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Critical comparison of retention models for optimisation of the separation of anions in ion chromatography III. Anion chromatography using hydroxide eluents on a Dionex AS11 stationary phase

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Abstract

Three ion chromatography (IC) retention models, namely the linear solvent strength model (LSSM), empirical end points model (EEPM) and three-point curve fitting using DryLab from LC Resources were evaluated in terms of their ability to predict retention factors for inorganic anions separated on a Dionex AS11 column using electrolytically generated hydroxide eluents. Extensive experimental retention data were gathered for 21 anions (fluoride, acetate, formate, bromate, chloride, nitrite, methanesulfonate, bromide, chlorate, nitrate, iodide, thiocyanate, succinate, sulfate, tartrate, oxalate, tungstate, phthalate, chromate, thiosulfate and phosphate) using hydroxide eluents of varying concentration. Although the purely theoretical LSSM was found to give adequate performance, the EEPM (in which a linear relationship is assumed between the logarithm of retention factor and the logarithm of eluent strength, but the slope is determined empirically) and DryLab performed better, with DryLab giving the best accuracy and precision of the three models. The EEPM and DryLab were also shown to have advantages in terms of their low knowledge requirements and ease of solution. Compared with IC using dual eluent species, the retention behaviour in IC using single eluent species was found to be easier to model by both theoretical and empirical approaches. © 1999 Elsevier Science B.V. All rights reserved.

Keywords: Retention models; Mobile phase composition; Computer-assisted chromatography; Hydroxide

1. Introduction

The optimal eluent composition in ion chromatography (IC) can be selected by using computer-assisted interpretive optimisation procedures that are based on a retention model providing a mathematical relationship for calculating the retention factor of an analyte under differing eluent conditions. The predictive ability of the retention model is the most important consideration in the success of the optimisation procedure. Thus, we have undertaken a systematic evaluation of IC retention models to choose the best model to include in developing a robust optimisation software package.

In two previous papers [1,2] we have compared the performance of seven theoretical retention models for predicting the retention factors of inor-

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ganic anions in non-suppressed IC (using phthalate eluents on three different stationary phases) and in suppressed IC (using carbonate–bicarbonate eluents on a Dionex AS4A stationary phase). The models studied were the linear solvent strength model [3–5] (which should not be confused with the linear solvent strength model used in reversed-phase liquid chromatography [6]), including the dominant equilibrium approach and the competing ion effective charge approach for eluents containing two competing ions, the dual eluent species model [7–9] (based upon the Hoover model [1,10]), Kuwamoto model [11], extended dual eluent species model [12], and the multiple species eluent/analyte model [13]. These studies showed that none of the theoretical models was sufficiently rugged to permit reliable prediction of retention factors, but that the retention data showed good linearity for a plot of log k' versus log [eluent], even when there were two competing ions present in the eluent. However, the slope of this relationship could not be predicted reliably from theory. In view of this, we have proposed that the best prediction of retention factors using minimal experimentation can be achieved by measuring retention factors for each analyte at several extremes of eluent composition (i.e., extremes of pH or concentration) and then interpolating retention factors at intermediate compositions by assuming a linear relationship between log k' and log [eluent]. This approach was referred to as an ''empirical end-points model''.

As indicated above, our previous studies were directed towards retention modelling for eluents with multiple competing ions since such eluents were in routine use for the vast majority of IC separations. Eluents with a single competing ion, such as hydroxide, have not found widespread use in the past because retention of analytes can be manipulated only by varying the concentration of the competing ion, rather than by varying the relative concentrations of two competing ions of different strength. This can be achieved easily using multiple species eluents such as phthalate–hydrogenphthalate mixtures or carbonate–bicarbonate mixtures. A further problem encountered with the use of hydroxide eluents is difficulty in preparing solutions of exact concentrations because of varying amounts of carbonate introduced by contamination with atmospheric carbon dioxide.

In recent months the development of an electrolytic eluent generator (Dionex EG40) has led to an increased interest in hydroxide eluents because there is no carbonate contamination and the hydroxide concentration can be controlled accurately through the current used in the electrolysis reaction. Reliable isocratic and gradient elution separations can be performed with this system. In view of the increased likelihood of more widespread use of hydroxide eluents and the consequent need to develop computer optimisation methods for these eluents, we have evaluated the reliability of retention models for use with hydroxide eluents.

