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Note

Gas chromatographic separation of enantiomers of O-acyl alcohols

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We have previously accomplished the first direct enantiomer separation of some chiral alcohols[1,2] but rather long retention times are required for efficient separation. More recently we showed the excellent separation of some alcohols and α-hydroxycarboxylic acid enantiomers in the form of N-isopropyl carbamates[3] with moderate retention times. König and co-workers[4,5] have also reported independently the same procedure for the enantiomer separation of alcohols, amines and hydroxy acids. However, alcohol enantiomers have never been resolved in the form of O-acyl derivatives.

We have found that some novel amide phases are suitable for the separation of some carboxylic acid ester enantiomers[6–8]. This result suggested that these phases might be effective as chiral stationary phases for the separation of O-acyl alcohol enantiomers. In this paper, we describe the separation of some O-acyl alcohol enantiomers using such chiral amide phases.

EXPERIMENTAL

Synthesis of stationary phase

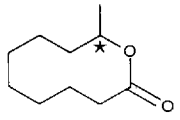
N-(1*R*,3*R*)-*trans*-Chyrsanthemoyl-(*R*)-1-(α-naphthyl)ethylamine (phase I)[6] and O-(1*R*,3*R*)-*trans*-chrysanthemoyl-(*S*)-mandelic acid (*R*)-1-(α-naphthyl)ethylamide (phase II)[8] were prepared as described previously.

Gas chromatography

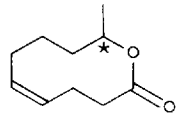
The experiments were carried out with a Shimadzu GC-7A gas chromatograph equipped with a flame-ionization detector. The glass capillary columns (40 m × 0.25 mm I.D.) were coated with a 5% solution of each stationary phase in chloroform.

Several chiral alcohols were O-acylated with acetic anhydride or trifluoroacetic anhydride.

Phoracantholide I and phoracantholide J were kindly supplied by Dr. Kenji Mori (Tokyo University, Tokyo, Japan).

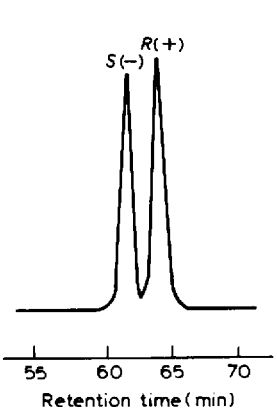


Phoracantholide I



Phoracantholide J

0021-9673/83/0000-0000/$03.00



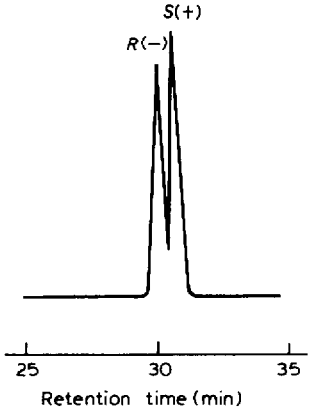


Fig. 1. Gas chromatogram of racemic O-acetyl-1-phenylethanol. Glass capillary column (40 m × 0.25 mm I.D.) coated with N-(1*R*,3*R*)-*trans*-chrysanthemoyl-(*R*)-1-(α-naphthyl)ethylamine. Temperature: 80°C. Carrier gas: helium at a flow-rate of 0.7 ml/min.

Fig. 2. Gas chromatogram of racemic phoracantholide I. Glass capillary column (40 m × 0.25 mm I.D.) coated with O-(1*R*,3*R*)-*trans*-chrysanthemoyl-(*S*)-mandelic acid (*R*)-1-(α-naphthyl)ethylamide. Temperature: 100°C. Carrier gas: helium at a flow-rate of 0.7 ml/min.

TABLE I

GAS CHROMATOGRAPHIC SEPARATION OF O-ACETYL ALCOHOL ENANTIOMERS

Glass capillary column (40 m × 0.25 mm I.D.). Carrier gas: helium at a flow-rate of 0.7 ml/min.

Compound	*Column temperature (°C)*	*Optically active stationary phase*					
		Phase I			*Phase II*		
		Retention time (min) α***			*Retention time (min)* α***		
		1st peak	*2nd peak*		*1st peak*	*2nd peak*	
O-Acetyl-1-phenylethanol	80	61.5(*S*)	64.0(*R*)	1.040	47.6(*S*)	50.4(*R*)	1.058
O-Trifluoroacetyl-1-phenyl-ethanol	80	8.4	8.4	1.000	6.1	6.3	1.026
O-Acetyl-1-phenyl-2,2,2-trifluoro-ethanol	80	17.5	18.0	1.029	15.0	15.4	1.028
O-Acetyl-1-(α-naphthyl)ethanol	150	116.1	120.2	1.035	84.2	87.3	1.037
O-Trifluoroacetyl-1-(α-naphthyl)ethanol	120	72.9	74.0	1.014	22.8***	23.4***	1.026***
O-Acetyl-Pantoyllactone	120	51.2(*R*)	53.1(*S*)	1.037	41.3(*R*)	42.7(*S*)	1.034
O-Trifluoroacetyl-Pantoyllactone	120	9.6	9.9	1.025	18.9§	19.3§	1.019§
Phoracantholide I	100	–	–	–	30.2(*S*)	30.8(*R*)	1.018
Phoracantholide J	100	–	–	–	41.1(*S*)	41.7(*R*)	1.016

* Measured from solvent peak.
** Separation factor (2nd/1st).
*** Measured at 130°C.
§ Measured at 100°C.

RESULTS AND DISCUSSION

The gas chromatographic results are given in Table I and typical chromatograms are shown in Figs. 1 and 2.

Several O-acetylated and O-trifluoroacetylated alcohols were resolved into their antipodes on the two chiral amide phases. It should be also emphasized that phoracantholide I and J, which are prepared by intramolecular acylation of chiral alcohols, can be separated on phase II without any pre-treatment.

The direct separation of such lactone enantiomers is significant for the determination of optical purity, as derivatization into their diastereoisomers is difficult with these compounds.

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