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Naphthalenesulfonates as mobile phases for anion-exchange chromatography using indirect photometric detection

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ABSTRACT

Naphthalenesulfonate mobile phases for chromatography with indirect photometric detection have been characterized for the separation and detection of common inorganic and organic anions using an ion-exchange column. The singly charged 2-naphthalenesulfonate (NMS) has shown good chromatographic performance for small singly charged inorganic anions such as F^- , Cl^- , NO_2^- , Br^- and NO_3^- with detection limits ranging from 20 to 4 ng. The doubly charged 1,5-naphthalenedisulfonate (NDS) was more efficient toward the separation and identification of small singly charged, multicharged, and large anions such as F^- , Cl^- , NO_2^- , Br^- , NO_3^- , SO_4^{2-} , $S_2O_3^{2-}$, I^- and SCN^- . Analysis of a mixture containing the above anions can be performed in less than 16 min, with detection limits in the 20–1 ng range. The linear response range of these mobile phases were from 100 ppm to the detection limits of all anions (0.05–1 ppm). Both NMS and NDS require no pH adjustment for indirect photometric detection use, and no system peak was observed in any chromatogram.

INTRODUCTION

Anion-exchange chromatography with indirect photometric detection is a simple but powerful technique for the separation and detection of non-UV-absorbing inorganic and organic anions. The crucial part of ion-exchange chromatography with indirect photometric detection chromatography (IEC-IPD) is the mobile phase, which plays roles as both an eluent and a means for detection. In IEC-IPD the ion-exchange column is first equilibrated with the light-absorbing mobile phase until a steady baseline with high absorbance background is reached. Mobile phases in IEC-IPD should have the ability of displacing the analyte ions from the stationary phase and selectively separating them. Detection in IEC-IPD is best accomplished by using an eluent which possesses a large molar absorptivity in order to achieve high sensitivity at a moderately low eluent concentration. The non-light-absorbing ions when eluted will be detected by observing the decrease in the absorbance. Negative peaks will be the result instead of the conventional positive peaks in the usual UV detection mode [1].

Most of the commonly used mobile phases in anion IEC-IPD are the salts of weak organic acids, such as phthalate, sulfobenzoate, and salicylate derivatives. These compounds possess relatively high molar absorptivities and, with their avail-

ability in pure form, are good candidates for anion IEC-IPD. However, the elution of analyte ions will be determined by the effective charge of the eluent, which in turn is dependent upon the pH of the mobile phase. Precise control of the mobile phase pH is found to be important to ensure reproducible elution and retention times [2-5].

Another drawback of these commonly used mobile phases is the presence of one or more extraneous (system or ghost) peaks, which are not related to the analyte sample. These peaks appear either between the analyte peaks in which a distorted chromatogram is the result, or appear at the end of the chromatogram, which in turn will increase the actual analysis time. Haddad and co-workers [2,3] have reported on two occasions the presence of a large system peak at 14 min, when using potassium hydrogenphthalate as the mobile phase. Brandt *et al.* [6], on the other hand, reported a system peak eluting in the middle of the chromatogram, when muconic acid was used as the eluent at pH 4.3. Sato [7] has reported in recent work a system peak when he used the acid form of 1,2-dihydroxybenzene-3,5-disulfonic acid as the eluent, but no system peak was observed when the salt was used. We have experienced in previous work [8], using 2-naphthol-3,6-disulfonate, a system peak eluting in the middle of the chromatogram. We were successful in reducing its size by 80%, by dissolving the sample in the mobile phase before injection. The mechanism and the conditions for the formation of the system peak are not fully understood. However, a theoretical study of the formation of the system peak has been recently published [9], in which the system peak was attributed to the elution of the neutral species (undissociated form) of the weak organic acid eluent in a reversed-phase retention mode. Consequently, when the salt of a strong acid was used no system peak was observed.

Our research group has been working on characterizing several new mobile phases for anion IEC-IPD with an effective charge independent of pH. We report here the results of optimizing the chromatographic conditions of 2-naphthalenesulfonate (NMS) and 1,5-naphthalenedisulfonate (NDS) as potential eluents for anion IEC-IPD. Both of these mobile phases generate ion chromatograms free of any system peaks. Moreover, these two eluents possess very large molar absorptivities and, consequently, detection limits of the separated anions are low.

