☆] For Part II. see Ref. [31].

notes (e.g. **10** and **27**). All α-campholene and fencholene compounds have one stereogenic centre corresponding to the configuration of the C-5 atom in the starting α-pinene (Fig. 1); some have several stereogenic centres due to condensation in the side chain. For the exact determination of the odour notes, also in dependence on diastereomers and enantiomers, methods are necessary for the exact differentiation of stereomers. Because all the stereomer mixtures were volatile, analysis was carried out by means of gas chromatography on chiral stationary phases.

From structurally similar classes of substances, numerous studies on furanes [9,10] are known. Likewise, lactones were analyzed on permethylated [11,12] or on different substituted alkyl derivatives [13] as well as on alkyl/acyl derivatives of the cyclodextrins [14–17]. However, the results did not permit any conclusion that the α-campholene and fencholene derivatives are separable. The first results for this class of compounds were recently presented by us [18]. In order to include also intermediate synthetics, a considerable extension of these studies was necessary. Moreover, the investigations into the connection between structure and retention for α-campholene and fencholene compounds on chiral phases fell well into line with more comprehensive studies [19] carried out on this problem.

Part I deals with the analysis of substances with one stereogenic centre. Here, mainly alcohols, but partially also compounds with other functional groups (ester, ether) are described. In Part II [31], the analysis of α-campholene and fencholene derivatives with two and more stereogenic centres is described. Often, a complete separation of all stereoisomers of such mixtures is only possible after coupling of columns.

2. Experimental

2.1. Investigated substances

The initial compounds α-campholene (**1**) and fencholene aldehyde (**2**) are obtained from α-pinene via α-pinene oxide (**i**): the $ZnBr_2$-catalyzed camphane rearrangement of the α-pinene oxide (Fig. 1) supplies α-campholene aldehyde (**1**) [7,20].

Fencholene aldehyde (**2**) is formed by fenchane rearrangement of the trans-pinocarveol (**ii**), which can be obtained from α-pinene oxide (**i**) with aluminiumisopropylate, with subsequent hydrobromination and dehydrobromination [7,8]. By analogy, the 3-ethyl compounds (**7–11**) can be obtained from ethylapopinene [21]. The configuration of the used α-pinene determines the configuration of the C-1 atom in the five-membered ring of the campholene and fencholene compounds (see Fig. 1). The configura-
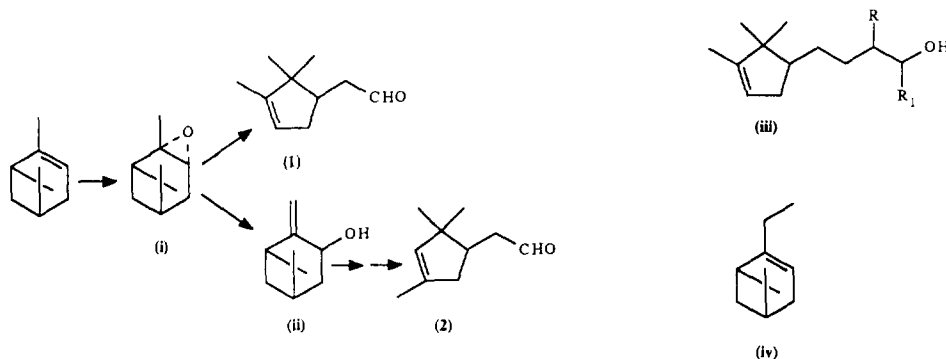

Fig. 1. Reaction scheme of the α-campholene and fencholene derivatives (**1**) and (**2**); α-pinene oxide (**i**); trans-pinocarveol (**ii**); fragrance compounds (**iii**) and ethylapopinene (**iv**).

tion of further stereogenic centres in compounds of the type **iii** results in the course of the condensation and reduction reactions.

The individual structures of substances investigated in Part I are shown in Fig. 2.

