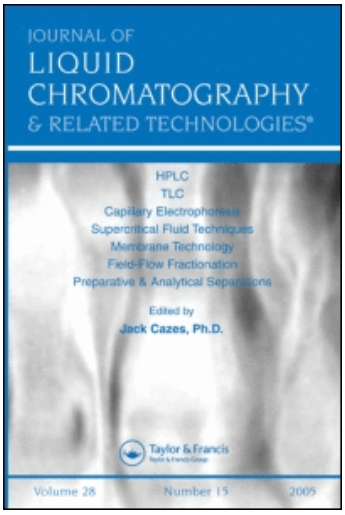

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RETENTION DEPENDENCE ON ORGANIC MODIFIER AND INTERACTION REAGENT CONCENTRATION IN REVERSED-PHASE ION-INTERACTION HPLC

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ABSTRACT

The effect played on ion-interaction chromatographic retention by the concentrations of the organic modifier and of the ion-interaction reagent as a function of the ionic strength is studied.

Experiments are performed for a series of analytes characterized by different chemical properties (nitrate, nitrite, iodide, ascorbic, orotic and p-aminobenzoic acids, aniline, benzylamine, p- and m-aminophenol) in the absence and in the presence of organic modifiers (acetonitrile and methanol) and of sodium perchlorate to control the ionic strength (I = 1.0 M).

Concentrations of the ion-interaction reagent (octylammonium o-phosphate) ranging between 1.0 and 60.0 mM and of the organic modifier (methanol and acetonitrile) ranging between 0 and 45% are considered.

Retention interdependence on the concentrations of ion-interaction reagent and of the methanol in the mobile phase is also studied.

The results are discussed and compared with literature data and the relevant role played on the retention by ionic strength is underlined.

INTRODUCTION

Many reversed-phase HPLC methods make use, in order to improve the chromatographic response, of ion-interaction reagents added to the mobile phase. The ion-interaction reagent forms with the analyte a ion-pair which, due to the increased lipophilic properties, is retained onto the reversed-phase surface; this process does not necessarily involve a modification of the stationary phase surface.

According to other hypotheses, the interaction reagent contained in the mobile phase is adsorbed onto the surface of the stationary phase through adsorption and electrostatic forces, giving rise to an electrical double-layer (1-4). The interaction properties of the originary reversed-phase packing material are therefore modified.

These techniques are generally refered as ion-interaction or ion-pair chromatography and they all make use of a reversed-phase stationary phase. As concerns the choice of the mobile phase, most of the methods utilize an organic-aqueous mixture which contains the interaction reagent, whilst a few others (5-8) make use of an aqueous solution of the interaction reagent.

Previous results obtained in this laboratory with mobile phases formed by aqueous solutions of different interaction reagents were in agreement with the hypothesis of a dynamic modification induced by

the flowing interaction reagent on the surface of the stationary phase. It can be anyway supposed that this modification does not involve the whole surface of the stationary phase (9-11).Two kinds of retention sites (12,13) therefore can simultaneously be present on the surface, namely conventional reversed-phase adsorption and ion-interaction sites produced in the dynamic modification.

The predominance in the retention process of the solute of ion-interaction or adsorption mechanism can be correlated (12) with the double-layer electrical potential and, through the Stahlberg equation (3), with the concentration of the ion-interaction reagent, in agreement with models recently proposed (14) by Zou and coworkers.Also the concentration of the organic modifier in the mobile phase plays an important role, due to the competition of the organic solvent for the column surface (15-18). Literature results, mostly obtained for aqueous-organic mobile phases, generally agree in observing a retention decrease when the concentration of the organic modifier in the mobile phase increases. Different behaviours were on the contrary observed as concerns the dependence of retention on the concentration of the interaction reagent.