EXPERIMENTAL

Instrumentation

The liquid chromatograph used in this study consisted of: a Model 510 high-performance liquid chromatographic (HPLC) pump, a Model U6K injector, a Model 490 programmable multiwavelength detector, and an IC-PAK anion-exchange column (5 cm × 4.6 mm I.D.), all from Waters Chromatography Division (Milford, MA, U.S.A.). A Model 5000 Fisher Recordall chart recorder (Austin, TX, U.S.A.) provided the output of chromatograms. A Varian Model DMS 90 UV-VIS spectrophotometer (Sunnyvale, CA, U.S.A.) was used for measuring absorption spectra of the mobile phases.

Reagents

Three kinds of NMS were studied in this work: NMS acid and NMS salt of 99.5% purity, both purchased from Eastman Kodak (Rochester, NY, U.S.A.), and NMS salt of 90% purity obtained from Aldrich (Milwaukee, WI, U.S.A.). The di-

sodium salt of NDS of 95% purity was purchased from Aldrich. Acetonitrile, HPLC grade, was received from Fisher Scientific (Fair Lawn, NJ, U.S.A.). Salts of the common anions, reagent grade or better quality, were obtained from different suppliers. A 1000-ppm stock solution of each anion was prepared and used for further dilutions. Triply distilled, deionized water was used for preparing all solutions. The Oxford water sample was obtained from the lab faucet and used without any treatment except dilution with triply distilled, deionized water.

A 0.01 *M* stock solution of each mobile phase was prepared and used for subsequent dilutions. All final mobile phases used in this study have been diluted with acetonitrile-water (10:90, v/v). This percentage of acetonitrile was found to be effective in reducing the unwanted hydrophobic interaction between the polymeric stationary phase and the naphthalene moiety of the mobile phase and provided smooth baseline noise [8]. Although other anion-exchange columns were tried, we found the IC-PAK column to give better performance with these mobile phases. This may be because this column packing has a relatively hydrophilic polymethacrylate-type polymer backbone, which should lessen any hydrophobic effect with these aromatic mobile phases.

RESULTS AND DISCUSSION

The UV absorption spectrum from 350 to 250 nm for 0.05 *mM* NMS superimposed on that of 0.02 *mM* NDS is shown in Fig. 1. The absorption maxima of the NMS and the NDS solutions occur at 275 and 285 nm, with molar absorptivities of 5160 and 13 100 l/mol cm, respectively. The 10-nm shift in the maximum absorption wavelength and the large difference in the molar absorptivities is mainly due to the

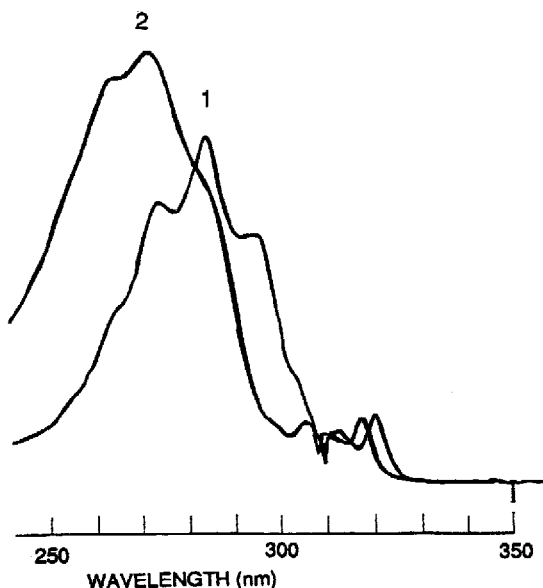


Fig. 1. UV spectra of (1) 0.02 *mM* NDS and (2) 0.05 *mM* NMS.

