ANION CHROMATOGRAPHY WITH LOW-CONDUCTIVITY ELUENTS

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SUMMARY

A new and very simple system is described for separation and determination of mixtures of inorganic and organic anions. The column is filled with a macroporous anion-exchange resin of 0.007 to 0.07 meq/g exchange capacity. The eluent is a very dilute (0.0001 to 0.00065 M) solution of an organic acid salt, such as potassium benzoate or phthalate. The eluent conductance is sufficiently low that the separated anions can be detected with a simple conductance detector. Numerous examples of actual separations are shown. In some cases concentrations of anions below 1 ppm could be detected. The applicability of the new system to real samples is demonstrated by several examples.

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INTRODUCTION

In recent years there has been a growing need for reliable analytical methods for the rapid, simultaneous determination of mixtures of common anions in dilute, aqueous solutions. Not until the development by Small, Stevens and Bauman[1] of the procedure which they call "Ion Chromatography" has the rapid separation and quantitative determination of inorganic anions (and certain cations) become possible. Their system, which is marketed as a commercial instrument by the Dionex Corp., uses a separation column containing a patented anion-exchange resin. The separated anions are detected by conductance after passing through a hydrogen-form cation exchange column (called a suppressor column) to remove most of the background conductance of the eluent.

This commercial instrument, called the "Ion Chromatograph", permits good resolution of many anion mixtures with high sensitivity, and it has therefore found many applications, especially in the analysis of environmental samples.[2] A major disadvantage of this instrument is that it uses a suppressor column that accumulates the ions from the eluent stream and

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which must be regenerated periodically to remove the unwanted ions. Another drawback is that the eluent must be a base, so that after neutralization by the suppressor column it will have a low conductivity. A solution of sodium carbonate and bicarbonate is the most commonly used eluent. Anions of very weak acids cannot be detected on the "Ion Chromatograph" because their acidic form is too weakly conducting.

This paper describes a very simple chromatographic system that permits a quick and quantitative separation of several common anions. The new system for anion chromatography uses a conductivity detector but requires no suppressor column. This has been achieved by two principal innovations: (1) use of a special anion-exchange resin of very low capacity, and (2) the adoption of an eluent having a very low conductivity. The eluent used is approximately a 5.0×10^{-4} M aqueous solution of potassium benzoate, potassium phthalate or ammonium o-sulfobenzoate. Very sharp separations of anions could be achieved, and in no case did the absence of a suppressor column prove to be an obstacle.

EXPERIMENTAL

Sample Solutions. Calibrated solutions of the potassium salts of various anions (fluoride, chloride sulfate, etc., as shown in the chromatograms) were prepared from analytical-grade reagents.

Ion-exchange Resins. Two kinds of ion exchangers served as the stationary phase of the analytical column, viz. the relatively high-capacity, commercially available Vydac SC anion exchange resin (bead size: 30-44 microns, capacity: 0.1 meq/g), and three low-capacity anion exchange resins specially prepared at this laboratory by the following method:

The copolymer used as starting material for the synthesis of the anion exchange resins were Rohm and Haas' macroreticular cross-linked polystyrene beads XAD-1. This resin is highly cross-linked and had excellent chemical and mechanical stability. The beads were ground and sieved, and the fraction of 150-160 mesh was collected for the synthesis.

A 3 g portion of that fraction was swollen in a mixture of 10 ml chloromethylmethyl ether, 10 ml methylene chloride and 3 ml nitromethane. When 3 g of zinc chloride were added to the mixture the reaction was initiated and was maintained at room temperature for up to 14 minutes, after which the reaction was quenched by adding water to the mixture. The beads were then filtered, washed with water and methanol and finally air dried.

The chloromethylated beads were aminated by adding liquefied trimethylamine and allowing the excess amine to evaporate overnight. The final product was washed with 1 M HCl, 2-propanol, and water, and was dried overnight at 60°C.

Anion-exchange resins of three different capacities were prepared. The first had a capacity of 0.04 meq/g, the second a capacity of 0.07 meq/g, and the third a capacity of 0.007 meq/g.

Eluents

Potassium benzoate: 6.5×10^{-4} M pH = 4.6, specific conductance = 6.59×10^{-5} mho/cm

Potassium biphthalate: 5×10^{-4} M pH = 4.40, specific conductance = 7.43×10^{-5} mho/cm

5×10^{-4} M pH = 6.20, specific conductance = 1.12×10^{-4} mho/cm

	6.5×10^{-4} M pH = 4.40, specific conductance = 9.05×10^{-5} mho/cm
	6.5×10^{-4} M pH = 6.20, specific conductance = 1.58×10^{-4} mho/cm
o-Sulfobenzoic acid ammonium salt	5×10^{-4} M pH = 5.8, specific conductance = 1.32×10^{-4} mho/cm

Apparatus. The chromatographic flow system used, shown in Fig. 1, consisted of the following parts:

1) A Milton-Roy pump (Model 396 Simplex) which serves to force the eluent to flow from the eluent tank through the sample loop, the analytical column, and the conductivity cell, at a flow rate of 2 ml/min.
2) A sample injection valve, which makes use of a sample loop and enables liquid increments as small as 50-100 microliters to be injected.
3) Two different analytical separation columns: For the Vydac resin and the XAD-1 0.007 meq/g resin, the dimensions of the column were 3 mm i.d. and 500 mm length; for the low-capacity XAD-1 0.04 meq/g a column 2 mm i.d. and 1000 mm long was used.
4) A fluid conductivity cell assembly which measures the conductivity of the solution emerging from the analytical column.
5) A conductivity detector, which continuously monitors the effluent's conductivity.
6) A recorder, which traces the output of the conductivity meter.

Initially, the eluent must be pumped through the system for 15-30 min before the baseline is stabilized and the sample can be injected. The back-pressure for the column of XAD resin is approximately 200 psi; that for the column of Vydac resin is about 450 psi.

RESULTS

Eluent

The eluent anion must be retained by the anion-exchange resin strongly enough that a very low concentration of the eluent salt will move anions to be separated down the chromatographic column. At the same time the conductance of the eluent should be low so that the separated anions (with an appropriate cationic counter ion) will give a detector signal well above that of the eluent background.

Potassium acid phthalate, pH 4.4, 5.0×10^{-4} M was the first eluent tried. It gave well-defined peaks for separation of simple mixtures of two or three common anions using a low-capacity (0.04 meq/g) XAD-1 resin column and a conductance detector. Further investigation revealed that 5.0×10^{-4} M solutions of potassium benzoate, potassium phthalate, and potassium sulfobenzoate (all at pH 6) are excellent eluents. A 6.5×10^{-5} M solution of potassium malonate, pH 6.1, is a much less effective eluent than any of the three listed above. Apparently a benzene ring in the chemical structure of the eluent salt plays a major role in enabling inorganic anions to be effectively eluted from the XAD-1 resin. Similarly, a 5.0×10^{-4} M solution of potassium perchlorate or potassium citrate was a rather ineffective eluent. Adjusted retention times for elution of inorganic anions with various eluents are summarized in Table I. Several experiments indicated

that there is little difference in effectiveness between the potassium and sodium salts of the different eluents.