2.2. Instrumentation

The GC–MS analyses were carried out on a Hewlett-Packard HP 5890 II gas chromatograph–HP 5971A mass-selective detector. Elec-
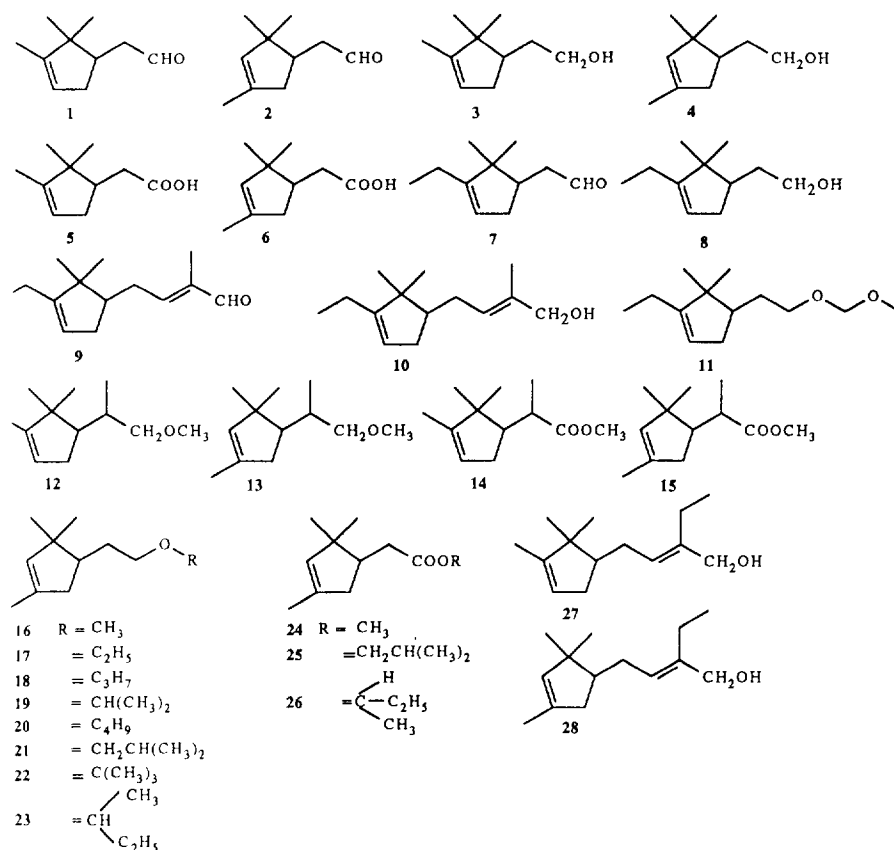