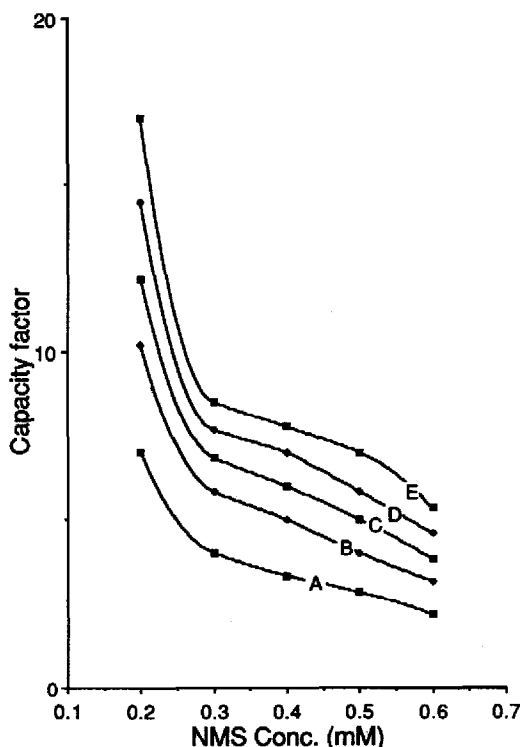


Fig. 2. Retention of inorganic analyte anions as a function of eluent concentration. Conditions: flow-rate, 1 ml/min; injection volume, 20 μ l; indirect UV detection at 280 nm; ambient temperature; solute concentration, 10 ppm. (A) F⁻; (B) Cl⁻; (C) NO₂⁻; (D) Br⁻; (E) NO₃⁻.

presence and position of the extra sulfonate group in NDS. Because of the common use and availability of the 280-nm filter in many UV detectors, most of this work was done at 280 nm as the detection wavelength, unless otherwise indicated. The molar absorptivities of NMS and NDS solutions at 280 nm are 4290 and 11250 l/mol cm, respectively. This represents a molar absorptivity decrease of only about 15% for both mobile phases.

The retention of fluoride, chloride, nitrite, bromide and nitrate as a function of NMS concentration is shown in Fig. 2. Larger and multicharged anions gave very long retention times and, consequently, were not studied using this mobile phase. As would be expected with all ion-exchange processes, the retention of these anions becomes shorter with increasing mobile phase concentration. The capacity factor (k') for these anions range from 2 to 7 with NMS concentration changing from 0.6 to 0.3 mM; below 0.2 mM, k' rises sharply to the range 7–17. At NMS concentrations higher than 0.6 mM, the separation of these anions becomes more difficult. Lower mobile phase concentrations provide higher sensitivities. The measured sensitivity (sensitivity = analyte peak area/its concentration) using 0.15 mM NMS is about 1.2 and 1.5 times higher than that using 0.3 and 0.6 mM NMS, respectively. This is due to the fact that small changes in absorbance can be easily detected in a low rather than a high background signal. In addition, the baseline noise level is much smaller at low

mobile phase concentrations; the peak-to-peak noise using 0.3 mM NMS is about one third that of 0.6 mM NMS, measured at 0.05 a.u.f.s. As a compromise, we have chosen 0.3 mM NMS–10% acetonitrile as the optimum concentration for this mobile phase. At this concentration, good sensitivity, separation performance, and reasonably short retention times can be achieved.

We have studied three kinds of NMS products. The acid form of NMS (Kodak product) was not completely soluble in water partly due to the presence of neutral impurities of naphthalene derivatives which can be easily noticed from the distinct smell. Extraction with chloroform and/or pH adjustment to 8 did not improve the solubility or the chromatographic performance of this acid. The NMS salt (Aldrich) gave a much better chromatographic performance. Analysis of a standard mixture of common inorganic anions using 0.3 mM NMS (Aldrich) as the mobile phase is shown in Fig. 3. All anions are well resolved, especially the fluoride ion peak which is well separated from the injection (solvent) peak and far from any interferences. However, the solubility of this product was not quite complete. Extraction with chloroform was not effective in removing the undissolved residues. A clear stock solution has been used throughout this study, after letting the impurities settle out for a few hours. The third product, NMS salt (Kodak), was very soluble in water with no residue and gave a fine chromatographic performance. Fig. 4 shows a separation of a 1-ppm standard anion mixture using 0.3 mM NMS (Kodak)–10% acetonitrile. The analysis time using this mobile phase is about half of that shown in Fig. 3, suggesting that the

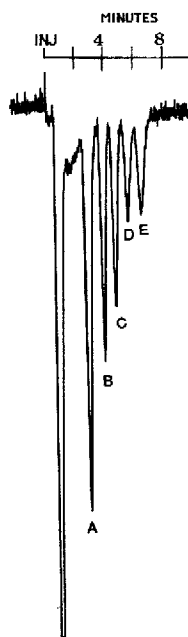


Fig. 3. Separation of a standard mixture of inorganic anions with IEC-IPD. Conditions as in Fig. 2 except: mobile phase, 0.3 mM NMS (Aldrich product)–10% acetonitrile; 0.05 a.u.f.s. Peak identification: 1 ppm each of (A) F^- , (B) Cl^- , (C) NO_2^- , (D) Br^- , and (E) NO_3^- .