Of the three recommended eluents, potassium benzoate has the mildest eluting ability. Using 0.04 meq/g XAD-1 resin and 5.0×10^{-4} M benzoate, an excellent separation of fluoride chloride and bromide was obtained (see Fig. 2). There is an interest in separating nitrate and nitrite; Fig. 3 shows excellent resolution of these two anions using benzoate eluent. However, benzoate is not an effective eluent for ions that are more tenaciously held by the XAD-1 resin, such as sulfate, iodide and thiocyanate.

Phthalate is intermediate in eluting ability of the three recommended eluents. Using a 5.0×10^{-4} M solution, anions are eluted more quickly when the eluent pH is raised from 4.4 to 6.1 (see Table I). For sulfate the pH effect is quite dramatic, the adjusted retention time going from 16.4 min at pH 4.4 to only 4.4 min at pH 6.1. However, thiocyanate has almost the same adjusted retention time at the two pH values (14.7 min and 15.0 min). The retention time of iodide changes only slightly with pH. Since more of the phthalate is present as the divalent anion at pH 6.1, it would be expected to be a better eluent at the higher pH. The background conductivity is greater at pH 6.1, making the anion peak heights lower. Fig. 4 shows the chromatogram for a mixture containing five different anions.

Ammonium o-sulfobenzoate, pH 5.8, was the most effective salt for eluting divalent anions, which are usually very strongly retained on the resin. The adjusted retention time of the chromate ion, for instance, is only 0.5 min on the 0.04 meq/g XAD-1 resin using 5.0×10^{-4} M sulfobenzoate. This retention time compares very favorably with the 25 min retention time obtained in the Dionex system using a mixture of sodium bicarbonate and

sodium carbonate as the eluent. The retention time of the oxalate anion is 3.5 min with sulfobenzoate and XAD-1 anion exchange resin, compared with 18 min in the Dionex system. An excellent separation of sulfate and thiosulfate is shown in Fig. 5 with sulfobenzoate at pH 7.3 as the eluent.

Resin

The type and capacity of anion exchange resin are important variables in anion chromatography. Rohm and Haas XAD-1, a macroporous polystyrene-divinylbenzene resin, was converted to an anion-exchange resin of low capacity by chloromethylation under very mild conditions, followed by amination with trimethylamine. This resin has excellent physical properties and is well suited to column chromatography under pressure. Most of the work was done with XAD-1 anion-exchange resin having a capacity of 0.04 meq/g. However, an XAD-1 resin of 0.07 meq/g and a third resin of very low capacity (0.007 meq/g) were also prepared and tested.

For several monovalent anions Gjerde and Fritz (4) showed that the selectivity coefficient is largely independent of resin capacity over the capacity range studied, 0.1 to 1.0 meq/g. It follows from this that the concentration of an anionic eluent required to elute any given anion from a resin column decreases with decreasing resin capacity. This is confirmed by the data in Table II, which compares the adjusted retention times of a number of anions from resin columns of different capacity. The data show appreciably longer retention times for all anions on the 0.07 meq/g resin compared to the 0.04 meq/g resin. Anions are eluted efficiently from the resin column at the lowest capacity (0.007 meq/g) by a phthalate eluent that is only 1.0×10^{-4} M or a benzoate eluent of only 2×10^{-4} M. Using eluents of such low conductivity enhances the sensitivity of the system and consequently quantitative

determinations of chloride and nitrate below 1 ppm could be achieved using this low-capacity resin.

The XAD-1 resin of 0.007 meq/g capacity is excellent for separation of anions that elute slowly from other resin columns. With 1.0×10^{-4} M phthalate (pH 7.1) as eluent, chloride, bromide and nitrate elute together, but ions such as iodide, thiocyanate, sulfate and thiosulfate have different retention times. Figure 6 shows an excellent separation of chloride, iodide, thiocyanate and sulfate; the last two ions are much better resolved than on the higher capacity resin used in Fig. 4. A nice separation of chromate, sulfate and thiosulfate is shown in Fig. 7. Furthermore, well defined peaks could be obtained for HPO_4^{2-} and ClO_4^- which could not be achieved with the higher capacity resins.

Since elution behavior of anions is highly dependent on resin capacity, separations will vary from one batch of resin to the next unless the resin capacity is carefully controlled. Fortunately resin capacity can be reproduced very nicely by controlling the conditions used for chloromethylation; two batches of resin had identical capacities (0.04 meq/g) and behaved similarly in actual use.

Several experiments were performed with a commercially-available resin, Vydac SC. This is a solid core silica resin with an organic anion-exchange resin chemically bonded to the porous surface layer of the resin beads. The anion-exchange capacity is listed by the manufacturer as 0.10 meq/g. Vydac is a proprietary resin and the exact nature of the organic part is unknown, although it does appear to contain a quaternary ammonium functional group.

The performance of a 50-cm column packed with Vydac SC was very good. Fig. 8 shows an excellent separation of five anions. The resolution was

much better than for similar separations attempted on a XAD-1 anion-exchange column. The relative retention times of different anions on Vydac are different from those obtained on XAD-1. Nitrate, in particular, elutes quite late from a Vydac column.

Some Practical Applications

The applicability of the method to real samples was demonstrated by a number of practical examples.

1) Determination of sulfate in water. The quantitative determination of ppm concentrations of sulfate in water is a widely studied problem; often a turbidimetric or spectrophotometric procedure is used for the analysis (3). Low concentrations of sulfate can be separated rapidly by anion chromatography, as shown in Fig. 9. Using an appropriate calibration plot, an accurate quantitative determination of the sulfate is possible.

The efficacy of the chromatographic method was demonstrated by separating a series of samples containing chloride, nitrate and sulfate on a 50-cm XAD-1 column with 6.5×10^{-4} M potassium phthalate (pH 6.2) as the eluent. The chloride and nitrate concentrations were held constant at 5.12 and 12.2 ppm, respectively, while the sulfate concentration was varied from 2.75 ppm to 13.75 ppm. A plot of sulfate peak height vs. concentration proved to be perfectly linear, so that an accurate quantitative determination of sulfate was possible. The chromatograms obtained point to the possible use of the new chromatographic system for routine water analysis for the simultaneous determination of three anions.