In order to lead a further contribution in this study, in the present paper a systematic study is carried out about the retention dependence on organic modifier and ion-interaction reagent concentration. In order to separately investigate on the effects of the factors, the use of aqueous and hydro-organic mobile-phases is compared. Octylammonium o-phosphate at pH 6.4 is used as the interaction-reagent in a very wide concentration range (between 1.0 and 60.0 mM). The effect of ionic strength is also investigated, by comparing two series of experiments performed with and without the correction for constant ionic strength. Taking into account that also the chemical properties of the analytes can play a role, analytes characterized by different chemical properties were chosen and namely: nitrate, iodide, nitrite, ascorbic acid, orotic acid, aniline, benzylamine, phenylurea, ethylenethiourea, 1,4-aminobenzoic acid, 1,4-amino- and 1,3-amino-phenol.

MATERIALS

Apparatus

Analyses were carried out with a Merck-Hitachi Lichrograph chromatograph Model L-6200, equipped with a two-channel model D-2500 Chromato-integrator, interfaced with a UV-Vis detector model L-4200 and a L-3720 conductivity detector of the same firm.

A Metrohom 654 pH-meter equipped with a combined glass-calomel electrode was employed for pH measurements and a Hitachi mod.150-20 spectrophotometer for absorbance measurements.

Chemicals and Reagents.

Ultrapure water from Millipore Milli-Q was used for the preparation of solutions. Octylamine and orotic acid were Fluka analytical grade chemical and sodium nitrate, sodium nitrite, sodium iodide, ascorbic acid, aniline, benzylamine, phenylurea and ethylenthiourea were Merck analytical grade chemicals. Ortho-phosphoric acid was C.Erba chemical.

METHODS

Chromatographic conditions. Procedure.

A 5 μm ODS-2 Spherisorb Phase Separation column fully end-capped and with a carbon load of 12% (0.5 mM/g) was used, together with a guard pre-column Lichrospher RP-18 (5 μm).

The solutions to be used as the mobile phase were prepared by adding the amount of o-phosphoric acid required to obtain the desired pH value to the amount of octylamine and of organic solvent needed to

prepare the prefixed concentrations. The pH value so obtained in aqueous-organic solution is also reported as an "operational" pH value (19). The same procedure was followed for the preparations of solution at controlled ionic strength. A constant I =0.10 M ionic strength was realized, for addition of sodium perchlorate, which does not absorb at the used wavelengths.

The chromatographic system was conditioned by passing the eluent through the column until a stable baseline signal was obtained; a minimum of 1 hour was necessary. The use of a premixed water-organic eluent flowing in isocratic conditions permitted to achieve shorter equilibration times. Between the uses of different mobile phases and after use, the stationary phase was washed by flowing water (1.0 ml/min for 15 min) and then a 50/50 v/v water/methanol or water/acetonitrile mixture (1.0 ml/min for 15 min).

Dead times were evaluated for each series of experiments through injections of $NaNO_3$ solutions (20 ppm) and conductometric detection of the unretained Na^+.

Spectrophotometric detection at 230 nm was employed.

RESULTS AND DISCUSSION

Effect of organic modifier concentration.

Table I reports the capacity factors k' obtained for the analytes investigated at different concentrations of methanol and acetonitrile. For both the solvents was observed a retention decrease which follows an exponential shape which could be fitted (correlation coefficient r^2 always >0.95) by the mathematical function $y = A\ e^{-kx}$. Figure 1 reports the plots of the natural logarithms of the capacity factors as a function of the per cent concentration of methanol (figure 1A) and acetonitrile (figure 1B), which were fitted by straight lines.

The observed decrease in retention can be ascribed, besides to the increasing solvent eluotropic strength with the increasing organic concentration, to the effect played by the organic modifier on the

TABLE I

Capacity factors as a function of organic modifier % concentration.
Stationary phase:Phase Separation ODS-2 Spherisorb 5 μm endcapped.
Ion-interaction reagent: octylammonium o-phosphate 5 0 mM pH 6.4.
Spectrophotometric detection:230 nm.
Flow-rate 0.7 ml/min and 1.5 ml/min for phenylurea and aniline.