2. Theory

Since the eluent contains only a single competing ion and the eluent pH is fixed, the linear solvent strength model (LSSM) is applicable. It should be noted that the other models listed earlier are designed for use with multiple eluent species and when applied to the case of a single eluent ion they can be simplified to predict the same retention behaviour as that by the LSSM. Eq. (1) describes the retention behaviour when hydroxide is used as an eluent:

$$\log k'_{\mathrm{A}} = C_1 - \frac{x}{y}\log[\mathrm{OH}^-] \qquad (1)$$

where x is the charge on the analyte anion, y is the charge on the eluent anion (-1 for hydroxide), k'_{A} is the retention factor and C_1 is a constant. This equation predicts a linear relationship between the logarithm of the retention factor and the logarithm of the eluent concentration. Furthermore, the slope of this linear relationship is equal to the negative ratio of the analyte and eluent charges.

The empirical end points model (EEPM) developed for multiple eluent competing ions can be easily simplified for the case of single eluent systems. The model is given by the following equation for the case of hydroxide as the only eluent species present:

$$\log k'_{\mathrm{A}} = C_1 + C_2 \log[\mathrm{OH}^-] \qquad (2)$$

The two chromatographic constants C_1 and C_2 can be solved numerically using a set of simultaneous equations. It can be seen that both the LSSM and the

EEPM predict the same linear relationship between the logarithms of the retention factor and the eluent concentration, but the slope of this relationship is determined theoretically in the former case and experimentally in the latter.

For single eluent systems there is a commercially available product that can be used for the prediction of retention times [14]. DryLab from LC Resources fits a quadratic term to the logarithms of the retention factor and concentration of the eluent, see Eq. (3). This is a similar approach to the empirical end points model, but has the quadratic fit has the potential for more accurate results. The main disadvantage to the use of DryLab is the requirement for one additional experiment over the end-points model and its inability to be used for multiple-species eluent systems where both eluent concentration and ratios are to be optimised simultaneously

$$\log k'_A = A + B \log[OH^-] + C(\log[OH^-])^2 \quad (3)$$

DryLab solves for the three chromatographic constants A–C numerically using a set of simultaneous equations.

3. Experimental

3.1. Instrumentation

The chromatographic instrumentation consisted of a Dionex DX-500 ion chromatograph (Dionex, Sunnyvale, CA, USA), consisting of an IonPac AS11 anion separator column (250×4 mm), an IonPac ATC-1 anion trap column and a ASRS-II self regenerating suppressor housed in an LC30 chromatography oven at 30°C, a Model EG40 Eluent Generator, a Model AS40 AutoSampler, a Model ED40 electrochemical detector operated in the conductivity mode and a Model GP40 gradient pump. The injection loop was 25 μl. All samples were analysed in duplicate with a flow-rate of 1.0 ml/min. The DryLab software was obtained from LC Resources (Walnut Creek, CA, USA).

3.2. Reagents and procedures

Eluents were prepared using purified water from a Milli-Q system (Millipore, Bedford, MA, USA) containing a 0.45-μm filter at the outlet. Sample solutions of fluoride, acetate, formate, bromate, chloride, nitrite, methanesulfonate, bromide, chlorate, nitrate, iodide, thiocyanate, succinate, sulfate, tartrate, oxalate, tungstate, phthalate, chromate, thiosulfate and phosphate were prepared by dissolution of analytical grade salts in the sodium form. Formate, nitrate, chromate, chloride, bromate, phthalate and nitrite were supplied from Ajax (Auburn, Australia), iodide, chlorate, phosphate, thiosulfate and tungstate from BDH Chemicals (Kilsyth, Australia), tartrate and oxalate from Mallinckrodt (Paris, KY, USA), fluoride and sulfate from Prolabo (Paris, France), bromide and methanesulfonate from Sigma (St. Louis, MO, USA) and thiocyanate and succinate from Aldrich (Milwaukee, WI, USA). The concentrations of the anions varied from 0.5 mg/l to 10 mg/l. The samples were analysed in duplicate at seventeen eluent concentrations ($[OH^-]$=3.720, 4.340, 4.960, 5.580, 6.200, 7.204, 8.438, 9.653, 11.01, 12.20, 13.45, 15.87, 18.23, 20.83, 23.37, 25.67 and 27.84 mM). If duplicate measurements were found to be inconsistent (to three decimal places), then further measurements were taken to ensure repeatability. The dead time of the column was determined by injecting a sample of deionised water into the column and measuring the retention time of the negative water peak.