2) Determination of nitrite in tap water. Because of its deleterious effects, there is a great interest in controlling the nitrite concentration in drinking water. Fig. 10 shows a chromatogram of tap water taken with a Vydac column

and 5.0×10^{-4} M phthalate eluent at pH 6.0. The small peak between chloride and sulfate with a retention time of 5.2 min was identified as nitrite. Standard additions of nitrite to the tap water increased the height of the nitrite peak and yielded a linear correlation between peak height and nitrite added. Extrapolation of this plot to zero added nitrite gave a nitrite concentration of 0.37 ppm in laboratory tap water.

3) Determination of nitrite and nitrate in curing salt. This product consists largely of ordinary table salt with small quantities of sodium nitrite and sodium nitrate added. The analytical problem was to determine these added salts in the presence of large amounts of sodium chloride. Nitrite was determined by injecting a sample containing 0.5 g/l of curing salt onto a Vydac column and eluting with the same eluent as used in the previous example. Then several chromatograms were recorded in which increasing amounts of nitrite were added. From a linear plot of peak height vs. nitrite added, the sodium nitrite concentration of the curing salt was found to be 0.27%.

A solution containing 4 g/l of curing salt was found to be necessary to obtain a well-defined peak for nitrate. The column and eluent were the same as used for the nitrite determination, but the retention time for nitrate was 16.5 min. Standard additions of nitrate again resulted in a linear dependence between peak height and concentration. Extrapolation of the plot gave a concentration of 0.34% sodium nitrate in the curing salt.

DISCUSSION

A feature common to all the chromatograms is the very first peak, which is related to the total concentration of the sample salts injected and is

called the "pseudo peak". When a sample is injected onto the column, the anions present in the sample replace the eluent anion from the anion-exchange resin at the top of the column. The displaced eluent anions, together with the sample cations, move with the solvent front to the conductivity detector. If this concentration of cations and anions is such that the conductance is greater than that of the eluent background, there will be a positive peak. In cases where the concentration is less than the eluent background, a negative peak will result.

The XAD-1 anion exchangers used in this work appear to have much stronger hydrophobic interactions with anions than with more conventional anion exchange resins. For example, iodide, thiocyanate, and other anions that are easily dehydrated are not eluted from 0.04 meq/g XAD-1 by most ionic eluents. Actually the resin environment is rather strongly hydrophobic with only about one benzene ring in 200 derivatized with the ionic quaternary ammonium functional group. The $-CH_2\overset{+}{N}(CH_3)_3I^-$ (or SCN^-) ion pair is stabilized by interaction with the hydrophobic resin environment, making it difficult to elute iodide (or thiocyanate) from the resin column. Anions that are not easily dehydrated (F^-, Cl^-, Br^-, $SO_4^=$, NO_3^-, etc.) form more hydrophylic ion pairs with the resin quaternary ammonium groups and probably are not stabilized appreciably by interaction with the hydrophobic resin environment. Therefore this latter group of anions is more readily eluted from the column and separations of the individual anions can be achieved.

CONCLUSIONS

A column containing an anion-exchange resin of very low capacity, together with a suitable eluent of low concentration and a conductivity detector, is

capable of giving quite acceptable and quick separations of mixtures containing several anions. The great simplicity of the method makes it feasible to construct an instrument dedicated to the analysis of a single type of sample. Or, a general purpose instrument could be used to quickly determine a variety of anions in different types of samples.

It is likely that the quality of separations can be improved over those reported here. For example, our apparatus has more valves and tubing than are really necessary, and this may well contribute to a certain amount of band spreading. Resins and resin columns more efficient than those used here can probably be developed, and these would give better resolution of samples containing several anions.

ACKNOWLEDGMENT

This work was supported by the U.S. Department of Energy, Office of Basic Energy Sciences, Chemical Sciences Division.

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Table I: Adjusted retention times (min) of anions on XAD-1, 0.04 meq/g with different eluents (t_o = 1.8 min)

Eluent Ion	benzoate $5x10^{-4}$ M, pH = 6.0	phthlalate $5x10^{-4}$ M, pH = 4.4	phthalate $5x10^{-4}$ M, pH = 6.1	sulfobenzoate $5x10^{-4}$ M, pH = 5.8	perchlorate $5x10^{-4}$ M, pH - 7.0	malonate $6.5x10^{-4}$ M, pH = 6.1
F^-	2.8	0.8	0.0	0.0	-	-
Cl^-	3.9	1.3	0.7	0.7	5.8	4.2
NO_2^-	4.8	1.4	0.8	1.3	-	3.8
Br^-	6.3	2.1	1.3	1.1	8.6	4.6
NO_3^-	7.2	2.2	1.4	1.3	9.4	6.7
$SO_4^=$	∞	16.4	4.4	3.3	∞	∞
I^-	∞	6.3	5.4	∞		
SCN^-	∞	14.7	15.0	∞		
$C_2O_4^=$			5.0	3.5		
$CrO_4^=$			0.8	0.5		
$SO_3^=$			4.5	3.2		
$S_2O_3^=$			7.5	5.1		

- negative or multiple peak

∞ very long retention times

Table II: Comparative adjusted retention times (min) for XAD-1 0.04 meq/g, XAD-1 0.07 meq/g, and XAD-1 0.007 meq/g

Ion	0.07 meq/g*	0.04 meq/g*	0.007 meq/g+
F^-	0.6	0.0	0.6
Cl^-	1.5	0.7	1.0
NO_2^-	2.0	0.8	1.4
Br^-	3.8	1.3	1.2
NO_3^-	4.5	1.4	1.5
SO_4^{2-}	11.5	4.4	5.5
I^-	∞	5.4	2.0
CNS^-	∞	15.0	4.0
$C_2O_4^{2-}$	12.8	5.0	5.7
CrO_4^{2-}	1.3	0.8	3.4
SO_3^{2-}	11.6	4.5	5.8
$S_2O_3^{2-}$	∞	7.5	8.1

*Eluent: 5×10^{-4} M KHP, pH = 6.0 t_o = 1.8 min

Column: 2 mm I.D. x 1000 mm

+Eluent: 1×10^{-4} M KHP, pH = 7.1 t_o = 1.5 min

Column: 3 mm I.D. x 500 mm

Figure Captions

Figure 1. Schematic representation of the liquid chromatograph.