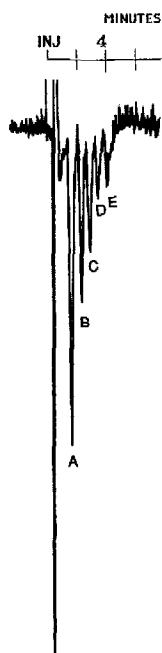


Fig. 4. Separation of a standard mixture of inorganic anions with IEC-IPD. Conditions as in Fig. 3 except: mobile phase, 0.3 mM NMS (Kodak product)-10% acetonitrile; detection wavelength, 280 nm, 0.05 a.u.f.s. Peak identification: 1 ppm each of (A) F^- , (B) Cl^- , (C) NO_2^- , (D) Br^- , and (E) NO_3^- .

Kodak product is higher in NMS content than the Aldrich product. This is in agreement with the supplier listing purity of 99.5 and 90%, respectively. Mobile phases using both brands of NMS salt required no pH adjustment and they showed very

TABLE I
LINEARITY RESPONSE FOR FIVE ANIONS BY IEC-IPD USING 0.3 mM NMS (KODAK PRODUCT)-10% ACETONITRILE AS THE MOBILE PHASE

Injected sample volume, 20 μ l.

Anion	Range (ppm)	Linear least-squares equation ($y = a + bx$) ^a	Correlation coefficient
Fluoride	100-0.2	$a = 1.23 \cdot 10^{-2} \pm 6.68 \cdot 10^{-3}$ $b = 3.71 \cdot 10^{-2} \pm 1.56 \cdot 10^{-4}$	0.9999
Chloride	100-0.1	$a = 1.78 \cdot 10^{-2} \pm 1.46 \cdot 10^{-2}$ $b = 6.92 \cdot 10^{-2} \pm 3.37 \cdot 10^{-4}$	0.9999
Nitrite	100-0.5	$a = -1.86 \cdot 10^{-3} \pm 2.83 \cdot 10^{-3}$ $b = 1.98 \cdot 10^{-2} \pm 6.51 \cdot 10^{-5}$	0.9999
Bromide	100-1	$a = -6.16 \cdot 10^{-3} \pm 5.03 \cdot 10^{-3}$ $b = 1.34 \cdot 10^{-2} \pm 1.04 \cdot 10^{-4}$	0.9998
Nitrate	100-1	$a = 1.21 \cdot 10^{-2} \pm 5.59 \cdot 10^{-2}$ $b = 1.42 \cdot 10^{-2} \pm 1.15 \cdot 10^{-4}$	0.9998

^a y = calculated peak area (cm^2); x = concentration (ppm).

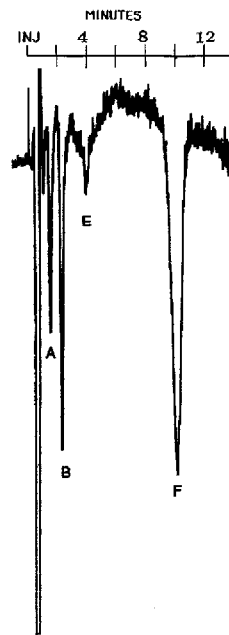


Fig. 5. Chromatogram of 0.2 ppm fluoride ion with IEC-IPD. Conditions as in Fig. 4.

Fig. 6. Chromatogram of 1% Oxford city water sample. Conditions as in Fig. 4 except: mobile phase, 0.3 mM NMS (Kodak product)-10% acetonitrile; detection wavelength, 275 nm; 0.02 a.u.f.s. Peak identification: A = F^- ; B = Cl^- ; E = NO_3^- ; F = SO_4^{2-} .

reproducible performances. The average relative standard deviation (R.S.D.) in retention time was 1.1% ($n = 4$). Moreover, chromatograms of both mobile phases showed no extraneous peak (system peak) even after 40 min of analysis.

The linearity response for the five anions studied extended from 100 ppm to the detection limits of each anion, using the optimized NMS mobile phase (Table I). The R.S.D. of the slope ranged from 0.33 to 0.81% with an average of 0.57% for the five anions. The correlation coefficient varied from 0.9998 to 0.9999. The average R.S.D. in the calculated peak area was about 1% ($n = 4$). Fig. 5 shows a chromatogram of fluoride ion at its reported detection limit. The signal-to-noise ratio for this early eluted anion is more than 3.