METHANOL

Analyte MeOH conc.	0%	5%	10%	15%	25%	35%	45%
Iodide	6.29	4.75	3.36	2.67	1.85	1.28	0.79
Nitrate	5.12	3.53	2.67	2.21	1.62	1.32	0.72
Nitrite	3.24	2.61	2.10	1.84	1.44	1.02	0.69
Ascorbic Acid	3.84	3.29	1.93	1.73	1.22	0.84	0.59
Benzylamine	0.72	0.57	0.47	0.34	0.25	0.24	***
Ethylenethiourea	0.84	0.58	0.43	0.38	0.25	0.21	***
Phenylurea	14.88	10.75	6.24	4.62	2.40	1.38	***
Aniline	14.25	7.16	5.07	3.80	2.44	1.58	***

ACETONITRILE

Analyte ACN conc.	0%	5%	10%	15%	25%
Iodide	6.29	3.65	2.80	1.76	0.68
Nitrate	5.12	2.60	1.92	1.28	0.44
Nitrite	3.24	1.93	1.57	1.04	0.37
Ascorbic Acid	3.84	1.65	1.14	0.79	0.32
Benzylamine	0.72	0.38	0.37	0.34	***
Ethylenethiourea	0.84	0.47	0.38	0.34	***
Phenylurea	14.88	6.58	3.85	2.30	0.89
Aniline	14.23	5.83	4.28	3.22	1.73

capacity of the modified surface of the stationary-phase, due to the competition which takes place for the surface itself between the organic modifier and the ion-interaction reagent.

Since the results of table I and figure 1 do not show any easy correlation between retention and chemical properties of the analytes investigated, the observed retention decrease could likely be ascribed more to the varied interaction properties of the moiety adsorbed onto the surface than to the analyte characteristic properties.

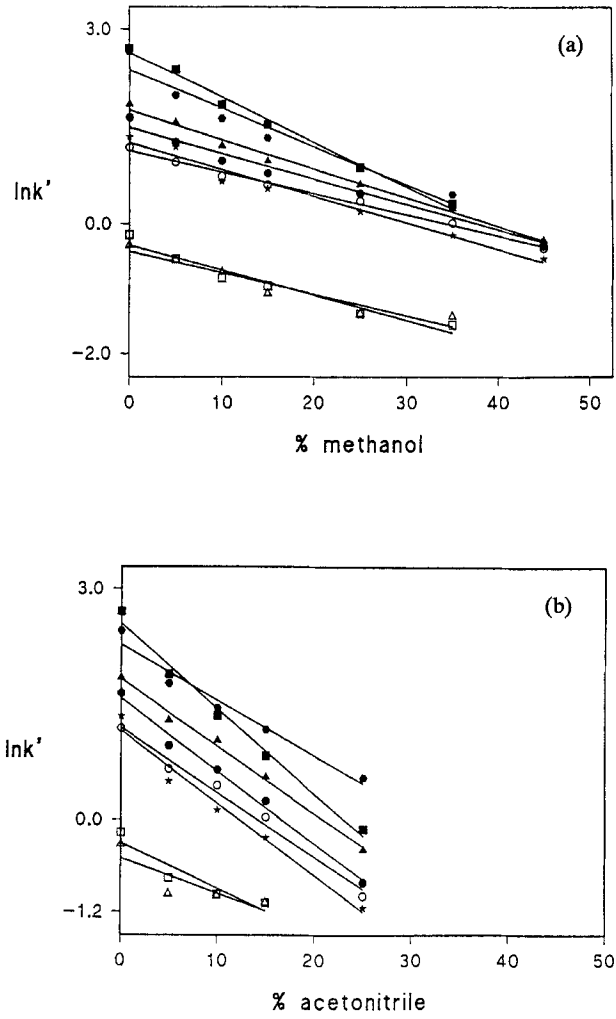


FIGURE 1 ln of capacity factor as a function of organic modifier % concentration: A-methanol, B-acetonitrile.
Stationary phase: Phase Separation ODS-2 Spherisorb 5µm endcapped. Ion-interaction reagent: octylammonium o-phosphate 5.0 mM pH 6.4. Spectrophotometric detection 230 nm. Flow-rate 0.7 ml/min and 1.5 ml/min for phenylurea and aniline.
Analyte: ▲-iodide; ● -nitrate; ○ -nitrite; ★ -ascorbic acid; - △ benzylamine; ● - aniline; □ -ethylenethiourea; ■ -phenylurea.