Figure 2. Separation of 4.8 ppm fluoride, 5.1 ppm chloride and 26.0 ppm bromide.

resin: XAD-1 0.04 meq/g

eluent: 6.5×10^{-4} M potassium benzoate, pH = 4.6

Figure 3. Separation of 16.0 ppm nitrite and 12.2 ppm nitrate.

resin: XAD-1 0.04 meq/g

eluent: 6.5×10^{-4} M potassium benzoate, pH = 5.0

Figure 4. Separation of 5.1 ppm chloride, 12.2 ppm nitrate, 39.0 ppm iodide, 44.1 ppm sulfate and 28.6 ppm thiocyanate.

resin: XAD-1 0.04 meq/g

eluent: 6.5×10^{-4} M KHP, pH = 4.4

Figure 5. Separation of 24.4 ppm nitrate, 22.0 ppm sulfate and 21.3 ppm thiosulfate.

resin: XAD-1 0.04 meq/g

eluent: 5×10^{-4} M ammonium o-sulfobenzoate, pH = 7.3

Figure 6. Separation of 7.7 ppm chloride, 29.5 ppm iodide, 28.6 ppm thiocynate and 16.5 ppm sulfate.

resin: XAD-1 0.007 meq/g

eluent: 1×10^{-4} M KHP, pH = 7.1

Figure 7. Separation of 17.8 ppm chromate, 22.0 ppm sulfate and 21.3 thiosulfate.

resin: XAD-1 0.007 meq/g

eluent: 1×10^{-4} M KHP, pH = 7.1

Figure 8. Separation of 5.1 ppm chloride, 16.0 ppm nitrite, 22.0 ppm sulfate, 26.0 ppm bromide, and 39.5 ppm nitrate.

resin: Vydac SC anion exchange

eluent: 5×10^{-4} M KHP, pH = 6.0

Figure 9. Separation of standard solutions of sulfate, 2.75 ppm to 13.75 ppm, from chloride and nitrate.

resin: XAD-1 0.04 meq/g

eluent: 5×10^{-4} M KHP, pH = 6.2

Figure 10. Separation of 0.37 ppm nitrite from chloride and sulfate in ISU tap water.

resin: Vydac SC anion exchange

eluent: 5×10^{-4} M KHP, pH = 6.0

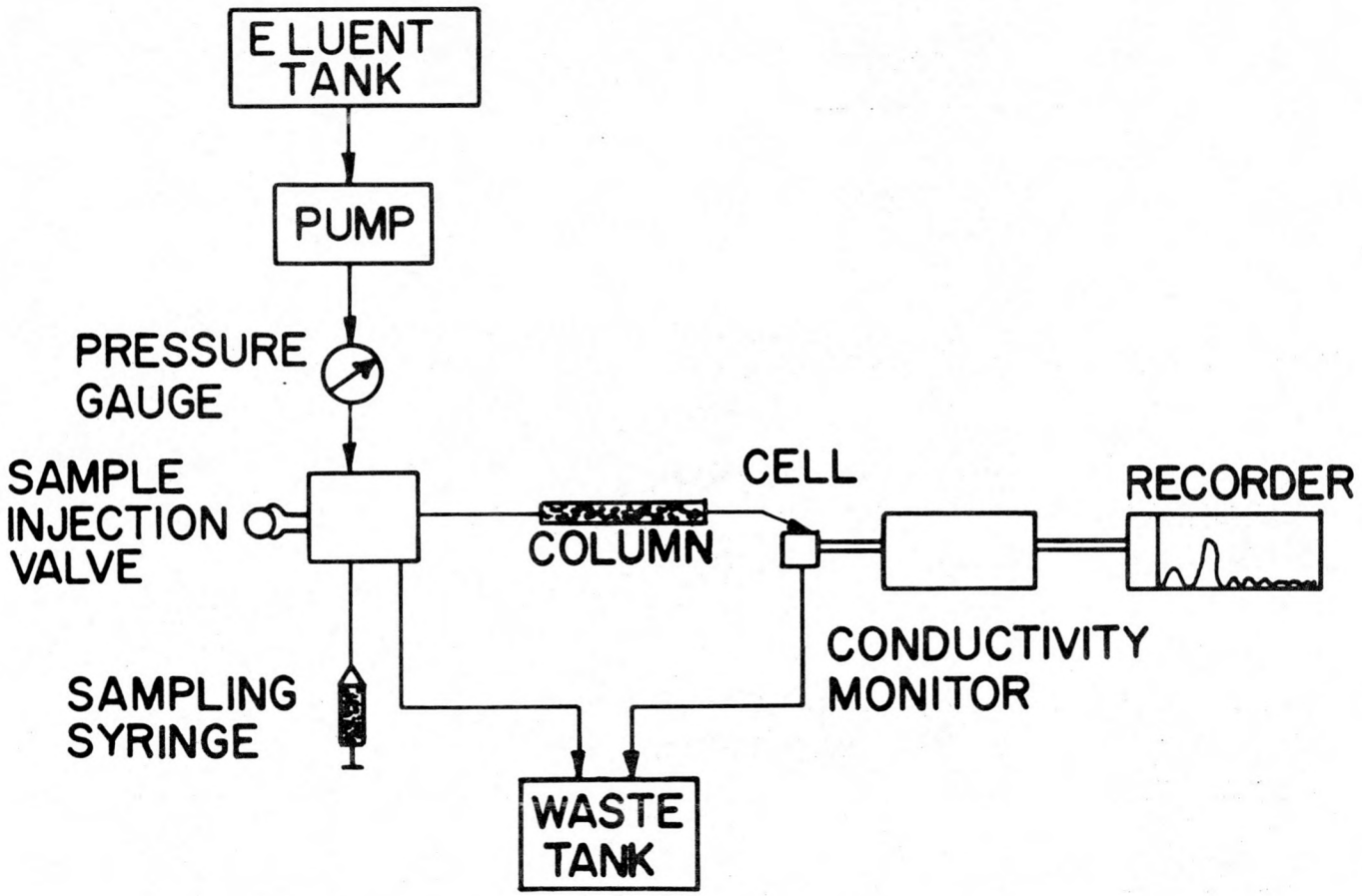

Figure 1. Schematic representation of the liquid chromatograph.