Fig. 2. Investigated substances. **1** = 2-(2,2,3-Trimethyl-3-cyclopentenyl)ethanal (α-campholene aldehyde); **2** = 2-(2,2,4-trimethyl-3-cyclopentenyl)ethanal (fencholene aldehyde); **3** = 2-(2,2,3-trimethyl-3-cyclopentenyl)ethanol; **4** = 2-(2,2,4-trimethyl-3-cyclopentenyl)ethanol; **5** = 2-(2,2,3-trimethyl-3-cyclopentenyl)acetic acid; **6** = 2-(2,2,4-trimethyl-3-cyclopentenyl)acetic acid; **7** = 2-(3-ethyl-2,2-dimethyl-3-cyclopentenyl)ethanal; **8** = 2-(3-ethyl-2,2-dimethyl-3-cyclopentenyl)ethanol; **9** = 2-methyl-4-(3-ethyl-2,2-dimethyl-3-cyclopentenyl)but-2-enal; **10** = 2-methyl-4-(3-ethyl-2,2-dimethyl-3-cyclopentenyl)but-2-enol; **11** = methoxymethyl-[2-(3-ethyl-2,2-dimethyl-3-cyclopentenyl)ethyl] ether; **12** = methyl-[2-(2,2,3-trimethyl-3-cyclopentenyl)propyl] ether; **13** = methyl-[2-(2,2,4-trimethyl-3-cyclopentenyl)propyl] ether; **14** = methyl-2-(2,2,3-trimethyl-3-cyclopentenyl)propionate; **15** = methyl-2-(2,2,4-trimethyl-3-cyclopentenyl)propionate; **16** = methyl-[2-(2,2,4-trimethyl-3-cyclopentenyl)ethyl] ether; **17** = ethyl-[2-(2,2,4-trimethyl-3-cyclopentenyl)ethyl] ether; **18** = [2-(2,2,4-trimethyl-3-cyclopentenyl)ethyl]propyl ether; **19** = isopropyl-[2-(2,2,4-trimethyl-3-cyclopentenyl)ethyl] ether; **20** = butyl-[2-(2,2,4-trimethyl-3-cyclopentenyl)ethyl] ether; **21** = 2-(2,2,4-trimethyl-3-cyclopentenyl)ethyl-(2-methylpropyl) ether; **22** = *tert.*-butyl-[2-(2,2,4-trimethyl-3-cyclopentenyl)ethyl] ether; **23** = but-2-yl-[2-(2,2,4-trimethyl-3-cyclopentenyl)ethyl] ether; **24** = 2-(2,2,4-trimethyl-3-cyclopentenyl)acetic acid methyl ester; **25** = 2-(2,2,4-trimethyl-3-cyclopentenyl)acetic acid (2-methylpropyl) ester; **26** = 2-(2,2,4-trimethyl-3-cyclopentenyl)acetic acid but-2-yl ester; **27** = 2-ethyl-4-(2,2,3-trimethyl-3-cyclopentenyl)but-2-enol; **28** = 2-ethyl-4-(2,2,4-trimethyl-3-cyclopentenyl)but-2-enol.

Table 1

No.	Column	Length (m)	Basic phase	Supplier
1	FS-CYCLODEX alpha I/P	50	OV-1701	CS-Chromatographie Service
2	CP-CD-β-2,3,6 M19	50	OV-1701	Chrompack
3	β-DEX 110	60	SPB 35	Supelco
4	γ-DEX 110	60	SPB 35	Supelco

tron impact ionization (70 eV) was used; the spectra were obtained in scan mode (mass range 35–400) by using helium as carrier gas. For all other analyses, a Hewlett-Packard 5890 II gas chromatograph, equipped with flame ionization detector and split/splitless injector, was available. As carrier gas, hydrogen with a split ratio of 1:100 was chosen.

The chiral resolution cR_s given in the tables was calculated according to Eq. 1.

$$cR_S = 1.177 \cdot \frac{t_{R(2)} - t_{R(1)}}{w_{h(1)} + w_{h(2)}} \tag{1}$$

where the indices 1 and 2 refer to the first- and second-eluting enantiomers, respectively.

The capacity factors k' given in the tables were calculated from the retention times of the stereoisomers and the dead time which was estimated by coinjection of methane.

2.3. Capillary columns

The columns used are shown in Table 1. All capillaries were 0.25 mm I.D. and have the film thickness $d_f = 0.25$ μm.

3. Results and discussion

In order to distinguish the target compounds from any possible impurities and by-products, all mixtures were investigated by GC–MS on chiral and non-chiral columns. The determination of the separation and capacity factors and of the chiral resolution cR_s took place by subsequent analyses by GC–flame ionization detection (FID) at different column temperatures after optimization of the chromatographic conditions. All the substances were analyzed within a temperature interval of about 50°C. In the tables, the separation and capacity factors for the individual compounds are given only at the temperature at which the resolution was highest.

The results of the initial substances used for the separation are shown in Table 2.

Several reports are known on the separation of the two enantiomers of α-pinene [22–25] (but not the α-pinene oxide) by means of capillary GC. Permethylated α-cyclodextrin dissolved in OV-1701 shows the highest enantioselectivity (Table 3) for the simple reduction and oxidation products of α-campholene and fencholene aldehyde with only short side chains (1–6).

