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Effect of mobile phase composition on the enantioselectivity of chromatographic separation on a quinine-bonded silica stationary phase

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Abstract

The effect of the nature and of the composition eluent on the enantiomer separation of 2,2,2-trifluoro-1-(9-anthryl)ethanol (TFAE) on a chiral stationary phase under HPLC conditions was studied. A series of solvents with different properties were examined as the mobile phase. The best enantioselectivity was achieved with pure benzene, methylene chloride and carbon tetrachloride. The results obtained are discussed in accordance with the three-point interaction model and solvatochromic parameters of the solvents. It is shown that the selectivity of enantiomer separation depends mainly on the hydrogen-accepting ability and dipolarity–polarizability parameters of the solvent. A simplified model of the retention and separation of TFAE enantiomers is proposed.

1. Introduction

Over the last decade there has been a considerable increase in the number of publications dedicated to enantiomer separations by HPLC with chiral stationary phases (CSPs). For enantiomer separation there should be at least a three-point interaction between the chiral bonded selector and the molecule of an optically active sample compound. These conditions are provided with different silica surface modifiers [1,2]. The development of new stationary phases remains the main way to improve the enantioselectivity of chromatographic separations [3–5]. In our opinion, however, the resources of already developed CSPs have not been exhausted and can be improved by optimization of the mobile phase composition.

A number of workers have attempted to find relationships between enantioselectivity and composition of achiral mobile phase in chiral phase liquid chromatography [6–9]. Most of the work has involved binary or tertiary solvent systems. Generally, these studies were concerned with elucidating the influence of polar additives to mobile phase on the enantioselectivity of separation. Siret *et al.* [7] found a unique reversal of the elution order of enantiomers on changing from hexane–2-propanol to hexane–chloroform or hexane–methylene chloride mobile phases. A similar result was observed by Pirkle *et al.* [9] but for normal- and reversed-phase modes. However, the effect of the nature of the organic solvent used as a single-solvent mobile phase on the separation of enan-

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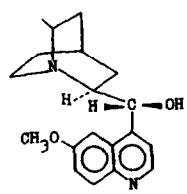
tiomers has not been investigated. In this study we considered both the influence of polar additives in the mobile phase and the nature of the organic solvent on the enantioselectivity of separation of (*R*,*S*)-2,2,2-trifluoro-1-(9-anthryl)ethanol [(*R*,*S*)-TFAE] on a quinine-bonded chiral stationary phase (CSP).

2. Experimental

The measurements were carried out with a liquid chromatographic system composed of a Beckman (Berkeley, CA, USA) Model 114M pump, a Rheodyne (Cotati, CA, USA) Model 7125 injection valve with a 20-μl loop and a Uvicord S 2238 UV detector (LKB, Bromma, Sweden) with 254- and 365-nm interference filters. The stainless-steel column (250 × 4.0 mm I.D.) was slurry packed with quinine-bonded silica. The CSP with bonded quinine was prepared by modification of the surface of silica (Silasorb Si 300, 5 μm; Lachema, Brno, Czech Republic) with the triethoxysilyl derivative of quinine:

$$\equiv Si{-}OH + (C_2H_5O)_3Si(CH_2)_2Quin \longrightarrow \equiv Si{-}O{-}Si(CH_2)_2Quin$$

where Quin =



A 10-g amount of silica dried under vacuum at 200°C was treated with the triethoxysilyl derivative of quinine in 100 ml of dry toluene at 100°C for 10 h. The modified silica was washed with benzene, diethyl ether, ethanol and acetone. The sample contained 5.6% C, which corresponds to 0.5 mmol/m^2 of bonded quinine molecules. Part of the prepared CSP was treated with trimethylchlorosilane for end-capping residual silanol groups.