Effect of ion-interaction concentration

A general disagreement is reported in literature as concerns the retention dependence on ion-interaction concentration. The retention dependence on ion-interaction concentration was shown (9,20-22) to be a function of the charge of the analyte, which in turn can affect the double-layer electrical potential. It follows that with increasing ion-interaction reagent concentration, a retention decrease can be observed when the charges of the analyte and of the ion-pairing agent are of the same sign (2), no retention dependence for uncharged analytes (9,22) and a retention increase when the charges of ion-pairing and solutes are of different sign (9,22).

When retention increases with the ion-interaction reagent concentration, different behaviours were observed and namely: a linear increase (21,23), a shape which reaches a plateau (24,28) or a parabolic dependence (5,9,11,29-35).

In order to consider the effect of the concentration of the ion-interaction reagent independently on the concentration of the organic solvent, we carried out this study by using as the mobile phase an aqueous solution of the interaction reagent.Furthermore, to study the retention behaviour in the most general conditions, a wide concentration range (between 1.0 and 60.0 mM) of the ion-interaction reagent and analytes characterized by different chemical properties were considered.

The capacity factors obtained are plotted in figure 2 as a function of the ion-interaction reagent concentration. Generally (a part p-aminophenol and benzylamine which show a small retention decrease with the increase of the ion-interaction reagent concentration) the shape of the capacity factors vs. concentration shows a maximum, whose entity and position varies for the different analytes investigated.

The comparison of our results with literature data show that the behaviour here obtained with aqueous mobile phase is comparable with the data obtained with hydro-organic mobile phases and, more important point, that the literature disagreement is only apparent, because it depends on the range of concentration explored. The

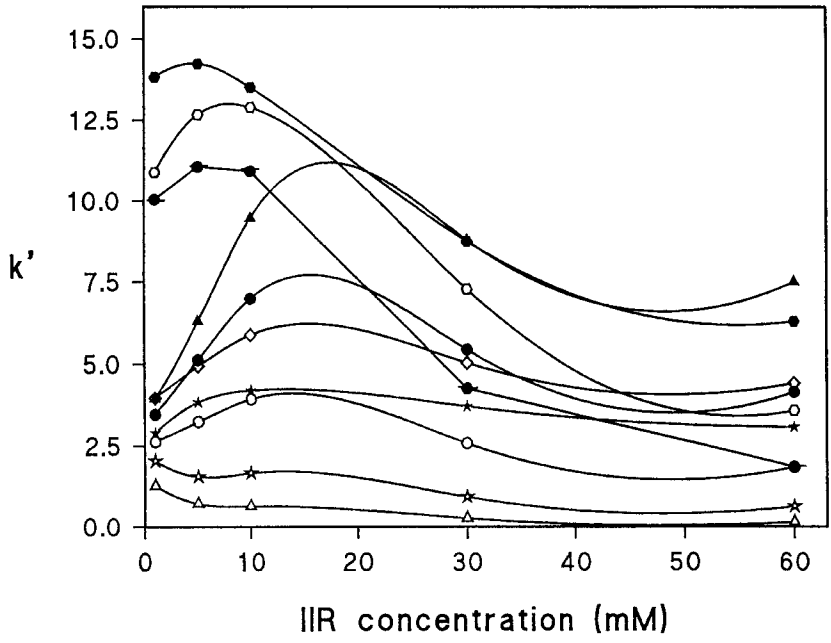


FIGURE 2 Capacity factor as a function of ion-interaction reagent (IIR) concentration.
Chromatographic conditions as in figure 1.
Analyte: ▲-iodide; ●-nitrate; ○-nitrite; ★-ascorbic acid; △ benzylamine; ⬣-aniline; ☆ p-aminophenol; ◇-m-aminophenol; ⬡p-aminobenzoic acid; —●— orotic acid.

complete behaviour follows in fact a parabolic shape and the other dependencies experimentally observed (21, 24-28) hold only for narrow concentration ranges.