The separation of the non-derivatized and

Table 2
Separation of initial substances

Compound	First enantiomer	Separation factor, α	Conditions
α-Pinene	(1S)-(−)	1.050	Column 2/70°C
α-Pinene oxide	(1R)-(−)	1.091	Column 2/100°C
Ethylapopinene	R-(−)	1.018	Column 3/120°C
Ethylapopinene oxide	S-(−)	1.042	Column 3/120°C

Table 3
Separation factors (α), capacity factors (k') and chiral resolution (cR_s) on permethylated α-, β- and γ-cyclodextrins for **1–8** at column temperature T

No.	α-CD (column 1)				β-CD (column 2)				β-CD (column 3)				γ-CD (column 4)			
	α	k'	T (°C)	cR_s	α	k'	T (°C)	cR_s	α	k'	T (°C)	cR_s	α	k'	T (°C)	cR_s
1	1.025	17.31 / 17.75	78	1.46	1.00	17.23	80	0.00	1.006	18.62 / 18.74	80	0.69	1.00	10.40	90	0.00
2	1.034	13.78 / 14.25	78	2.80	1.00	11.39	88	0.00	1.00	9.36	90	0.00	1.00	7.65	88	0.00
3	1.021	24.87 / 25.39	90	1.62	1.00	32.24	90	0.00	1.00	9.57	110	0.00	1.00	10.28	110	0.00
4	1.023	21.72 / 22.22	88	1.41	1.008	8.99 / 9.07	110	0.64	1.009	7.91 / 7.98	110	0.82	1.00	7.61	107	0.00
5	1.030	34.18 / 35.21	110	1.52	1.013	48.60 / 49.22	110	0.96	1.013	39.60 / 40.13	110	1.09	1.00	25.70	120	0.00
6	1.045	20.47 / 21.39	115	2.24	1.059	27.86 / 29.51	115	2.99	1.062	16.85 / 17.89	120	4.63	1.016	19.08 / 19.38	120	1.16
7	1.00	41.78	80	0.00	1.00	21.71	110	0.00	1.00	19.40	110	0.00	a			
8	1.00	32.23	100	0.00	1.011	29.85 / 30.18	105	1.29	1.010	24.56 / 24.80	120	1.58	a			

a No values determined.

highly polar acids 5 and 6 is remarkable, espe-
cially because as a rule the analysis of carbonic
acids on cyclodextrin derivatives took place after
the derivatization to esters [26,27]. Only occa-
sionally separations of non-derivatized com-
pounds are reported [4]. Resolution is satisfac-
tory, also on permethylated β-cyclodextrin (Fig.
3).

However, on non-polar stationary phases (OV-
1), basically asymmetric peaks appear for these
compounds, irrespective of their sample con-
centration and chromatographic conditions. The
good separability on the cyclodextrin phases
used should be due to the fact that the non-polar
chiral selector is dissolved in medium-polar poly-
siloxanes.

As in the conditions for enantiomer separa-
tion, the retention time becomes very long for
the carbonic acids (for the α-campholene acid 5
especially) it should be reduced by shortening
the columns accordingly, as recommended ear-
lier [28]. Therefore 5 and 6 were analyzed on a
25-m capillary with permethylated β-cyclodextrin
dissolved in polysiloxane.

The results from a comparison with the 50-m
capillary (Table 4) show that the chiral res-
olution decreases with shorter columns. If the
enantioselectivity of the chiral selector is small, a
separation of the enantiomers is not possible (5
on the short column). A simultaneously decrease
of the column temperature to obtain a higher
separation factor [29] also cannot be recom-
mended, because the difference in retention
times on the long and the short columns would
be negligible small. Therefore we employ a
shorter column only if the separation factor is
higher than 1.04 (see also [30]).