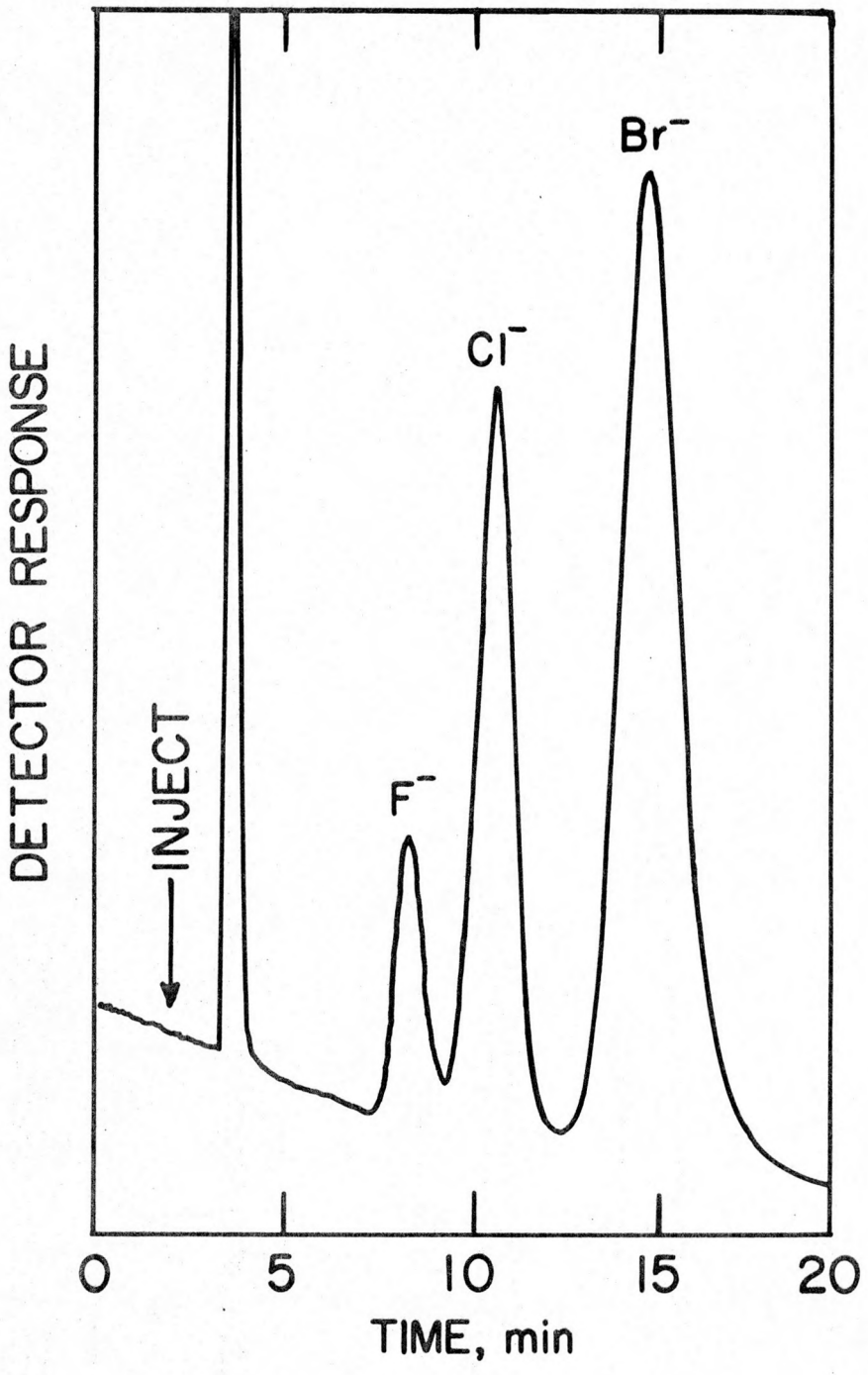

Figure 2. Separation of 4.8 ppm fluoride, 5.1 ppm chloride and 26.0 ppm bromide.
resin: XAD-1 0.04 meq/g
eluent: 6.5×10^{-4} M potassium benzoate, pH = 4.6

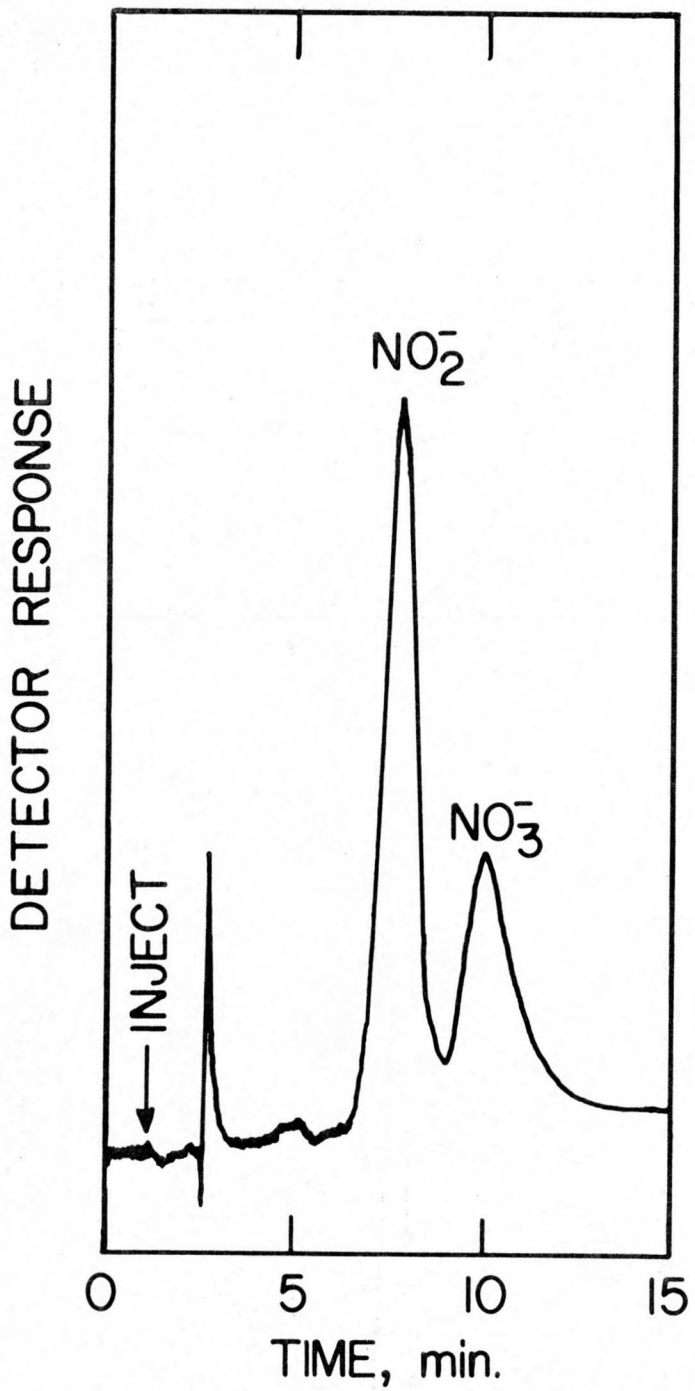

Figure 3. Separation of 16.0 ppm nitrite and 12.2 ppm nitrate.
resin: XAD-1 0.04 meq/g
eluent: 6.5×10^{-4} M potassium benzoate, pH = 5.0