The portion of the curve to the left of the maximum can be easily explained, in agreement with literature: the stationary phase surface is becoming more ionogenic due to the adsorption of hetaerons onto the surface and the overall energy increases due to ion-ion interactions. A maximum of retention is then reached in correspondence of the surface capacity (maximum adsorption capability) of the stationary phase. Since retention also depends on the chemical properties of analytes

and on their ability to form ion-pairs, the parabolic shapes are different for the different analytes.

The problem with all the existing theories seems to lie to the right of the curve maximum, also observed for bare-silica (35) and polymer-based (17,29) stationary-phases. Why the decrease in retention?

Decrease has been ascribed to the formation in the mobile phase of micelles and to the taking place of competitive adsorption reactions when the concentration of the interaction reagent exceeds the critical micelle concentration (11,30,31). But if retention depended on the critical micelle concentration, the retention dependence for the same chromatographic conditions would have to be similar for all the analytes (36) and the maximum of retention would correspond to the critical micellar concentration (CMC) value of the interaction reagent (40). These conditions are not verified for the data reported in figure 2. As concerns the CMC range, literature does not report the value for octylammonium ortho-phosphate but the CMC for octylammonium chloride (37) is available and equal to 90 mM, which is a very higher concentration than those at which we observe the maxima of retention (5-10 mM).

Another hypothesis ascribes the retention decrease to the increase of counter-ion concentration (29,34,36). Counter-ions can exert a shielding effect towards the accessibility for the analytes to the stationary-phase surface and can also remarkably affect the double-layer potential. In agreement with this suggestion, phosphate concentration, which in our experiments increases with the increasing octylammonium concentration, could be responsible for these effects.

It must be anyway taken into consideration that in the above experimental conditions, in which the ion-interaction reagent concentration varies from 1.0 to 60.0 mM, the total ionic strength of the mobile phase remarkably varies. In order to verify if the retention behaviour as a function of the ion-interaction reagent concentration and in particular if the retention decrease observed for the higher concentrations could depend on the ionic strength variations, a series of experiments (below reported) was also performed, in which the total

ionic strength in the mobile phase was kept constant (at concentration of 0.10 M) for addition of sodium perchlorate.

The effect of the organic modifier concentration on the retention dependence on the ion-interaction concentration.

In order to collect further information about the roles played on the retention by the ion-interaction reagent and the organic modifier concentrations, three new series of experiments were performed for concentrations of octylammonium phosphate ranging between 3.0 and 25.0 mM, at prefixed methanol concentrations of 5, 15 and 45%. The values obtained for the capacity factors are presented in table III and in figures 3 and 4. The shapes clearly show as the retention dependence on the ion-interaction concentration also depends on the methanol concentration, as on the other hand theorectically predicted by Horvath (5). The following considerations can be drawn out. A decrease in the curve maxima universally occurs upon the addition of organic modifier.Then, the shape of the curves about the maxima becomes sharper and more pronounced , even while the overall k' values decrease from neat aqueous solutions. Moreover, for some ions (nitrite, nitrate, iodide) the maxima of the curve shift to the left (lower ion-interaction concentration) with increasing organic, while for phenylurea and ascorbic acid the maxima remain approximately constant. Aniline shifts left but to an intermediate degree. In all the instances, the retention of ionic solutes with the increasing of hetaeron concentration, initially rises, reaches a maximum and then falls.