As the synthesized compounds were obtained
from (−)- or (+)-α-pinene with a great en-
antiomeric excess, it was possible to ascertain the
elution order of the enantiomers. With the ex-
ception of 1 on column 3, the (1R)-enantiomers
elute before the (1S)-enantiomers on all the
columns.

The separation of the α-campholene com-
pounds 9, 10 and 27 on permethylated cyclodex-
trin phases is not possible. This is remarkable
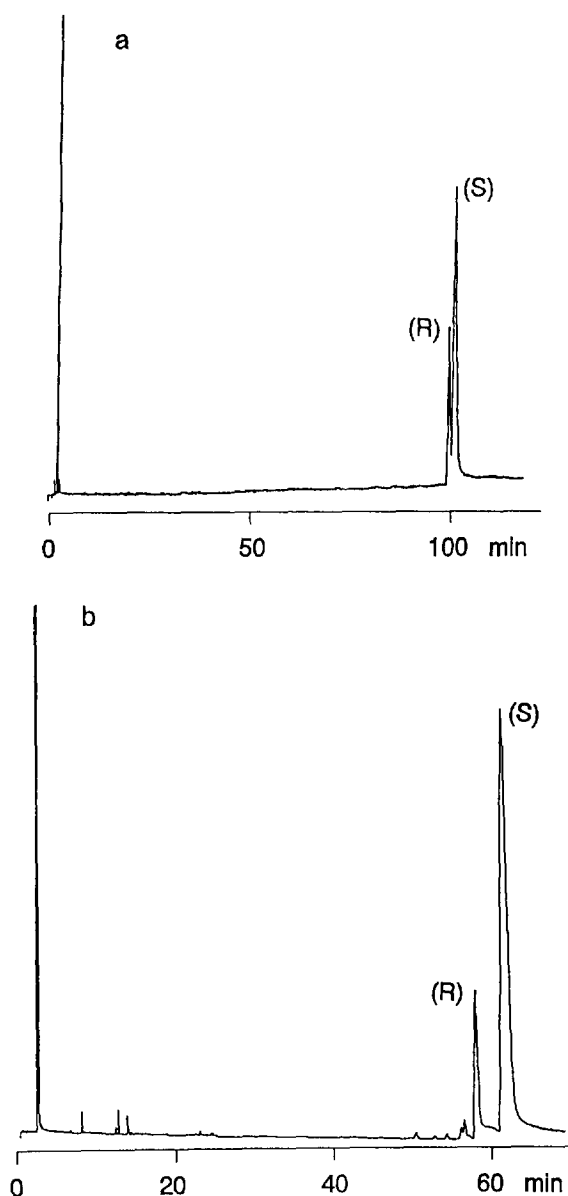because the respective fencholene compound

Fig. 3. Chromatograms of (a) α-campholenic acid (5) and (b)
fencholenic acid (6) on permethylated β-cyclodextrin (col-
umn 2). Column temperature: 110°C (a) or 115°C (b);
injection and FID temperatures: 275 and 250°C, respectively;
carrier gas: hydrogen; split ratio 1:100.

(28) is separated in the same way as α-cam-
pholene derivatives with alkyl chains comparable
with 9 and 10 (see [31]). Accordingly, the posi-
tion of substituents on the five-membered ring
and the conformative mobility as well as the

Table 4
Separation factors (α), capacity factors (k') and chiral resolution (cR_s) on capillaries with different length coated with permethylated β-cyclodextrin for α-campholene acid (5) and fencholene acid (6)

No.	50-m Capillary				25-m Capillary			
	α	k'	T (°C)	cR_s	α	k'	T (°C)	cR_s
5	1.009	19.36 19.53	125	0.78	1.00	19.70	125	0.00
	1.013	48.60 49.22	110	0.96	1.011	49.68 50.29	110	0.55
6	1.050	15.09 15.84	125	3.11	1.047	15.30 16.03	125	2.35
	1.059	27.86 29.51	115	2.99	1.059	28.74 30.45	115	2.58

distance of functional groups from the stereogenic centre in the ring have an influence on the separability of the enantiomers of α-campholene and fencholene derivatives.