A chromatogram of 1% tap city water is shown in Fig. 6, using 0.3 mM NMS (Kodak). The components of the water sample were identified and quantified using a standard mixture of all the components except fluoride. The fluoride ion quantitation was done individually because of variation of the bicarbonate ion present in the triply distilled water which we found affected the fluoride ion area calculation. To overcome this problem both samples and standards were prepared at the same time and diluted with the same triply distilled water. The concentrations of each component found in Oxford city water were 1.16 mg/l fluoride, 25.5 mg/l chloride, 2.26 mg/l nitrate and 52.6 mg/l sulfate. These results agreed well (within $\pm 10\%$) with the most recent tap water analysis data published by the city water authority.

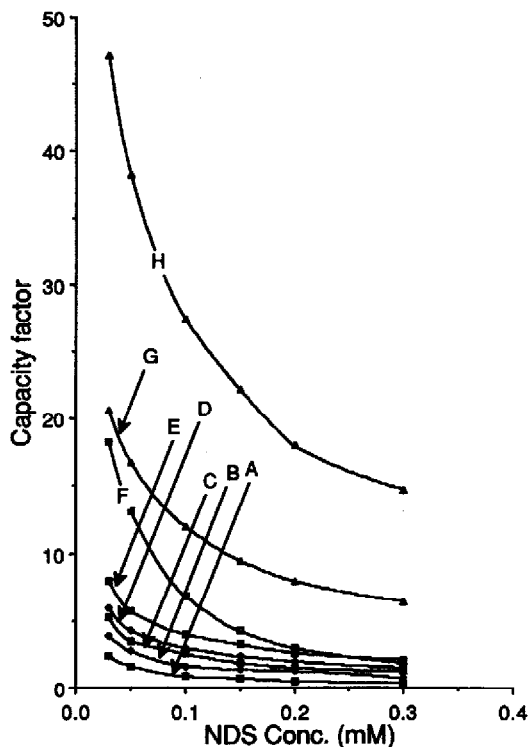


Fig. 7. Retention of inorganic anions as a function of eluent concentration. Conditions as in Fig. 2. (A) F^- ; (B) Cl^- ; (C) NO_2^- ; (D) Br^- ; (E) NO_3^- ; (F) SO_4^{2-} ; (G) I^- ; (H) SCN^- .

Another mobile phase that has been studied in this work is NDS disodium salt. The retention of various common anions as a function of NDS concentration is shown in Fig. 7. As with NMS, the capacity factors for the small singly charged anions such as F^- , Cl^- , NO_2^- , Br^- , and NO_3^- ranged from 2 to 7. For the doubly charged and large anions such as SO_4^{2-} , I^- , and SCN^- , the k' values were from 3 to 47 as the mobile phase concentration was varied between 0.3 and 0.03 mM. For mobile phase concentrations higher than 0.2 mM, separation of these anions becomes more difficult due to the overlap of the early eluting anions with the injection peak. For mobile phase concentrations lower than 0.1 mM, the retention of these anions becomes longer and consequently peak broadening of the late eluting anions is observed. The baseline noise measured at 0.05 a.u.f.s. was 2.7 times larger and the calculated sensitivity was 0.81 times smaller for 0.15 mM compared to 0.05 mM NDS. These results are, again, in agreement with the theory of indirect detection methods [10].

The optimized mobile phase selected, considering factors such as sensitivity, resolution, and analysis time, was 0.15 mM NDS–10% acetonitrile. Fig. 8 shows a typical example for the separation of a mixture containing nine monovalent and divalent anions using the optimized mobile phase. The analysis times for small monovalent anions such as F^- , Cl^- , NO_2^- , Br^- , and NO_3^- and for divalent anions such as