Statistical analyses of the performance of the retention models were carried out using retention data acquired directly from the system described below. All calculations were performed using Microsoft Excel 97 on a Pentium MMX 200 computer with 64 MB of SDRAM, running Windows NT workstation v4.0 sp3.

3.3. Methods for solution of models

Only one experimental data point was required to solve the parameter C_1 for the LSSM, which could be calculated by rearranging Eq. (1). Values for k'_A and $[OH^-]$ for each analyte were obtained from experimental data with an eluent concentration of 5.58 mM.

Two experimental data points were required to solve for the two chromatographic constants C_1 and C_2 in the EEPM. The chromatographic constants were determined using the following equations:

Table 1
Retention times (min) for 21 analytes using a Dionex EG40 eluent generator to create hydroxide eluents at concentrations shown on a Dionex AS11 IonPak column with an eluent flow-rate of 1.00 ml/min (the void time was 1.42 min)

	[OH−] (m*M*)																
	3.72	4.34	4.96	5.58	6.20	7.20	8.44	9.65	11.01	12.20	13.45	15.87	18.23	20.83	23.37	25.67	27.84
Fluoride	1.70	1.65	1.62	1.60	1.58	1.56	1.55	1.53	1.50	1.50	1.50	1.49	1.47	1.46	1.45	1.45	1.45
Acetate	1.76	1.71	1.67	1.64	1.62	1.59	1.57	1.55	1.53	1.52	1.51	1.50	1.49	1.48	1.48	1.47	1.46
Formate	1.90	1.82	1.77	1.73	1.69	1.66	1.62	1.59	1.57	1.56	1.55	1.53	1.51	1.50	1.49	1.48	1.48
Methansulfonate	1.99	1.91	1.84	1.80	1.76	1.71	1.67	1.63	1.62	1.58	1.58	1.54	1.52	1.51	1.50	1.49	1.49
Bromate	2.69	2.49	2.36	2.27	2.18	2.09	2.00	1.92	1.85	1.81	1.78	1.73	1.67	1.65	1.62	1.60	1.60
Chloride	2.91	2.68	2.52	2.41	2.30	2.18	2.09	2.00	1.92	1.87	1.84	1.78	1.72	1.69	1.67	1.63	1.62
Nitrite	3.40	3.09	2.88	2.74	2.60	2.45	2.31	2.19	2.09	2.03	1.98	1.90	1.84	1.79	1.76	1.71	1.70
Bromide	5.61	4.96	4.52	4.24	3.94	3.63	3.34	3.07	2.89	2.74	2.63	2.43	2.31	2.22	2.13	2.06	2.03
Chlorate	6.30	5.51	5.01	4.70	4.34	3.98	3.65	3.35	3.12	2.96	2.83	2.62	2.46	2.35	2.25	2.17	2.13
Nitrate	5.96	5.25	4.77	4.47	4.14	3.79	3.49	3.21	2.99	2.84	2.72	2.53	2.38	2.28	2.20	2.11	2.06
Iodide	26.10	22.17	19.65	18.23	16.21	14.43	12.78	11.28	10.20	9.36	8.72	7.65	6.86	6.31	5.84	5.39	5.14
Thiocyanate	–	–	–	36.32	32.08	28.43	24.89	21.78	19.48	17.84	16.52	14.29	12.74	11.56	10.65	9.60	9.08
Succinate	15.18	11.31	8.96	7.50	6.24	5.04	4.14	3.41	2.95	2.65	2.44	2.15	1.97	1.86	1.76	1.69	1.65
Tartrate	18.73	13.82	10.88	8.98	7.48	5.94	4.80	3.88	3.30	2.96	2.68	2.32	2.09	1.95	1.83	1.74	1.70
Sulfate	26.77	19.54	15.21	12.48	10.25	7.99	6.32	4.96	4.15	3.62	3.24	2.69	2.37	2.18	2.01	1.88	1.82
Oxalate	32.90	23.95	18.64	15.32	12.41	9.67	7.58	5.92	4.86	4.19	3.73	3.05	2.64	2.39	2.17	2.02	1.94
Tungstate	58.27	42.05	32.42	26.42	21.26	16.30	12.53	9.58	7.69	6.53	5.65	4.40	3.67	3.21	2.81	2.53	2.38
Phthalate	83.00	59.83	45.88	37.20	29.99	22.72	17.27	13.06	10.41	8.63	7.44	5.63	4.59	3.97	3.40	3.00	2.78
Chromate	–	–	–	58.60	46.99	35.29	26.78	20.02	15.66	12.97	10.99	8.18	6.45	5.46	4.58	3.93	3.59
Thiosulfate	–	–	–	51.23	40.57	30.63	23.28	17.37	13.67	11.33	9.63	7.17	5.77	4.86	4.10	3.55	3.25
Phosphate	157.8	110.2	82.60	66.72	50.62	37.02	26.81	18.97	14.34	11.42	9.46	6.58	4.98	4.00	3.33	2.83	2.57
System (CO_3)	17.67	13.18	10.47	8.72	7.30	5.82	4.73	3.83	3.29	2.93	2.68	2.32	2.09	1.95	1.85	1.75	1.71