Optically pure (*S*)- and (*R*)-TFAE isomers were purchased from Aldrich (Milwaukee, WI, USA). Chromatographic-grade hexane, 2-propanol, methylene chloride, carbon tetrachloride and chloroform (J.T. Baker) and purified benzene, toluene, *o*-, *m*- and *p*-xylene, bromobenzene, benzonitrile and 1,1-dichlorethane were used for mobile phase preparation. The elution order of enantiomers was determined on the retention of pure (*S*)- and (*R*)-TFAE.

3. Results and discussion

Among the most useful CSPs are the "brush-type" phases, which consist of a silica matrix with covalently bonded chiral groups. Most "brush-type" CSPs contains at least three functional groups required for chiral recognition in accordance with Dalgliesh's work [10]. For quinine this concept is applicable. Quinine and TFAE provide a hydroxyl group within the molecules suitable for intermolecular hydrogen bonding, and they contain aromatic fragments for $\pi-\pi$ interactions. These interactions combined with steric hindrance are responsible for the separation of optical isomers of arylalkylcarbinols and binaphthols with quinine-type CSPs [11–15].

3.1. Role of the structure of arylalkylcarbinols on retention and enantioselectivity of separation

Arylalkylcarbinols are the most often investigated substrates for optical resolution with quinine CSPs. The following conclusions have been drawn regarding the retention and separation of optical isomers of these compounds. First, it has been observed that steric hindrance of the alkyl group on the assymmetric centre is important for obtaining a good separation factor [11,14]. However, the volume of CF_3, CH_3 and C_2H_5 substituents on the chiral centre of alkylphenylcarbinols can be calculated to be insufficient for the appearance of steric hindrance by interaction with quinine residues. There was no visible resolution for the corresponding racemic alkylphenylcarbinols on this chiral phase [14]. Second, it has been shown that

the donor–acceptor properties of substituents of the phenyl rings play an important role. With decreasing electron-donating ability of a *para* substituent on the phenyl ring of carbinols [4-CH_3 >unsubstituted > 4-Cl > 4-$(CH_3)_2N$ > 4-CF_3 > 3, 4-Cl_2] the capacity factor for the first eluted enantiomer increases from 2.3 to 6.8. At the same time, the enantioselectivity of separation is slightly changed from 1.05 to 1.10 [14]. The replacement of a methyl group with a trifluoromethyl group adjacent to the chiral centre of carbinols causes an increase in the α and k' values. As concluded, the hydrogen bond between the C_9-OH group of the bonded quinine with the OH group of arylalkylcarbinols remains a strong interaction that defines the enantioselectivity of separation.

3.2. Chiral recognition model of bonded quinine to 2,2,2-trifluoro-1-(9-anthryl)ethanol

TFAE was chosen for this study because it is a well investigated compound; it meets many requirements for multi-point interactions of selector and selectand and has been separated with α values between 1.1 and 1.17 using three chiral stationary phases containing quinine residues covalently attached to the silica surface via different routes as a chiral selector [11,12,15]. The absorption maximum of TFAE at 320–360 nm allows mobile phase solvents that are UV absorbing up to 280 nm or higher to be used.

It can be assumed that the retention mechanism of TFAE on quinine CSPs is similar to that for polar compounds on amino-bonded stationary phases in normal-phase LC. There are three models, proposed by Snyder and Shunk [16], Scott and Kucera [17] and Hennion *et al.* [18], which are applicable for the description of retention on polar amino-bonded phases. For the correct choice one should consider the main types of interaction of TFAE isomers with a quinine CPS:

(1) Usually, a "one-to-one" interaction of the solute (TFAE) with the quinine moiety of a "brush-type" CSP is considered for chiral recognition.

(2) A TFAE molecule can displace any number of preadsorbed solvent molecules. However, with separated enantiomers the distinction in retention and enantioselectivity of separation is defined by displacement of different numbers of solvent molecules that interact with definite functional group(s) near the asymmetric centre of the CSP. The C_9-OH group of bonded quinine would be one of them.

(3) The interaction between TFAE and solvent molecules can contribute to k'. This interaction may be of some importance for α if related to group(s) which is (are) responsible for chiral recognition (*e.g.*, the OH group in the TFAE molecule).