The decrease of the dielectric constant induced by the increasing of the organic content will reduce the surface concentration of hetaerons with the resulting loss in retention. In agreement, the hydrophobic portions of solute molecules will diminish in retention, resulting in more pronounced slopes for the hydrophobic ions with respect to the harder inorganic ions. The trend is evident in figure 3 and 4 where the iodide, nitrate and nitrite ions decrease by ca. 30-50% from

TABLE II

Capacity factor as a function of ion-interaction reagent concentration.
Stationary phase: Phase Separation ODS-2 Spherisorb 5 μm endcapped.
Ion-interaction reagent: 5.0 mM octylammonium o-phosphate pH 6.4.
Spectrophotometric detection: 230 nm. Flow-rate 0.7 ml/min.

	ion-interaction reagent concentration				
Analyte	**1.0 mM**	**5.0 mM**	**10.0 mM**	**30.0 mM**	**60.0 mM**
Iodide	3.95	6.29	9.47	8.79	7.50
Nitrate	3.46	5.12	6.98	5.43	4.16
Nitrite	2.63	3.24	3.93	2.58	1.86
Ascorbic acid	2.90	3.84	4.20	3.71	3.09
Benzylamine	1.28	0.72	0.64	0.26	0.14
Aniline	13.83	14.23	13.51	8.75	6.29
p-aminophenol	2.04	1.56	1.67	0.93	0.63
m-aminophenol	3.97	4.93	5.87	5.03	4.43
p-aminobenzoic acid	10.89	12.67	12.89	7.27	3.59
Orotic Acid	10.05	11.07	10.93	4.26	1.88

TABLE III

Capacity factor as a function of ion-interaction and methanol concentration.
Ion-interaction reagent: 5.0 mM octylammonium o-phosphate pH 6.4.
Stationary phase: Phase Separation ODS-2 Spherisorb 5μm endcapped.
Spectrophotometric detection 230 nm.
Flow-rate 0.7 ml/min, for phenylurea and aniline 1.5 ml/min.

Analyte	**5% 3 mM**	**15% 3mM**	**45% 3 mM**	**5% 5 mM**	**15% 5 mM**	**45% 5 mM**	**5% 10 mM**	**15% 10 mM**	**45% 10 mM**	**5% 25 mM**	**15% 25 mM**	**45% 25 mM**
Iodide	3.39	2.16	0.51	4.67	2.62	0.77	4.15	2.60	0.82	5.01	2.59	0.84
Nitrite	2.17	1.63	0.47	2.56	1.80	0.67	2.64	1.64	0.73	2.02	1.41	0.72
Nitrate	2.78	1.80	0.50	3.67	2.17	0.70	3.29	2.12	0.77	3.18	2.07	0.78
Ascorbic acid	2.20	1.51	0.44	2.61	1.70	0.57	2.68	1.46	0.64	1.77	1.21	0.64
Phenylurea	9.69	4.53	0.99	10.02.	4.27	***	9.00	4.61	0.95	9.39	4.72	0.96
Aniline	7.81	4.29	1.21	7.16	3.80	***	6.46	3.78	1.15	5.48	3.31	1.08