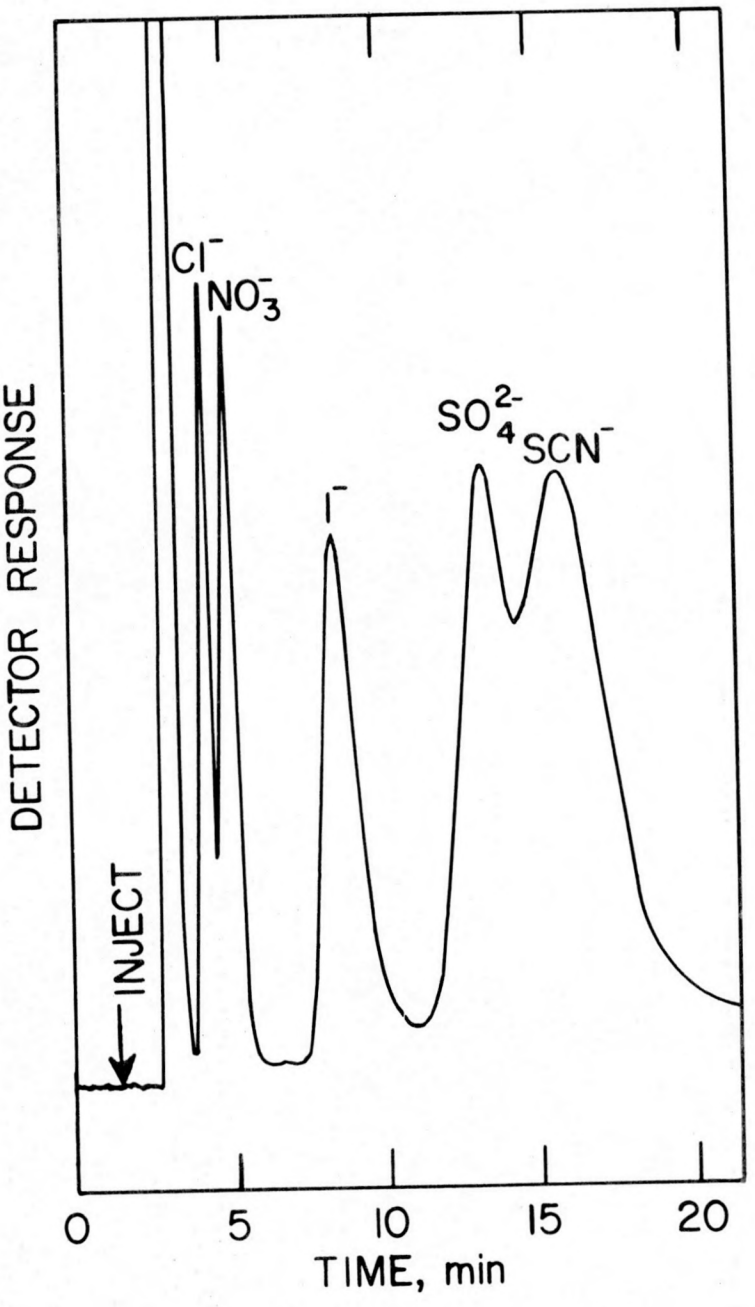

Figure 4. Separation of 5.1 ppm chloride, 12.2 ppm nitrate, 39.0 ppm iodide, 44.1 ppm sulfate and 28.6 ppm thiocyanate.
resin: XAD-1 0.04 meq/g
eluent: 6.5×10^{-4} M KHP, pH = 4.4

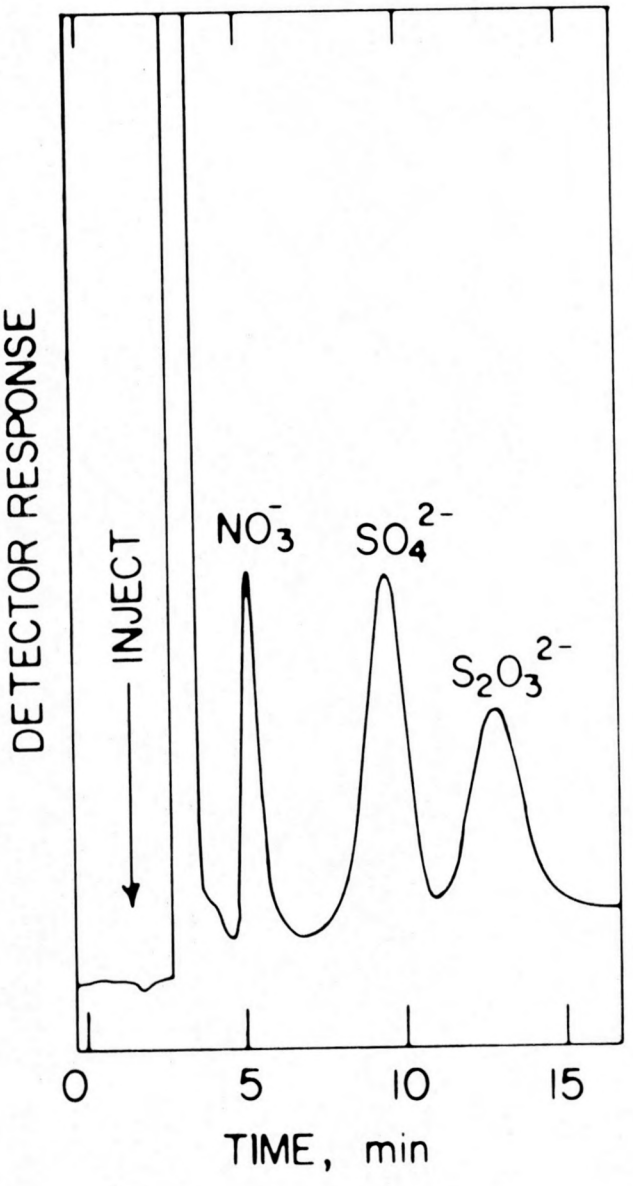

Figure 5. Separation of 24.4 ppm nitrate, 22.0 ppm sulfate and 21.3 ppm thiosulfate.
resin: XAD-1 0.04 meq/g
eluent: 5×10^{-4} M ammonium o-sulfobenzoate, pH = 7.3

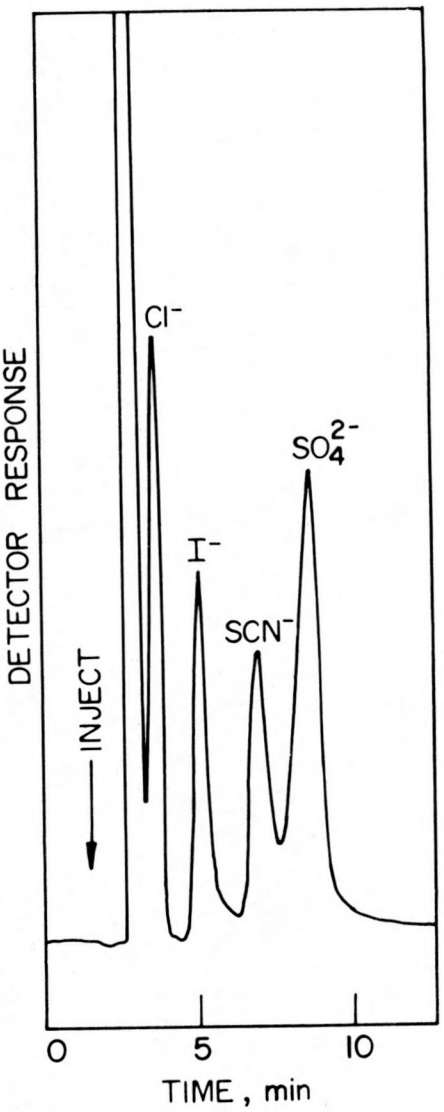

Figure 6. Separation of 7.7 ppm chloride, 29.5 ppm iodide, 28.6 ppm thiocynate and 16.5 ppm sulfate.
resin: XAD-1 0.007 meq/g
eluent: 1×10^{-4} M KHP, pH = 7.1