The above statements are generally satisfactory for the model proposed by Hennion *et al.* [18] for the retention of polar solutes on amino-bonded phases. This model can be used for a description of the retention of TFAE on a quinine CSP with hexane–2-propanol mixtures as mobile phase.

For one of enantiomers of TFAE one can propose that the formation of a hydrogen bond between the C_9-OH group of the quinine residue and the OH group of TFAE leads to displacement of at least one additional molecule of preadsorbed 2-propanol in comparison with its optical antipode. This leads to a dependence of enantioselectivity α on the nature of the mobile phase.

3.3. Influence of the nature of the mobile phase

Several mobile phases have been used for the separation of TFAE enantiomers on quinine CSPs, *e.g.*, hexane–2-propanol [11,13,15], methylene chloride, acetonitrile–methylene chloride [12] and benzene [15], and different separation factors were obtained. In order to evaluate this effect we studied the influence of the nature of the mobile phase on the enantioselectivity of separation by using binary and single-solvent mobile phases.

Binary mobile phases

In the first step, different binary solvent systems consisting of hexane, benzene, carbon tetrachloride, toluene and *o*-, *m*- and *p*-xylene and with various contents of 2-propanol and

methanol were studied as mobile phases. It was found (Table 1) that an increase in the alcohol content led in all instances to a decrease in the retention times of the TFAE isomers and in enantioselectivity (Figs. 1 and 2). These results confirm that hydrogen bonding between the quinine CSP and TFAE is a determining factor for enantioselectivity in aprotic solvents. According to this, a different number of polar solvent molecules may be displaced from quinine residues during the formation of surface diastereomeric complexes.

As shown in Fig. 3, the plots of log α *versus* content of 2-propanol for different bulk solvents are not straight and are closely related to the curve for titration of quinine groups by 2-propanol in hexane. Fig. 3 shows only one region (−log [IP] from 2 to 2.8–3.0) that is approximately linear. In this region the hydrogen-bonding interactions between the solute and quinine became stronger with decrease in the 2-propanol content. A further decreasing in the polar solvent content should lead to a convex curve owing to the growing contribution to hydrogen bonding interactions of traces of water present in the organic solvent. The enantioselectivity for pure bulk solvents is dependent on their water content. For high concentrations of 2-propanol (2–5%) the curve is concave. This may be connected with self-association of 2-propanol molecules in the mobile phase and stabilization of the quinine–2-propanol associate.

A similar dependence (Fig. 3) was obtained for (*R*, *S*)-β,β-binaphthol and hexane–2-propanol mobile phases with various contents of the polar constituent. It should be mentioned that in accordance with NMR investigations [19], the hydrogen bonding is the dominant interaction in the chiral recognition of β,β-binaphthol with bonded quinine.

Dobashi *et al.* [8] investigated the retention mechanism and the role of hydrogen bonding in the separation of enantiomers of *N*-acetyl-D,L-leucine O-methyl ester on a diamide-bonded L-valine chiral stationary phase. The calculated values of log α for hexane–2-propanol mobile phases show a similar dependence (Fig. 3) as obtained for TFAE and β,β-binaphthol with quinine CSPs. The chiral recognition of the diamide CSP–*N*-acetyl-D,L-leucine O-methyl ester is connected with stronger hydrogen-bonding interactions than for the TFAE–quinine CSP system, so the "titration curve" of log α *versus* N_{IP} was observed at higher concentrations of 2-propanol in hexane. It should be noted that the hydrogen bonding was also the main interaction for the diamide-bonded L-valine CSP.

On replacement of hexane with other non-polar organic solvents, *e.g.*, benzene or carbon tetrachloride, the character of the log α–N_{IP} relationship does not change significantly (Fig. 3). A similar dependence of log α as a function of 2-propanol content was obtained.