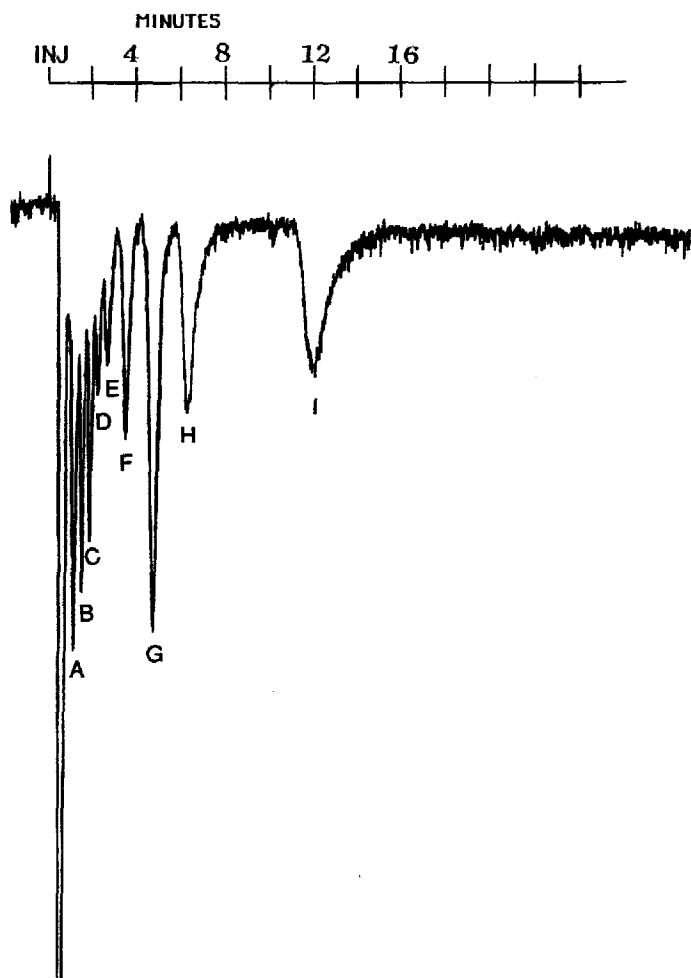


Fig. 8. Separation of a standard mixture of inorganic anions with IEC-IPD. Conditions as in Fig. 3 except: mobile phase is 0.15 *M* NDS-10% acetonitrile. Peak identification: 0.5 ppm each of (A) F^- , (B) Cl^- , (C) NO_2^- , (D) Br^- , (E) NO_3^- and (F) SO_4^{2-} and 2.5 ppm each of (G) $S_2O_3^{2-}$, (H) I^- , and (I) SCN^- .

SO_4^{2-} and $S_2O_3^{2-}$ are less than 4 and 6 min, respectively. The retention time for large anions such as I^- and SCN^- is 14 min, which is good considering IEC-IPD of these anions is rare [11]. This overall short analysis time makes NDS a more favorable mobile phase for simultaneous separation of these three groups of anions.

Linearity response for the eight anions studied with NDS is shown in Table II. With the exception of fluoride ion, the linear response for all anions extended from 100 ppm to the detection limit of each anion. Because of its early elution, the fluoride ion peak overlapped with the injection peak especially at concentrations higher than 50 ppm, which makes the peak-area measurements inaccurate. The average R.S.D. of the calculated peak area was 0.75% ($n = 4$). The R.S.D. of the slope ranges from 1.4 to 0.30% with an average of 0.62%, while the correlation coefficient for all anions

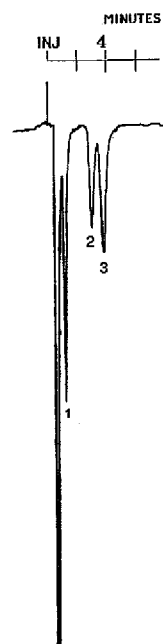
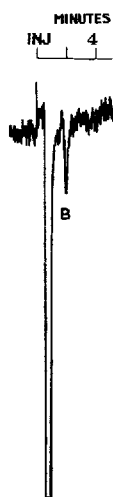


Fig. 9 Chromatogram of 0.05 ppm chloride ion. Conditions as in Fig. 8.

Fig. 10. Separation of an organic anion mixture with IEC-IPD. Conditions as in Fig. 8 except using 0.2 a.u.f.s. Peak identification: 5 ppm each of (1) lactic acid, (2) maleic acid, and (3) tartaric acid.

TABLE II

LINEARITY RESPONSE FOR EIGHT ANIONS BY IEC-IPD USING 0.15 mM NDS-10% ACETO-NITRILE AS THE MOBILE PHASE

Injected sample volume, 20 μ l.