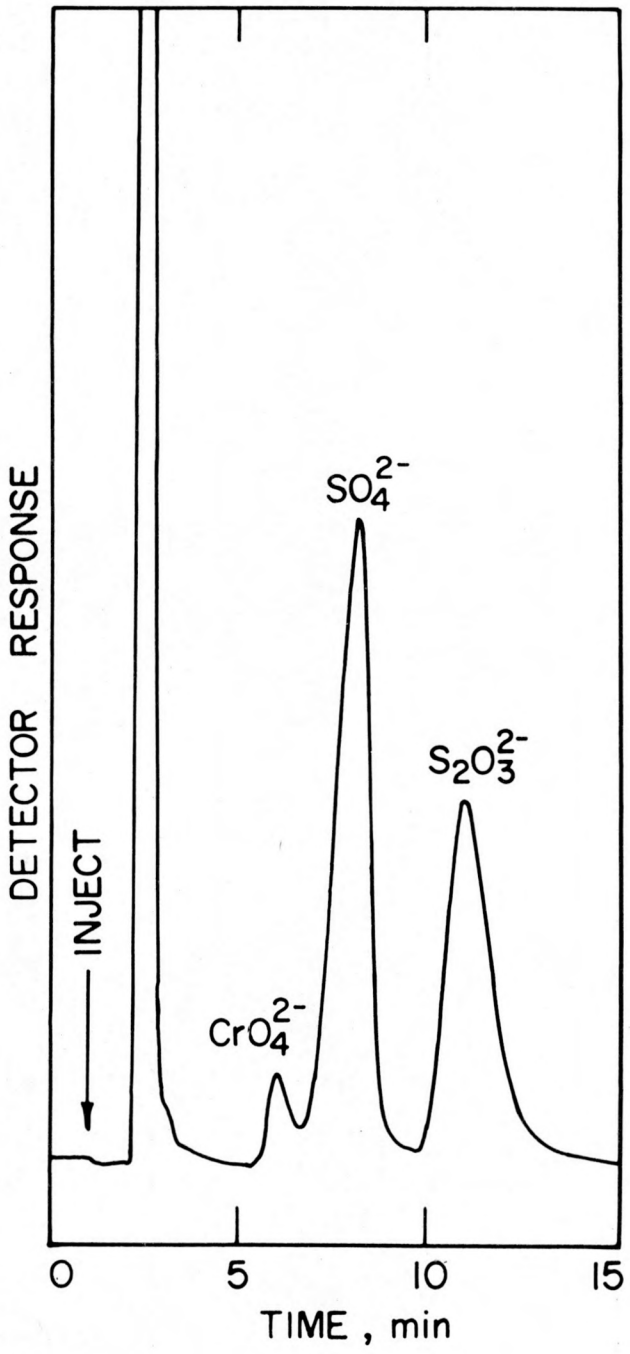

Figure 7. Separation of 17.8 ppm chromate, 22.0 ppm sulfate and 21.3 thiosulfate.
resin: XAD-1 0.007 meq/g
eluent: 1 x 10^{-4} M KHP, pH = 7.1

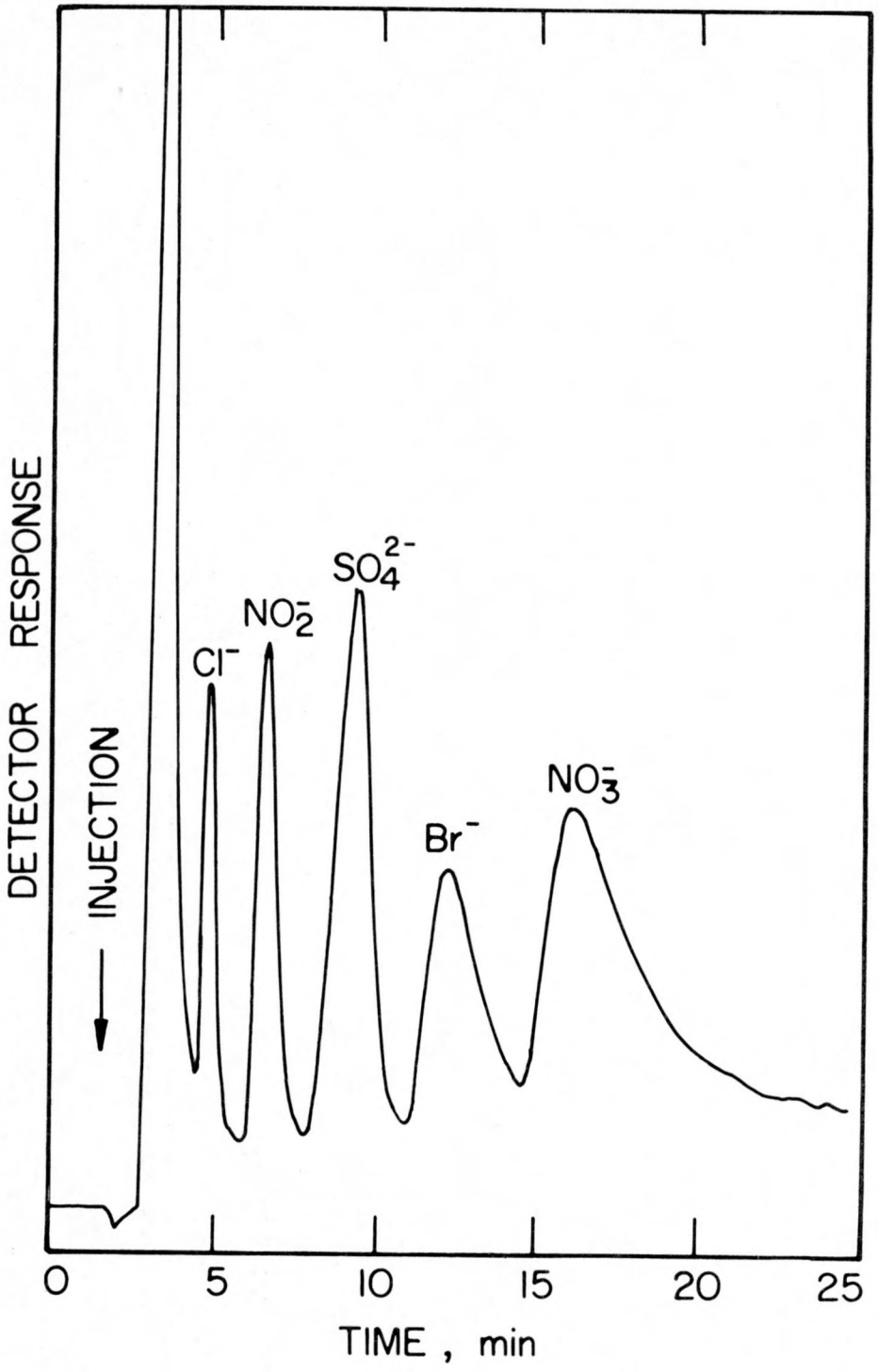

Figure 8. Separation of 5.1 ppm chloride, 16.0 ppm nitrite, 22.0 ppm sulfate, 26.0 ppm bromide, and 39.5 ppm nitrate.
resin: Vydac SC anion exchange
eluent: 5×10^{-4} M KHP, pH = 6.0