The results for the ethers 12, 13 and 16–23, and esters 14, 15 and 24–26 are given in Table 5. For better comparison, ethers and esters with several stereogenic centres are not discussed in Part II but already here.

The polarity of the compounds decreases from

Table 5
Separation factors (α), capacity factors (k') and chiral resolution (cR_s) on permethylated α- and β-cyclodextrins for esters and ethers of α-campholene and fencholene derivatives at column temperature T

No.		α-CD (column 1)				β-CD (column 2)			
		α	k'	T (°C)	cR_s	α	k'	T (°C)	cR_s
12	D1[b]	1.00	29.50	70	0.00	1.00	28.64	70	0.00
	D2	1.025	32.47 33.28	70	1.29	1.00	32.57	70	0.00
13	D1	1.00	21.95	70	0.00	1.00	20.28	70	0.00
	D2	1.00	24.19	70	0.00	1.009	23.79 24.01	70	0.72
14	D1	1.00	28.31	85	0.00	1.00	26.69	85	0.00
	D2	1.018	30.60 31.15	85	1.05	1.009	29.31 29.58	85	0.74
15	D1	1.00	21.46	85	0.00	1.00	19.22	85	0.00
	D2	1.00	22.42	85	0.00	1.00	21.57	85	0.00
16		1.00	28.85	60	0.00	1.011	29.04 29.36	60	0.85
17		1.017	32.89 33.46	65	0.94	1.025	31.46 32.26	65	1.59
24		1.016	22.62 22.98	80	0.90	1.030	22.47 23.14	80	1.18
25			a			1.015	21.80 22.13	105	1.38
26	D1		a			1.022	24.07 24.61	100	2.20
	D2		a			1.012	24.33 24.61	100	1.09

a No values determined.
b The marking of the individual diastereomers was done schematically by D1 and D2, with D1 corresponding to the diastereomers eluting first on the polar stationary phases (Carbowax) and D2 corresponding to the diastereomer eluting last.

the corresponding alcohols via the esters to the ethers. Connected with this a decrease is found in the enantioselectivity of permethylated α- and β-cyclodextrin both for the α-campholene and the fencholene derivatives. The results show that dipole interactions between the alcohols (or the esters and ethers derived from it) and the chiral selector of the stationary phase contribute to the enantiomer differentiation on permethylated cyclodextrins but they are not their sole cause.

In comparison with 13 and 15, the separation factors for 16 and 24 are, as expected, slightly larger both on permethylated α- and on β-cyclodextrin. Because this is also the case for α-campholene and fencholene derivatives with two stereogenic centres [31], the better separation must be due to the smaller steric hindrance of the geminal methyl groups on the five-membered ring by the α-positioned methyl substituent in the alkyl side chain.

From the methyl ether 16 to the ethyl ether 17, the enantioselectivity increases initially on both phases, but gets completely lost with the further extension of the side chain so that 18–23 cannot be separated into the enantiomers. In contrast, the "chiral separation power" will change only slightly in the case of the polar esters 25 and 26. Also the acetates of the α-campholene and fencholene alcohols 3 and 4 are excellently separated into the enantiomers.

With the increasing space requirement of the alkyl substituents, the ether oxygen of the compounds is sterically more shielded so that for interactions with cyclodextrin it is available only to a limited extent, whereas one free oxygen atom is further available for interactions in the esters. These results also are in favour of a contribution to the enantiomer differentiation from polar interactions.

In the separation of 16 and 17 as well as 24 and 25, the R-enantiomers elute before the S-enantiomers in each case. It is remarkable that this elution sequence for 17 and 24 reverses on permethylated α-cyclodextrin (Fig. 4).