Anion	Range (ppm)	Linear least-squares equations ($y = a + bx$) ^a	Correlation coefficient
Fluoride	50-0.05	$a = -4.54 \cdot 10^{-2} \pm 2.65 \cdot 10^{-2}$ $b = 1.41 \cdot 10^{-1} \pm 1.39 \cdot 10^{-3}$	0.9997
Chloride	100-0.05	$a = -1.41 \cdot 10^{-2} \pm 1.52 \cdot 10^{-2}$ $b = 8.73 \cdot 10^{-2} \pm 3.67 \cdot 10^{-4}$	0.9999
Nitrite	100-0.05	$a = -1.43 \cdot 10^{-2} \pm 8.19 \cdot 10^{-3}$ $b = 7.00 \cdot 10^{-2} \pm 1.98 \cdot 10^{-4}$	0.9999
Bromide	100-0.1	$a = -1.07 \cdot 10^{-2} \pm 6.33 \cdot 10^{-3}$ $b = 3.87 \cdot 10^{-2} \pm 1.46 \cdot 10^{-4}$	0.9999
Nitrate	100-0.5	$a = -5.80 \cdot 10^{-2} \pm 2.12 \cdot 10^{-2}$ $b = 4.96 \cdot 10^{-2} \pm 4.62 \cdot 10^{-4}$	0.9997
Sulfate	100-0.1	$a = -1.18 \cdot 10^{-2} \pm 8.67 \cdot 10^{-3}$ $b = 6.07 \cdot 10^{-2} \pm 1.89 \cdot 10^{-4}$	0.9999
Iodide	100-1	$a = -7.91 \cdot 10^{-3} \pm 2.63 \cdot 10^{-3}$ $b = 1.81 \cdot 10^{-2} \pm 5.41 \cdot 10^{-5}$	0.9999
Thiocyanate	100-1	$a = -1.75 \cdot 10^{-2} \pm 3.26 \cdot 10^{-2}$ $b = 4.94 \cdot 10^{-2} \pm 6.71 \cdot 10^{-4}$	0.9995

^a y = calculated peak area (cm^2); x = concentration (ppm).

TABLE III
DETECTION LIMIT COMPARISON FOR VARIOUS ANIONS

Signal-to-noise ratio ≥ 3 ; sample volume, 20 μ l.

Anion	Concentration ^a			
	NMS		NDS	
	ppm	ng	ppm	ng
Fluoride	0.2	4	0.05	1
Chloride	0.1	2	0.05	1
Nitrite	0.5	10	0.05	1
Bromide	1	20	0.1	2
Nitrate	1	20	0.5	10
Sulfate	—	—	0.1	2
Iodide	—	—	1	20
Thiocyanate	—	—	1	20

^a Using the optimized mobile phases: 0.3 mM NMS and 0.15 mM NDS both in 10% acetonitrile.

varied from 0.9997 to 0.9999. Fig. 9 shows a chromatogram of chloride ion at its detection limit of 0.05 ppm with a signal-to-noise ratio of more than 4.

A typical chromatogram for the separation of a mixture of organic anions containing lactic, maleic and tartaric acids is shown in Fig. 10. These three hydroxycarboxylic acids are well separated in the order as expected based on their size, pK_a values, and number of carboxylate groups. Lactic acid (mol. wt. 90) has a pK_a of 3.86. The diprotic maleic acid (mol. wt. 116) has pK_a values of 1.94 and 6.22, while tartaric acid (mol. wt. 150) has pK_a values of 3.22 and 4.81.

A detection limit comparison of all anions using the optimized mobile phases of NMS and NDS is shown in Table III. The detection limits of the early eluting anions range from 0.1 to 1 ppm (2–20 ng) and 0.05 to 0.5 ppm, (1–10 ng) using NMS and NDS, respectively. Lower detection limits for NDS are observed compared to that of NMS. This can be attributed to two main factors. First, the molar absorptivity of NDS is about 2.5 times larger than that of NMS. Secondly, since NDS is doubly charged, its elution tendency is much stronger, which consequently means lower mobile phase concentrations and lower baseline noise. Fig. 11 shows a slope comparison of two linearity runs for fluoride and nitrite ions using the optimized NMS and NDS mobile phases. It is quite clear that the slope for NDS is much higher than that for NMS. This trend has been consistently observed for all anions studied in this work.

Furthermore, NDS has proven to be even more favorable than the previously optimized IEC-IPD mobile phase, naphtholdisulfonate (NODS) [8], which has the same effective charge of -2 . The NDS analysis time in Fig. 8 is about 5 min shorter than that using NODS, although the same mobile phase concentrations and compositions were used. The detection limits for F^- , Cl^- , NO_2^- , and Br^- are 10 times lower using NDS than those using NODS as the mobile phase, while the detection limits for larger and multicharged anions such as NO_3^- , SO_4^{2-} , I^- , and SCN^- ranged from 2 to 10 times higher. These trends can be explained by lower baseline noise using NODS