The nature of the polar additive to the mobile phases remains important. Stuurman *et al.* [12] investigated the influence of the type of alcohol and water as polar additives to the mobile phase on the retention and separation of TFAE. There was no significant difference between 2-propanol, *tert.*-butanol and 1-pentanol, and a slight decrease of retention and selectivity was noted for methanol as additive. The influence of water was the same as for the addition of alcohols. The author's conclusion [12] about the weak competition of polar additives (alcohols, water) with (*R*)- and (*S*)-TFAE for interaction sites of quinine residues is not, however, in good agreement with our results.

Single-solvent mobile phases

One could propose that the separation factor α should be increased significantly when pure aprotic solvents are used as mobile phases. Therefore, it was of interest to compare the effects of pure solvents with different properties and polarities on chiral recognition and enantioselectivity. Different solvents were investigated as mobile phases for the separation of (*R*)- and (*S*)-TFAE on the quinine-bonded CSP. The characteristics of the chosen solvents are summarized in Table 2.

The data in Table 3 show enantioselectivity for (*R*,*S*)-TFAE, confirming the supposition that the hydrogen donor–acceptor ability of a pure solvent is an important part of chiral recognition with a quinine CSP. The retention times of the

Table 1
Influence of 2-propanol content on retention and separation of (*S*)- and (*R*)-TFAE

Solvent	2-Propanol content (%, v/v)	k'_1	$1/k'_1$	α
Hexane	0.5	17.99	0.06	1.10
		(13.96)[a]	(0.07)	(1.10)
	1.0	8.01	0.13	1.08
		(6.33)	(0.16)	(1.06)
	2.5	3.93	0.26	1.07
		(2.29)	(0.44)	(1.04)
	5.0	1.72	0.58	1.06
		(1.23)	(0.81)	(1.03)
	10.0	0.87	1.14	1.03
		(0.62)	(1.61)	(1.02)
	20.0	0.58	1.73	–[b]
Benzene	0	2.80	0.36	1.46
	0.1	2.67	0.37	1.42
	0.2	1.57	0.64	1.35
	0.4	1.21	0.83	1.24
	0.5	1.13	0.89	1.25
	1.0	0.96	1.02	1.23
	2.0	0.57	1.75	1.20
Toluene	0	1.36	0.74	1.28
	0.2	1.03	0.97	1.23
	1.0	0.86	1.16	1.17
o-Xylene	0	1.74	0.57	1.26
	0.2	1.10	0.91	1.20
m-Xylene	0	2.00	0.50	1.35
	0.2	1.22	0.85	1.25
	1.0	0.88	1.14	1.22
p-Xylene	0	1.44	0.69	1.26
	0.2	1.22	0.82	1.22
	1.0	0.88	1.14	1.18
Bromobenzene	0.2	1.17	0.86	1.10
Carbon tetrachloride	0	15.04	0.07	1.53
	0.1	14.21	0.07	1.39
	0.2	8.56	0.12	1.28
	0.4	4.69	0.27	1.19
	1.0	1.89	0.52	1.13
	2.0	0.93	1.07	1.09
	5.0	0.49	2.04	1.0
Carbon tetrachloride	0.1[c]	14.36	0.07	1.32
	0.2	10.72	0.09	1.22
	0.5	4.78	0.21	1.09
	1.0	2.31	0.43	1.04

[a]Data obtained with undried hexane.
[b]No resolution of peaks.
[c]Methanol was used as a polar component.