$$C_2 = \log\left(\frac{k'_{A_1}}{k'_{A_2}}\right) / \log\left(\frac{[OH^-]_1}{[OH^-]_2}\right) \quad (4)$$

$$C_1 = \log k'_{A_1} - C_2 \log[OH^-]_1$$

Values for k'_A and $[OH^-]$ for each analyte were obtained from experimental data with eluent concentrations of 5.58 and 25.67 m*M*.

Three experimental data points were required to use the three-point model of DryLab. Values for the retention times, void time and $[OH^-]$ were entered into the program from experimental data with eluent concentrations of 5.58, 12.20 and 25.67 m*M*.

4. Results

4.1. Prediction of retention times using the models

The retention data obtained for the range of eluent concentrations and analytes studied are shown in Table 1. The parameters for each model were determined for a specific analyte anion using a single data point for the LSSM, two data points for the EEPM and three data points for DryLab (see Experimental). The models were then used to predict retention data for all 21 analytes using each of the eluent compositions in the data set. This gave a total of $21 \times 17 = 357$ predicted retention times for each model. Table 2 shows a typical set of results for sulfate and lists the measured retention times for each eluent composition, together with those predicted by each model. The experimental data are also plotted as log k' versus log $[OH^-]$ in Fig. 1. The excellent R^2 value of 0.99979 and slope of -2.056 highlight the almost ideal behaviour of these data, as expected from an eluent of such high purity.

From these data the percentage normalised difference (% d_i) between the measured and predicted retention times for a particular ion (i) were calculated using the following equation:

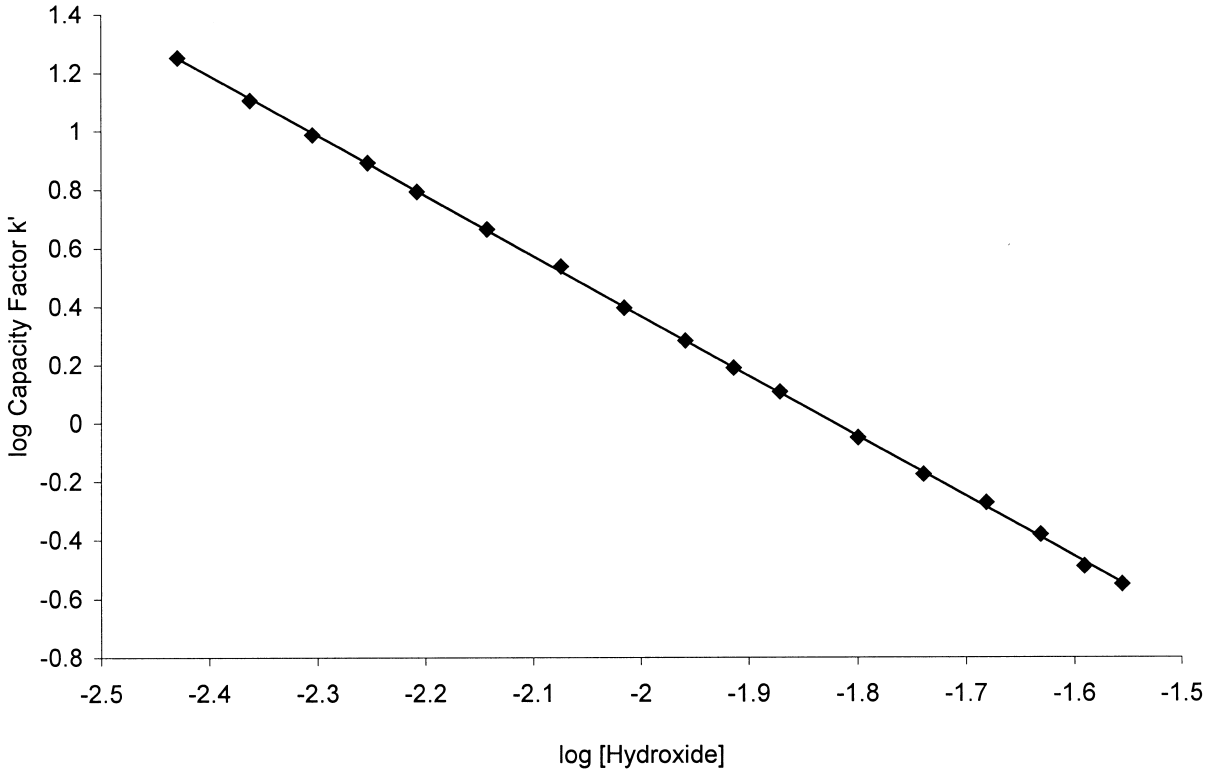


Fig. 1. Plot of log retention factor vs. log hydroxide concentration for sulfate on a Dionex IonPac AS11 stationary phase (R^2 value 0.99979, slope = −2.056).

Table 2
Experimentally measured retention times (Exp.) and predicted retention times: linear solvent strength model (LSS), empirical end-points model and three-point model of DryLab for sulfate on a Dionex IonPak AS-11 column with hydroxide as the only eluent

Eluent concentration (m*M*)	Retention time (min)			
	Exp.	LSS model	EEP model	DryLab
3.72	26.77	26.31	27.16	–[a]
4.34	19.54	19.70	20.09	–[a]
4.96	15.21	15.42	15.56	15.52
5.58	12.48	12.48	12.48	12.48
6.20	10.25	10.38	10.30	10.32
7.20	7.99	8.06	7.91	7.96
8.44	6.32	6.26	6.09	6.13
9.65	4.96	5.12	4.95	4.99
11.01	4.15	4.26	4.11	4.14
12.20	3.62	3.74	3.59	3.62
13.45	3.24	3.32	3.19	3.22
15.87	2.69	2.79	2.67	2.70
18.23	2.37	2.46	2.36	2.38
20.83	2.18	2.21	2.13	2.15
23.37	2.01	2.05	1.98	1.99
25.67	1.88	1.94	1.88	1.89
27.84	1.82	1.86	1.81	1.82

[a] No data were predicted for these eluent compositions.

$$\%\, d_i = \frac{t_{R_{act}} - t_{R_{pred}}}{t_{R_{act}} + t_{R_{pred}}} \cdot 2 \cdot \frac{100}{1} \qquad (5)$$

Table 3 shows the values of % d_i for the data given in Table 2, i.e., for the elution of sulfate. If the values of % d_i are now averaged for a particular ion (to give % d_{av}), graphical representations of the performance for each model can be obtained, as shown in Fig. 2.

4.2. Overall results for all analytes

Data similar to Tables 2 and 3 could be generated for each of the 21 analytes, however the volume of data necessitates the use of a statistical approach in order that trends may be identified. The statistical operations carried out on these data are the same as those carried out for the non-suppressed and suppressed data reported previously [1,2] in order that a direct comparison can be made with these earlier studies.

The accuracy of the models for all analytes using various hydroxide concentrations can be assessed by the global average of the percentage normalised differences (% d_{glob}), which should be zero. The precision for each model at various hydroxide concentrations for all analytes is given by the standard deviation of the percentage normalised differences [$SD_{(\%\ d)}$] which should be equal to the error of the experimental data. From the same data the correlation coefficient can be calculated, which should be unity.