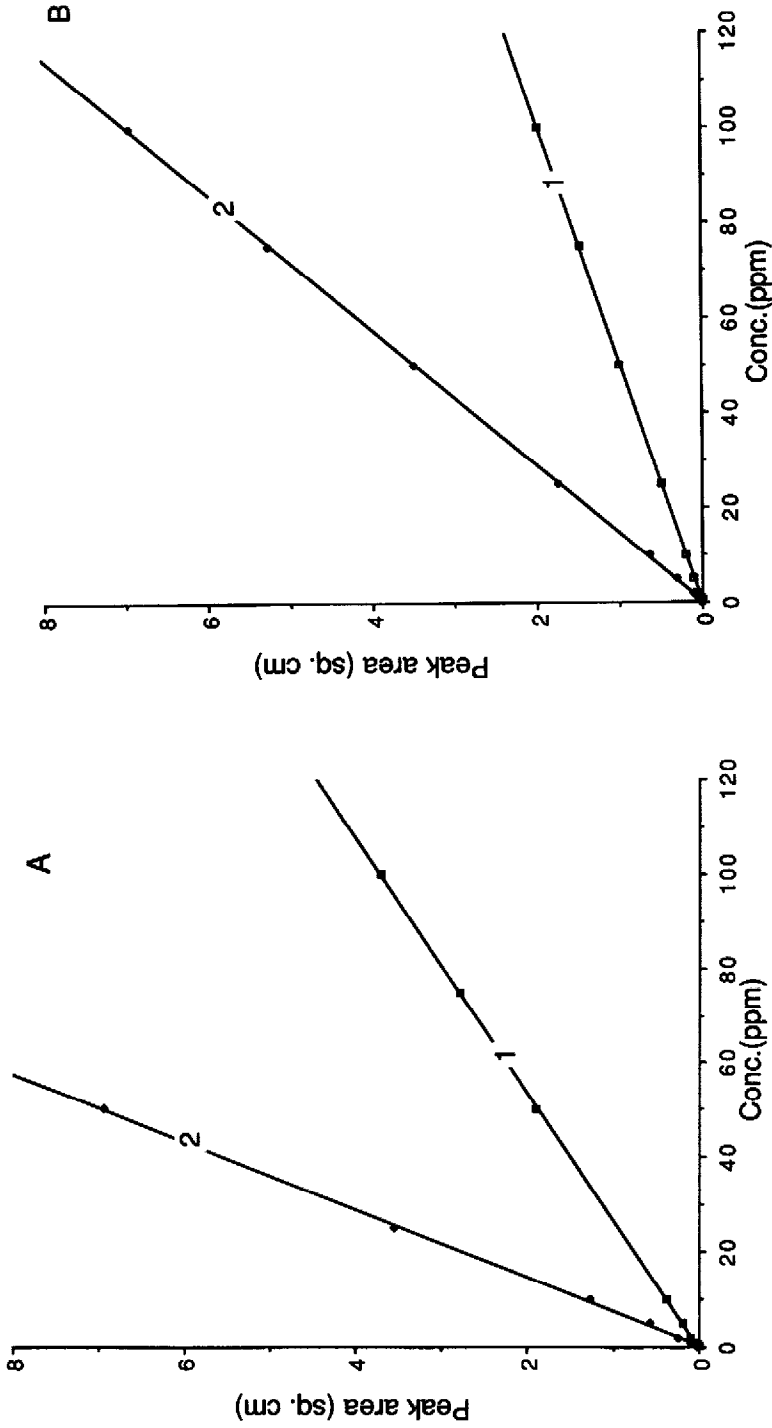


Fig. 11. Linearity run comparison of (A) fluoride ion and (B) nitrite ion, using (1) 0.3 mM NMS and (2) 0.15 mM NDS both in 10% acetonitrile. Conditions as in Figs. 4 and 8.

for the late-eluting peaks and system peak interference with the early eluting peaks with NODS. No system peak was observed with NDS and consequently no special treatment of the analyte samples was necessary before analysis. In addition, no problems were experienced with the nitrite ion separation using NDS as the mobile phase. In contrast, using NODS, the nitrite ion peak extended from the positive to the negative direction, affecting the resolution of the neighboring ions.

This work has demonstrated the applicability of NMS and NDS as potential mobile phases for anion IEC-IPD. NMS has been found quite suitable for the analysis of small and singly charged anions, with high efficiency. NDS, on the other hand can be used for the separation and identification of both small singly charged as well as large multicharged anions, with very high sensitivity. Both mobile phases required no pH adjustment and the absence of any system peak in the chromatograms allowed easy peak identification. These mobile phases, furthermore, caused no degradation of the analytical column and constant column efficiency was maintained.

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