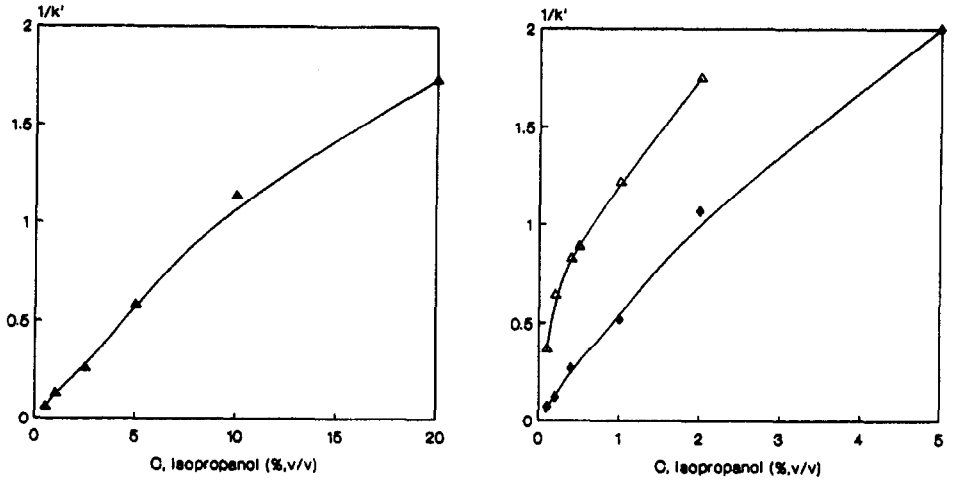


Fig. 1. Plot of $1/k_1'$ of (*R*)-TFAE *versus* concentration of 2-propanol in (▲) hexane, (△) benzene and (◆) carbon tetrachloride.

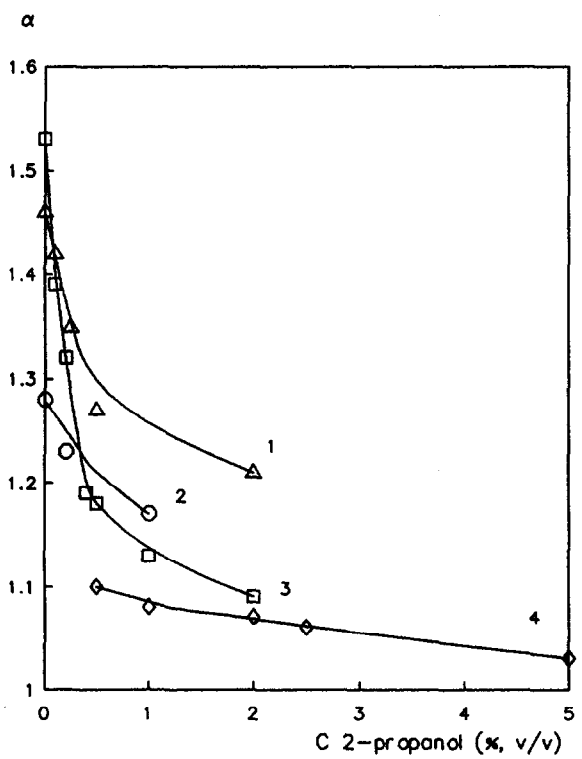


Fig. 2. Plot of the enantioselectivity α of (*R*,*S*)-TFAE *versus* the content of 2-propanol in (1) benzene, (2) toluene, (3) carbon tetrachloride and (4) hexane.

weakly retained enantiomer of TFAE are not in a good agreement with the polarity of the chosen solvents. Thus, k_1' values of 0.41 and 11.91 were observed respectively; with dibutyl ether ($P = 1.65$) and carbon tetrachloride ($P = 1.56$), the corresponding values of α are 1.00 and 1.53, respectively. The correlation of enantioselectivity with solvent selectivity parameters was carried out to obtain a better understanding of the results obtained and optimization of the mobile phase.

Two scales are mainly used to classify solvent properties in liquid chromatography. The first and most common is the Snyder solvent triangle [21] and the other is the solvatochromic scale of Kamlet *et al.* [22]. Both of them characterize hydrogen donor–acceptor ability and dipolarity–polarizability properties of solvents. It should be noted that a good correlation has been established between the solvent triangle and solvatochromic scales of solvent strength and selectivity [21].