Table 3
Normalised percentage differences (% d_i) between the measured and predicted retention times: linear solvent strength model (LSS), empirical end-points model and three-point model of DryLab for sulfate on a Dionex IonPak AS-11 column with hydroxide as the only eluent

Eluent concentration (m*M*)	Normalised % Difference (% d_i)		
	LSS Model	End Points Model	DryLab
3.72	1.752	−1.462	–[a]
4.34	−0.830	−2.784	–[a]
4.96	−1.357	−2.253	−2.018
5.58	0.000	0.000	0.000
6.20	−1.247	−0.486	−0.681
7.20	−0.808	0.951	0.376
8.44	1.011	3.677	3.052
9.65	−3.085	0.208	−0.603
11.01	−2.697	1.059	0.241
12.20	−3.139	0.868	0.000
13.45	−2.514	1.638	0.619
15.87	−3.541	0.655	−0.371
18.23	−3.582	0.480	−0.421
20.83	−1.526	2.289	1.386
23.37	−1.986	1.550	1.000
25.67	−3.280	0.000	−0.531
27.84	−2.409	0.638	0.000
Average (% d_{av})	−1.62	0.39	0.13

[a] No data were predicted for these eluent compositions.

Data for % d_{glob}, $SD_{(\%\ d)}$ and the correlation coefficient for each model are given in Table 4. The success of each retention model in predicting retention times can now be assessed. The overall performance of each model can be best demonstrated using the correlation coefficient between the predicted and experimental retention times. This is shown in Fig. 3 as a graph of the correlation coefficient for each analyte as well as the overall performance of each model for all analytes.