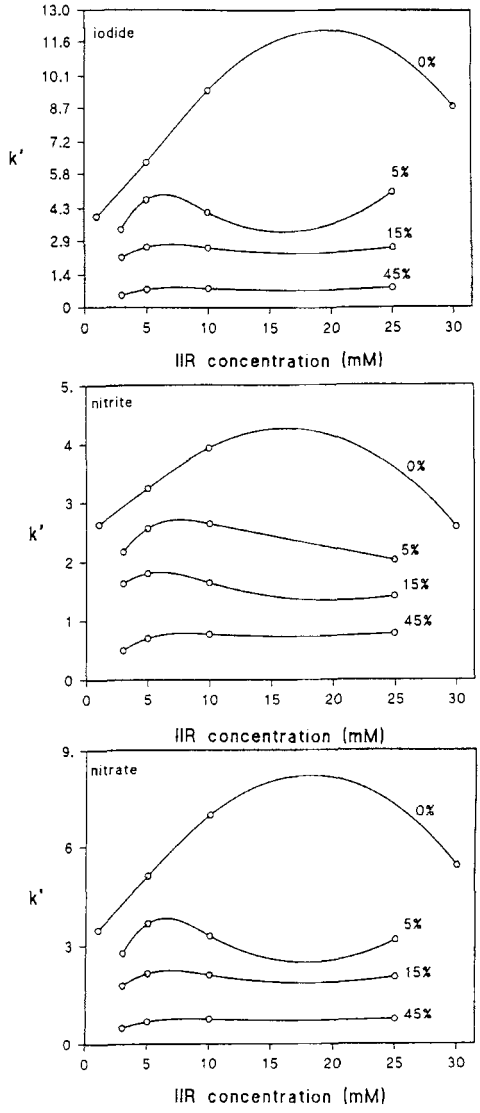


FIGURE 3 Capacity factor as a function of ion-interaction reagent (IIR) concentration for different methanol % concentrations. Chromatographic conditions as in Fig.1. Analytes: iodide, nitrate and nitrite.

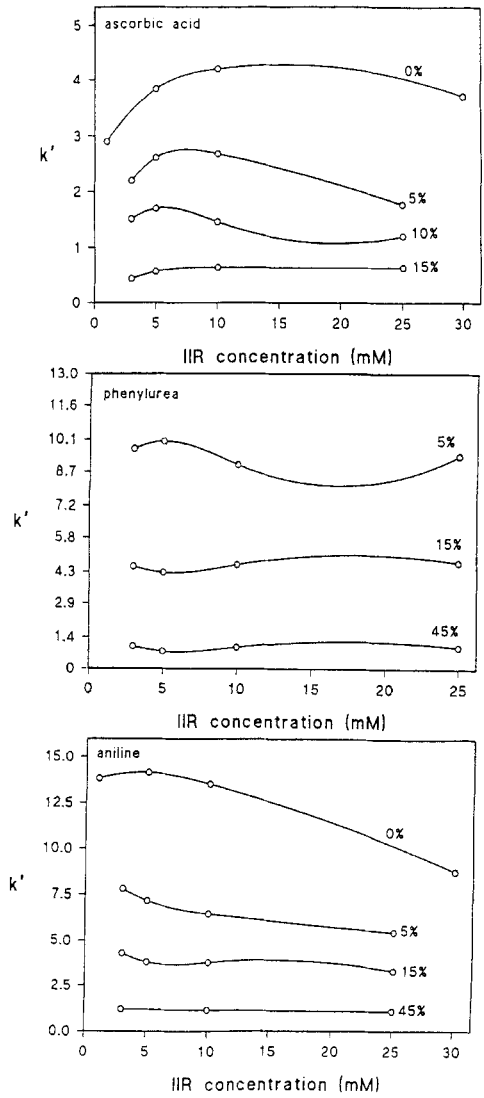


FIGURE 4 Capacity factor as a function of ion-interaction reagent (IIR) concentration for different methanol % concentrations. Chromatographic conditions as in Fig.1. Analytes: aniline, phenylurea and ascorbic acid.

0 to 55% of methanol, while the bulkier ions decrease by 50% or more from 5 to 10% of methanol.

Here again, a decreasing retention to the right of the maximum for the curves of figures 3 and 4 can be observed.

The curves are progressively flatting with the increasing methanol concentration and for the highest methanol concentrations investigated, the retention is practically independent on ion-interaction reagent concentration.

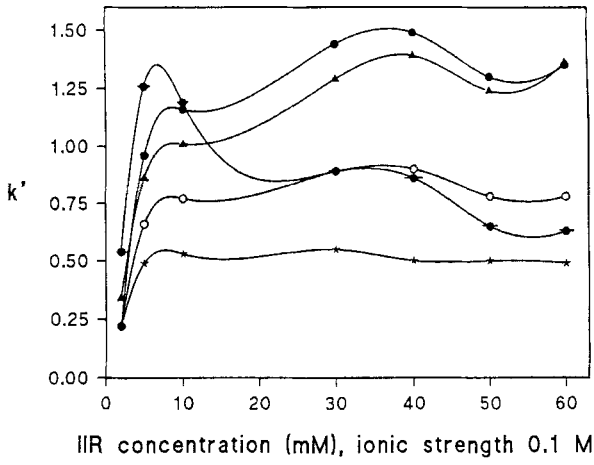
The effect of the ionic strength on the retention dependence on the ion-interaction concentration.