The solvatochromic scale of Kamlet *et al.* includes the parameters π, α, β and δ, describing the solvent dipolarity–polarizability, hydrogen bond acidity, hydrogen bond basicity and the polarizability correction factor, respectively. The

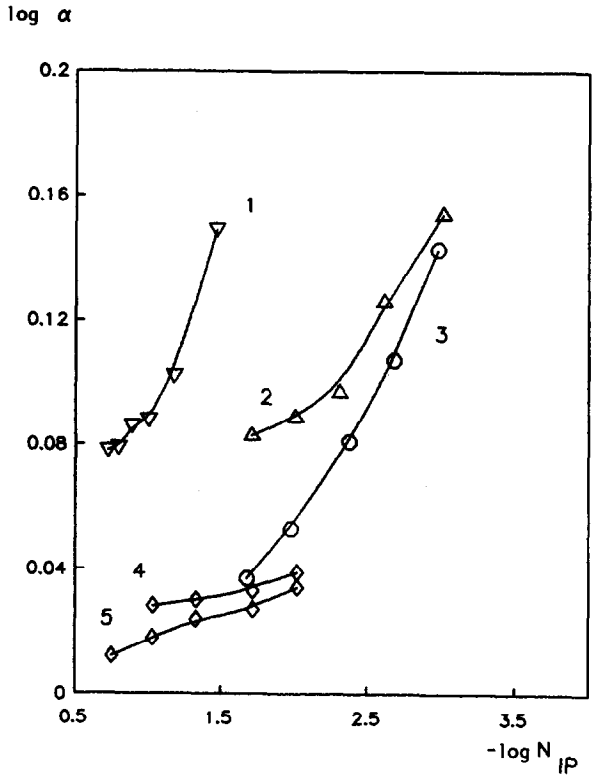


Fig. 3. Plot of log α *versus* log (content of polar solvent in the mobile phase). Stationary phases: (1) diamide-L-valine CSP and (2–5) quinine CSP. Solutes: (1) N-acetyl-D,L-leucine O-methyl ester, (2–4) (*R,S*)-TFAE and (5) (*R,S*)-β, β-binaphthol. Mobile phases: (1,4,5) hexane–2-propanol, (2) benzene–2-propanol and (3) carbon tetrachloride–2-propanol. Curve 1 was calculated from literature data [8].

values of these parameters are presented in Table 2. The polarizability correction term δ is zero for non-chlorinated alyphatic solvents, 0.5 for polychlorinated aliphatics and 1.0 for aromatic solvents. The correlations of enantioselectivity with the corresponded solvatochromic parameters are presented in Fig. 4.

Often these solvent parameters are used as linear energy parameters in an LSER (linear solvation energy relationship) [21]. Following the above reasoning and in accordance with previous results [21]:

$$\alpha = \alpha_0 + s\pi + a\alpha^* + b\beta + d\delta \quad (1)$$

The corresponding strong correlation of enantioselectivity with π, α^*, β and δ observed for TFAE is

$$\alpha = 1.54 + 0.13\pi - 0.67\alpha^* - 1.24\beta - 0.16\delta \quad (2)$$

$n = 10$, $r = 0.886$, F value = 4.57, significance = 0.063. The values of the parameters indicate that

Table 2
Solvatochromic parameters of solvents used as mobile phases

Solvent	Solvatochromic parameters[a] [22]		
	π	α^*	β
Hexane	−0.04		
Benzene	0.59	0	0.10
Toluene	0.55	0	0.11
Ethylbenzene	0.48		0.12
m-Xylene	0.47	0	0.13
p-Xylene	0.51	0	0.12
Methylene chloride	0.82	0.30	0
Chloroform	0.58	0.44	0
Ethylene dichloride	0.81	0	0
Carbon tetrachloride	0.28	0	0
Dibutyl ether	0.24	0	0.46
2-Propanol	0.48	0.76	0.95
Bromobenzene	0.79	0	0.06
Benzonitrile	0.90	0	0.41

[a] π = Dipolarity and polarizability; α^* = hydrogen bond donating acidity; β = hydrogen bond accepting basicity.