There are several other important factors that must

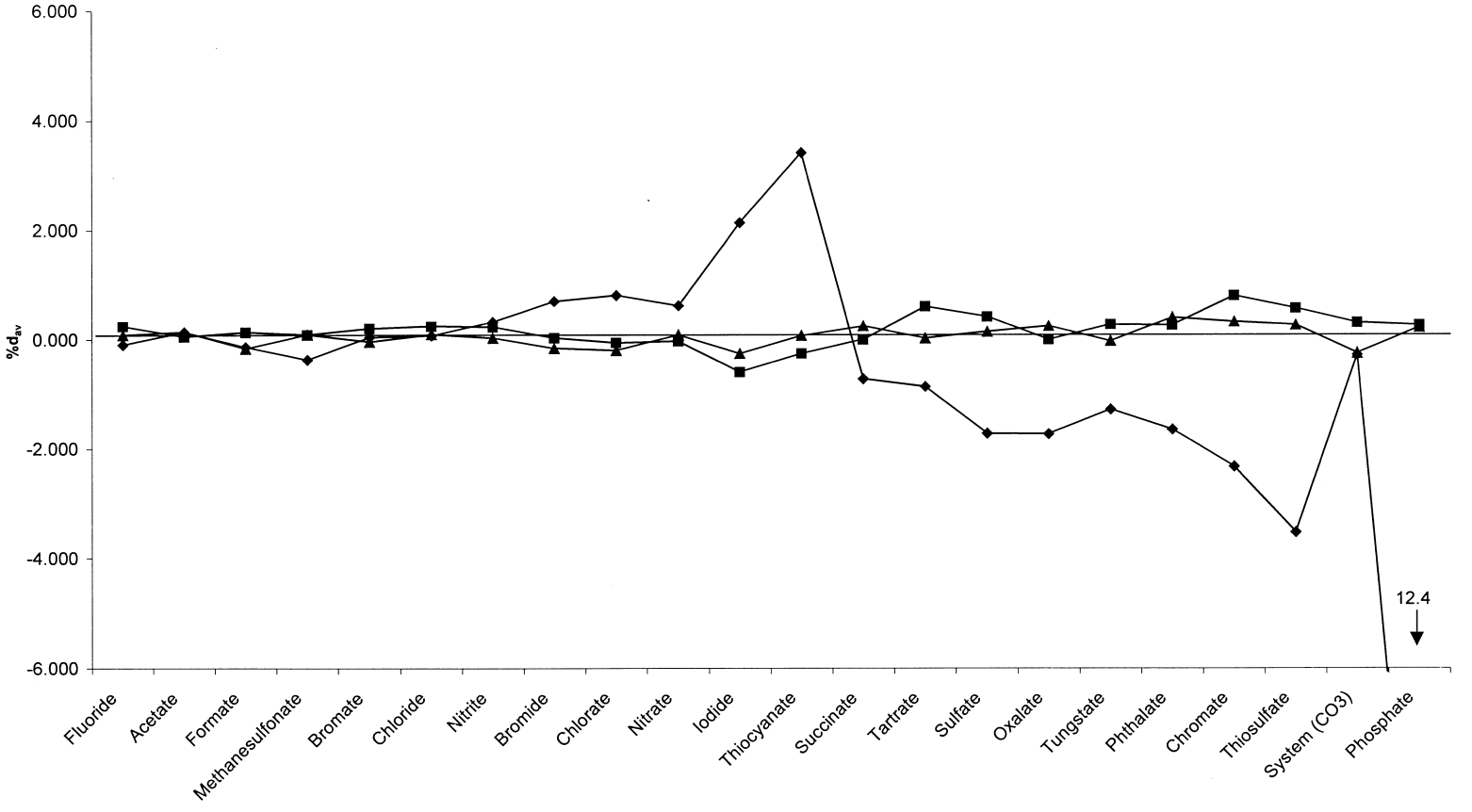


Fig. 2. Average of the normalised percentage differences between predicted and experimental data on the Dionex AS11 column using hydroxide as the eluent. ♦, Linear solvent strength model; ■, empirical end points model; ▲, three-point model of DryLab.