Even before, Armstrong et al. [32] and Bicchi et al. [33] had found an inversion of the elution sequence on α- and β-cyclodextrin when using similar cyclodextrin derivatives for selected
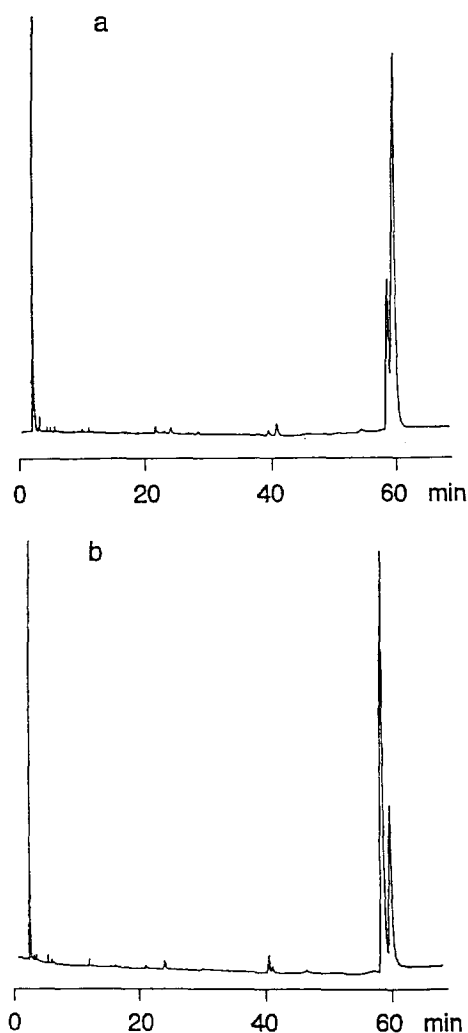

Fig. 4. Chromatograms of 17 on (a) permethylated α-cyclodextrin (column 1) and (b) permethylated β-cyclodextrin (column 2). Column temperature: 65°C; injection and FID temperature: 250°C; carrier gas: hydrogen; split ratio 1:100.

species. Consequently, by selecting the respective stationary phase, it can be made sure that an enantiomer present in excess will elute as second peak in each case. Furthermore, errors that might occur in the determination of the enantiomeric ratio or of the enantiomeric excess will be reduced in this way, for example by the peak area of a component with a small share being influenced by the tailing of the respective surplus component.

Table 6
Separartion factors (α), capacitiy factors (k') and chiral resolution (cR_s) on permethylated β-cyclodextrin for α-campholene and fencholene derivatives with longer alkyl side chains (flavour compounds)

No.	β-CD (column 2)				β-CD (column 3)			
	α	k'	T (°C)	cR_s	α	k'	T (°C)	cR_s
27	1.00	22.19	130	0.00	1.00	21.25	130	0.00
28	1.019	16.44	130	1.69	1.016	15.60	130	1.65
		16.76				15.85		

α-Campholene and fencholene derivatives with woody and sandal notes (e.g. **10, 27, 28**) can only be separated on permethylated β-cyclodextrin phases. Separation factors for two selected representatives are given in Table 6. In all cases, the R-enantiomer elutes before the S-enantiomer.

Different odour notes of α-campholene and fencholene compounds correlate with the different separability of the two derivatives. Obviously, the conformation differences which are due to the different position of the vinylic methyl group in the five-membered ring influence both odour notes and enantiomer separation.

4. Conclusions

Beginning with the starting substances for the synthesis of woody and sandal wood fragrances of α-campholene and fencholene types all obtained compounds can be separated very well into their enantiomers by using permethylated cyclodextrins dissolved in medium-polar polysiloxanes. For aldehydes, alcohols and acids with short alkyl side chains we employ permethylated α-cyclodextrin; for compounds with longer alkyl chains permethylated β-cyclodextrin can be recommended.

The separation is slightly dependent on the polarity of the compounds and the position of methyl substituents to each other. If there is a steric hindrance of the methyl groups on the five-membered ring, the separation will deteriorate.

Acknowledgement

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