On the basis of the above considerations, a series of experiments were performed at constant ionic strength I (I= 0.10 M for sodium perchlorate). The results obtained for concentration of octylammonium o-phosphate ranging between 2 and 60 mM, are reported in table IV and in figure 5. It must be said that in general the presence of sodium perchlorate strongly decreases the retention and this can be explained through a shielding effect which the added electrolyte exerts towards the accessibility for the solute of the active sites present on the stationary-phase surface. The general decrease of retention did not

TABLE IV

Capacity factor as a function of ion-interaction reagent concentration, at constant ionic strength 0.10 M ($NaClO_4$).
Stationary phase: Phase Separation ODS-2 Spherisorb 5 μm, endcapped.
Ion-interaction reagent: 5.0 mM Octylammonium o-phosphate, pH=6.4.
Spectrophotometric detection: 230 nm. Flow-rate: 0.7 ml/min.

	ion-interaction reagent concentration						
Analyte	**2.0 mM**	**5.0 mM**	**10 mM**	**30 Mm**	**40 mM**	**50 mM**	**60 mM**
Iodide	0.34	0.86	1.01	1.29	1.39	1.24	1.36
Nitrate	0.22	0.96	1.16	1.44	1.49	1.30	1.32
Nitrite	0.22	0.66	0.77	0.89	0.90	0.78	0.78
Ascorbic acid	0.22	0.49	0.53	0.55	0.50	0.50	0.49
Aniline	10.81	10.24	8.66	6.93	6.74	5.46	5.45
Orotic Acid	0.54	1.26	1.19	0.89	0.86	0.65	0.63



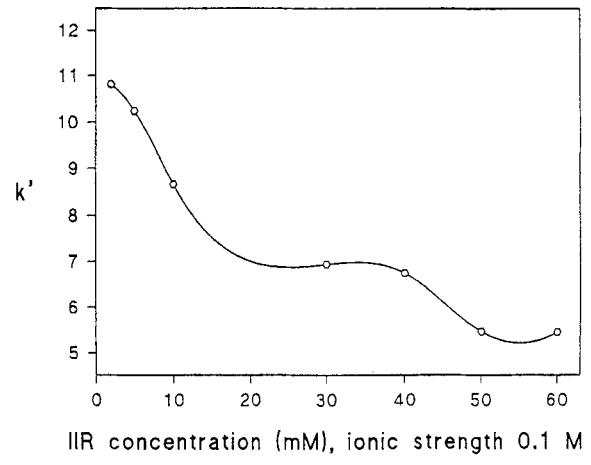


FIGURE 5 Capacity factor as a function of ion-interaction reagent (IIR) concentration at constant ionic strength I=0.10 M for $NaClO_4$. Chromatographic conditions and symbols as in figures 1 and 2.

allow us to investigate all the analytes before considered, because some of them, under the experimental conditions used, do not show appreciable retention times. The retention decrease due to the addition of the electrolyte ranges between 50% and 79% for nitrate, iodide, nitrite, orotic and ascorbic acids, while is very lower (maximum 20%) for aniline. This may be due to the more lipophilic properties of aniline and to the predominance in the retention process of partition mechanism with respect to ion-interaction mechanism.

As concerns the dependence of the retention on the ion-interaction concentration, nitrite, nitrate, iodide and ascorbic acid show an initial increase and then a plateau behaviour. On the contrary orotic acid and aniline show a characteristic and similar shape with the presence of two different maxima and a general decrease in the curve to the right of the major maximum.

As a result, the retention decrease observed for the higher ion-interaction reagent concentrations (table II, figure 2) can be ascribed to the uncontrolled ionic strength only as concerns the behaviour of the more hydrophilic ions. The addition of the electrolyte seems on the contrary not to significantly affect the behaviour of the more lipophilic species, such as aniline and orotic acid. A different retention mechanism, in which adsorption forces predominate on electrostatic ones, could perhaps be hypothesized for these species.

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