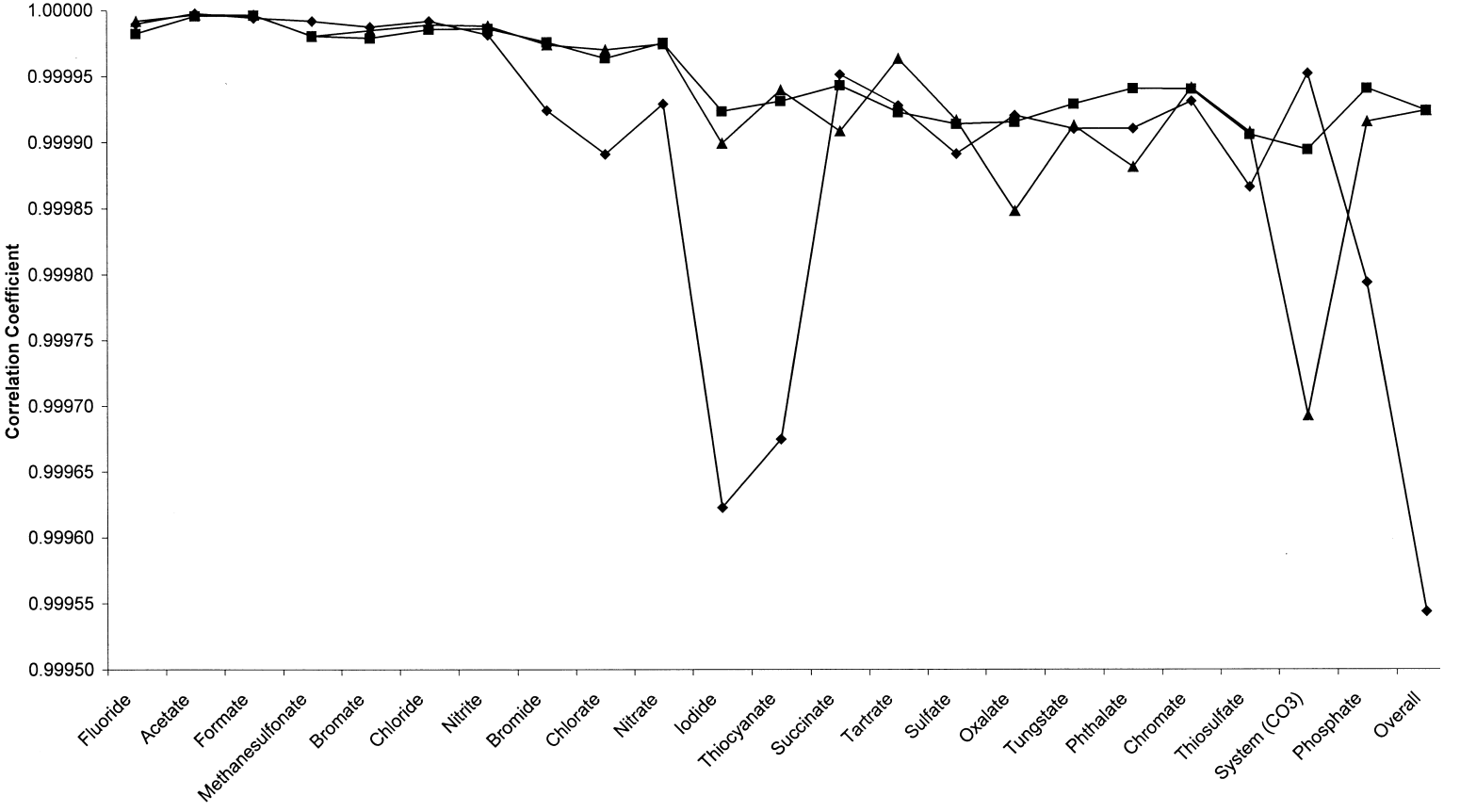


Fig. 3. Correlation coefficient between predicted and experimental data for each individual analyte and overall on the Dionex AS11 column using hydroxide as the eluent. ♦, Linear solvent strength model; ■, empirical end points model; ▲, three-point model of DryLab.

be considered when comparing the utility and performance of the retention models. The number of experiments required to solve the model should be as small as possible in order to maximise the speed of any optimisation routine using that model. In terms of ease of solution, all models could be solved in a straightforward manner. The amount of knowledge of the system required to find a solution is also a significant consideration and it can be noted that the LSSM requires knowledge of the charges on the eluent and analyte ions. In some cases, the exact charge on a given analyte may not be known unless careful calculations are performed.

5. Discussion

Figs. 1 and 2, and Table 4 provide information on which the performance of the retention models can be assessed. Several trends are evident and can be compared with trends noted previously for models applied to multiple species eluents [1,2].

(i) The LSSM gave consistently good performance for most anions, but poor results were observed when the model was applied to analytes with a high polarisability (iodide and thiocyanate) and to phosphate, which has a charge that varies with the concentration of hydroxide in the eluent.

(ii) The EEPM and DryLab gave good performance for all analytes, with DryLab showing a slight superiority in accuracy and precision. It must be pointed out that the measurement error for the experimental data is in the order of one percent. The standard deviations of both the DryLab and EEPM are very close to this error value.

(iii) The LSSM showed positive errors for singly charged analytes and negative errors for analytes with a charge greater than −1. Errors for the EEPM and DryLab were smaller and showed no correlation with the charge on the analyte.

(iv) The accuracy of the models was found to be directly proportional to the number of experimental data points necessary for their solution. Thus the LSSM, which required only one data point, was the least accurate, and the DryLab model, which required three data points, was the most accurate.

(v) The data in this paper and those published previously [1,2] for all of the seven retention models applied to dual and single eluent species systems in both suppressed and non-suppressed IC show several trends. First, single eluent species systems were the easiest to model, with the worst performed model (LSSM) giving much better predictions of retention factors than the best performed dual eluent species model [1,2]. Second, suppressed dual eluent species systems are the next easiest to model, with non-suppressed dual eluent species systems being by far the most difficult to model. The EEPM was the only model that was applicable to all systems and constantly gave superior results to all other models.

Taking into account the data on the accuracy and precision of each model and the factors that need to be considered in terms of ease of solution, it can be seen that the EEPM requires minimal input data but provides reliable prediction of retention factors. The three point curve fitting provided by DryLab requires more experimental data than the EEPM and gives increased accuracy and precision. An additional advantage of the EEPM as a basis for optimisation is that its accuracy can be further improved by iteration during the optimisation process. This is achieved by breaking the overall search area into smaller areas in which the distance between known data points is less than that in the original search area. DryLab is also capable of this same feature. Finally, it should be noted that DryLab is available commercially, whereas the LSS and EEP models require in-laboartaory software or manual calculations.

Table 4
Global average normalised percentage differences (% d_{glob}), and correlation coefficients (Corr.) between the predicted and experimental data and standard deviations of the percentage normalised differences [$SD_{(\% d)}$] for the retention models (the best result in each category is shown in bold face)

Model	% d_{glob}	$SD_{(\% d)}$	Corr.
Linear solvent strength	−0.847	3.749	0.999543
Empirical end points	0.166	1.140	**0.999923**
DryLab	**0.056**	**1.013**	0.999782

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