Table 3
Effect of the nature of the solvents on the enantioselectivity of the separation of (*S*)- and (*R*)-TFAE

No.	Mobile phase	*P* [21]	k'_1	k'_2	α
1	Benzene	3.19	2.80	4.09	1.46
2	Toluene	2.68	1.36	1.74	1.28
3	Ethylbenzene		1.65	1.87	1.14
4	*o*-Xylene		1.74	2.19	1.26
5	*m*-Xylene		2.00	2.70	1.35
6	*p*-Xylene	2.55	1.44	1.81	1.26
7	Dichloromethane	4.29	0.80	1.14	1.43
8	Chloroform	4.31	0.41	0.52	1.19
9	Carbon tetrachloride	1.56	15.04	23.01	1.53
10	Dichloroethane		0.26	0.40	1.50
11	Dibutyl ether	1.65	0.41	0.41	1.00
	Hexane–dichloromethane (60:40)		8.17	11.36	1.39
	Hexane–benzonitrile (80:20)		1.74	1.81	1.04

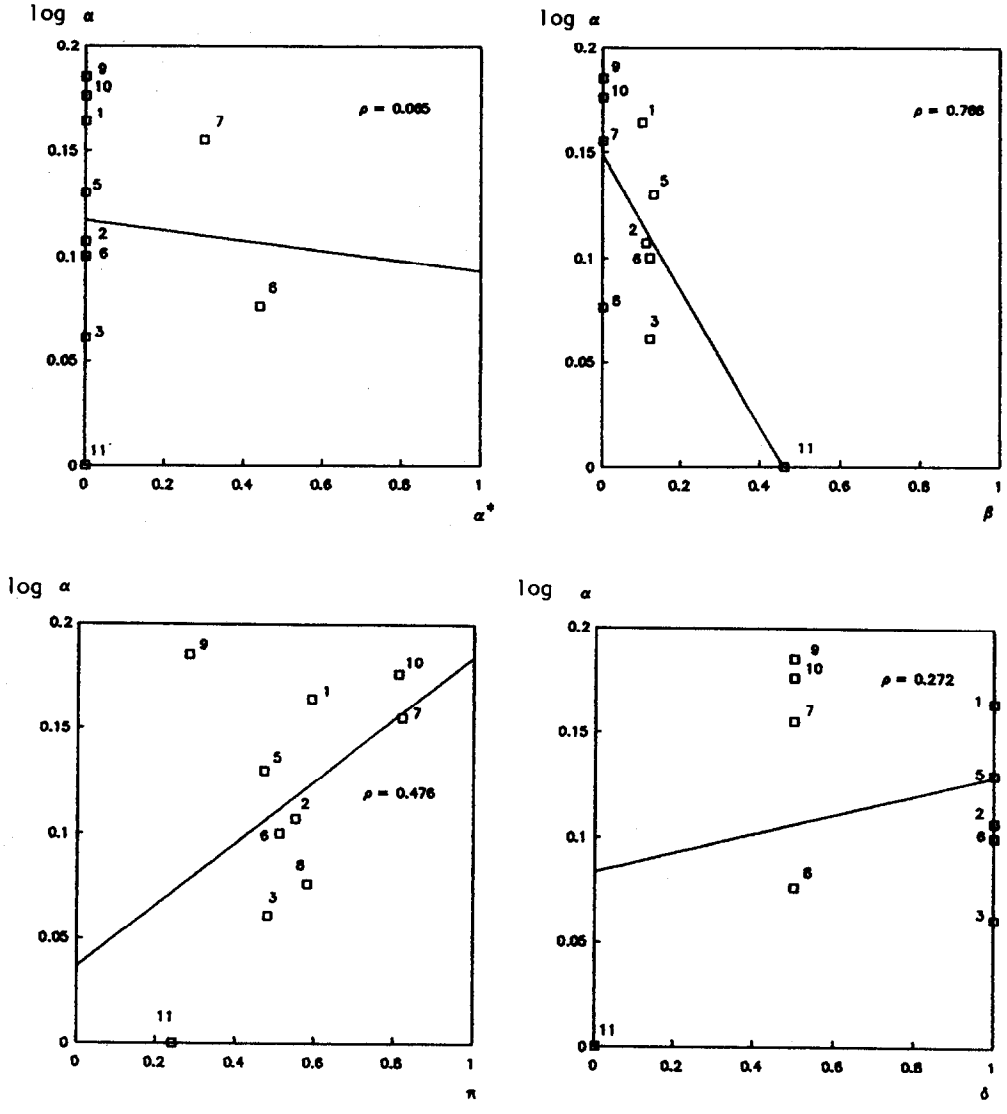


Fig. 4. Plot of enantioselectivity (α) *versus* solvatochromic parameters of organic solvents used as mobile phases. The numbering of the points for different solvents corresponds to those in Table 3.

a dipolarity–polarizability parameter was responsible for the separation of (*R*,*S*)-TFAE on the quinine CSP. Generally, it should be noted that the best enantioselectivity was achieved for solvents with weak hydrogen donor–acceptor ability (α, β) and high dipolarity–polarizability properties (π). The value of the free term 1.54 in eqn. 2 is equal to the hypothetical enantioselectivity in pure hexane.

The multiple linear regression analysis of the data obtained could be of more significance. Unfortunately, there were insufficient data for a representative analysis owing to the limited number of available organic solvents with known selectivity parameters and appropriate eluting power.