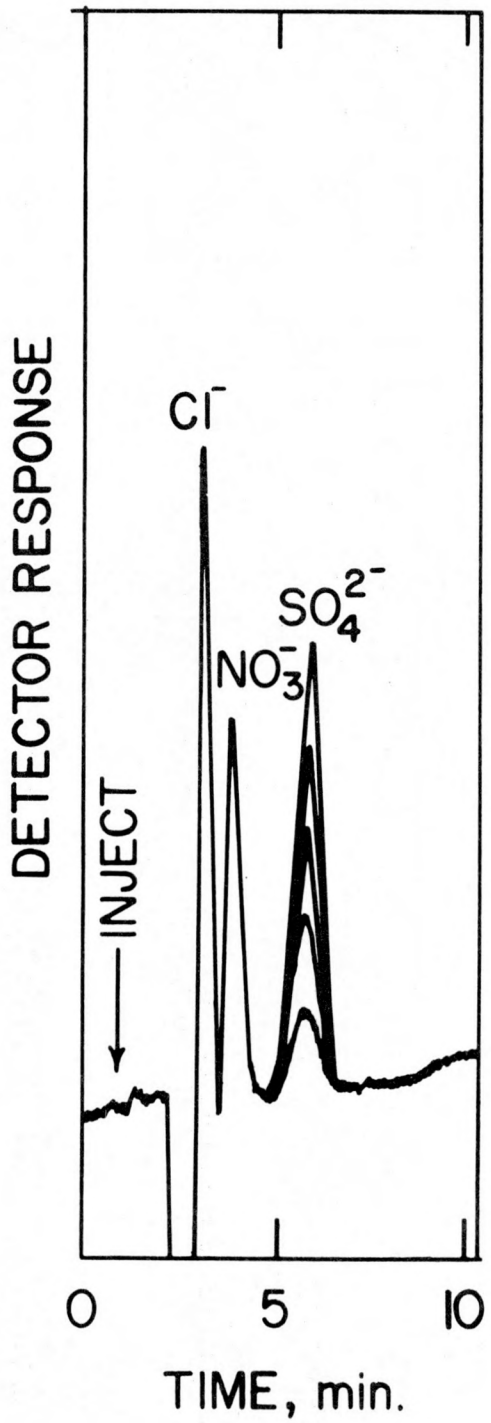

Figure 9. Separation of standard solutions of sulfate, 2.75 ppm to 13.75 ppm, from chloride and nitrate.
resin: XAD-1 0.04 meq/g
eluent: 5×10^{-4} M KHP, pH = 6.2

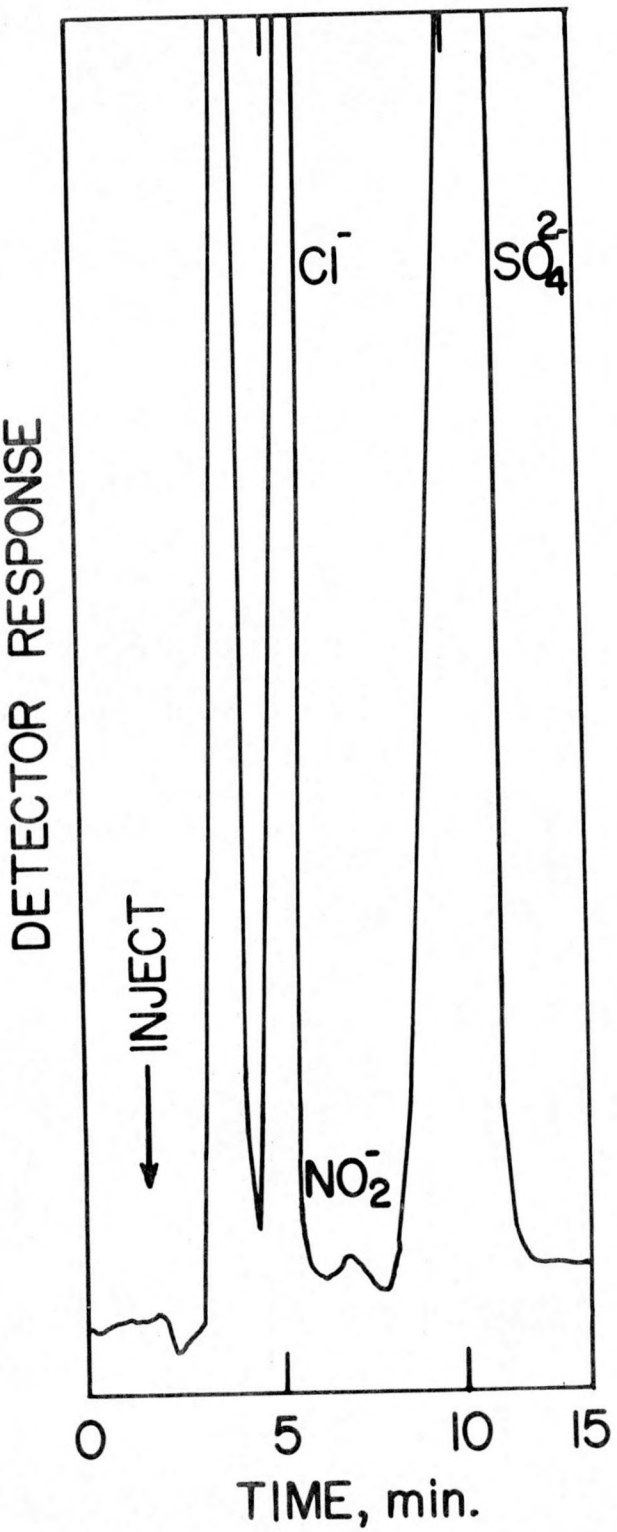

Figure 10. Separation of 0.37 ppm nitrite from chloride and sulfate in ISU tap water.
resin: Vydac SC anion exchange
eluent: 5×10^{-4} M KHP, pH = 6.0