In practice, the use of non-polar and aprotic mobile phases in the absence of polar admixtures considerably increased the enantioselectivity for (*R*,*S*)-TFAE on a CSP with bonded quinine. The

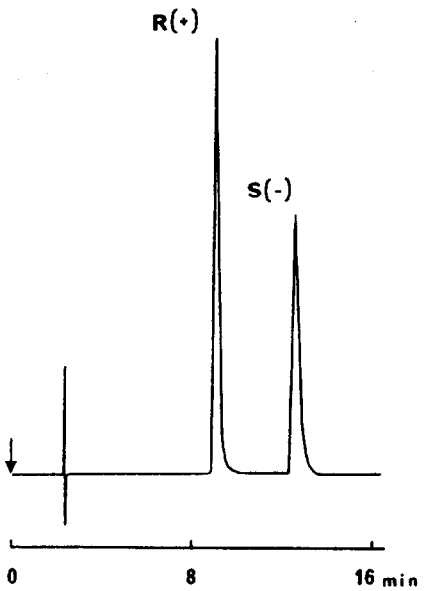


Fig. 5. Chromatographic separation of (*R*)- and (*S*)-TFAE on the quinine CSP. Mobile phase, carbon tetrachloride; flow-rate, 1 ml/min; UV detection at 365 nm.

best separation was achieved with carbon tetrachloride as mobile phase (Fig. 5).

This approach of correlating solvent characteristics could be useful for other CSPs that are thought to bind significantly via a hydrogen-bonding mechanism. A similar dependence of the enantioselectivity on the nature of solvent has already been observed in a study of asymmetric catalysis using alkaloids (quinine, ephedrine) covalently bonded to silica gel as a catalyst in the Michael reaction between conjugated cycloalkenones and aromatic thiols [23].

4. Conclusions

The solvent-induced effect on the chiral recognition of (*R*,*S*)-TFAE by a quinine CSP seems to be of great importance. The enantioselectivity is optimum with weakly polar, aprotic solvents as mobile phases. The presence of polar components in the mobile phase might change the enantioselectivity significantly. The results obtained confirmed the priority of the formation of a hydrogen bond between the chiral analyte TFAE and bonded quinine as CSP. The optimization of the mobile phase composition can be considered as an important means of improving the enantioselectivity of separation on other chiral stationary phases but with dominant hydrogen-bonding interactions for chiral recognition.

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