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**REPORT**

**NIM- No. 2132**

**THE ASSESSMENT OF PELLICULAR  
ANION-EXCHANGE RESINS FOR THE  
DETERMINATION OF ANIONS  
BY ION CHROMATOGRAPHY**

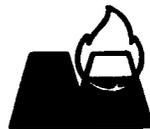
by

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30th October, 1981



# NATIONAL INSTITUTE FOR METALLURGY

ANALYTICAL CHEMISTRY DIVISION

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THE ASSESSMENT OF PELLICULAR ANION-EXCHANGE RESINS FOR  
THE DETERMINATION OF ANIONS BY ION CHROMATOGRAPHY

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## **SYNOPSIS**

Because pellicular anion-exchange resins suitable for the determination, by ion chromatography, of anions with alkaline eluents were unavailable in South Africa at the inception of this work, an attempt was made to prepare such resins. In this study it is shown that the pellicular resins produced are more efficient than the surface-aminated resins used previously. The simultaneous separation and determination of five common anions is demonstrated. The method was applied to the analysis of uranium leach liquors, effluent samples, and a solid sample of ferric oxide (goethite).

## **SAMEVATTING**

Omdat pellikulêre anioonruilharse wat geskik is vir die bepaling van anione met alkaliese uitwasmiddels deur ionchromatografie, nie by die aanvang van hierdie werk in Suid-Afrika beskikbaar was nie, is daar 'n poging aangewend om sulke harse te berei. In hierdie studie word daar getoon dat die pellikulêre harse wat gemaak word, doeltreffender is as die oppervlakgeamioneerde harse wat vroeër gebruik is. Die gelyktydige skeiding en bepaling van vyf gewone anione word beskryf. Die metode is toegepas op die ontleding van uraanloogvloei-stowwe, monsters van uitvloei-sels en 'n vaste ferrioksied(goethiet)monster.

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## 1. INTRODUCTION

The determination, by automatic conductimetry, of various anions was first introduced by Small *et al.*<sup>1</sup>, who used pellicular anion-exchange resins and basic eluents to achieve separation. With this technique, the conductance of the eluent background is suppressed by neutralization in a strongly acidic cation-exchange column, which simultaneously converts solute anions to their highly conductive acids. The term 'ion chromatography' (IC) was coined by these authors<sup>1</sup> as an 'umbrella' term for all automatic procedures involving the separation of ionic species by chromatographic techniques.

In an attempt to simplify and improve that technique, Gjerde *et al.*<sup>2</sup> proposed a modified approach that involves the separation of anions on surface-aminated, macroreticular resins of very low capacity with very dilute solutions of hydrogen phthalate or benzoate as the eluents. Because of the low conductivity of the eluents employed, a second column is not required for the suppression of eluent conductance.

The latter technique was evaluated in an earlier investigation<sup>3</sup> at NIM, and promising results were obtained for a number of anions. However, the resins employed lacked efficiency and only three anions could be separated and determined simultaneously. Also, an eluent different from that used for the separation of, e.g., phosphate and sulphate, had to be used to effect the separation of fluoride. The commercially available pellicular Vydac 301 SB resin that was tested in that investigation gave good separations of various common anions, but did not allow the determination of fluoride. Pellicular resins with a polystyrene core, which are suitable for the separation of anions, were not available in South Africa at that stage. It was therefore decided that an attempt should be made to prepare resins that would allow the separation, in one elution, of mixtures of anions such as fluoride, chloride, nitrate, phosphate, and sulphate. Initial experiments involved the use of surface-aminated XAD-1 resins in which the particle size had been decreased considerably. This resulted in difficulties in the manual packing of the columns and in a build-up of pressure within the chromatographic system. Another attempt involved gentle chloromethylation and subsequent gentle amination of a spherical polymer of the gel type (from Diamond-Shamrock, U.S.A.), which has a definite particle size (20 to 40  $\mu\text{m}$ ). This resin was easily packed, and the columns could be operated at relatively low back-pressures. However, although fluoride and chloride could be separated on this resin, its overall performance was poor, and band broadening and tailing were observed. A potentially useful material for the column was prepared by the coating of TEE SIX (Teflon that is 150 to 170 mesh in size, from Analab, U.S.A.) with 2 per cent Aliquat 336. With an eluent consisting of potassium hydrogen phthalate, there was early elution of most anions, but no separation took place. A  $10^{-3}$  M solution of sodium phenolate achieved the separation of a number of anions (Figure 1), a cationic-suppressor column being employed to reduce the background conductivity of the eluent. Because a considerable length of time would have been needed for this approach to be optimized and for more TEE SIX to be obtained, it was decided that an attempt should be made instead to prepare a pellicular resin similar to that first described by Small *et al.*<sup>1</sup>.

## 2. PREPARATION OF THE RESIN

Initially, it was thought that a spherical anion-exchange resin could be produced of which the particles would be small enough to serve as the surface layer of a pellicular resin. However, it soon became apparent that the stirring rates obtainable with the existing equipment during polymerization were too low for suitably small beads to be produced; instead, relatively large beads were obtained varying widely in size. These beads were then ground and used in the preparation of pellicular resins, as is described in detail in the next sections. In the preparation of the pellicular anion-exchange resin, advantage was taken of the strong electrostatic interaction between polycationic and polyanionic particles, i.e. of their tendency to stick to one another. A resin consisting of beads with small diameters that have functional groups of sulphonic acid attached to their surfaces was treated with an aqueous suspension of strongly basic anion-exchange particles, so that a thin layer of these particles surrounded each resin bead. This kind of resin has the advantage over conventional resin of having much-reduced diffusion paths, which result in faster exchange (i.e. mass transfer) of the solutes and, consequently, in greatly improved performance of the column.

### 2.1. Preparation of the Anion-exchange Resin

Four steps were involved in this preparation: polymerization, chloromethylation, amination, and grinding of the finished product in a Siebtechnik mill.

DN 14 (0,24 g), Natrosol (0,1 g) (both of which are suspension stabilizers), and calcium chloride (0,5 g) were dissolved, in that order, in 40 ml of pre-warmed water. The reaction vessel was placed in a water-bath, the temperature being raised to 88°C, and stirred while a monomer mixture consisting of 25 g of styrene, 3 g of divinylbenzene (DVB), and 0,1 g of benzoyl peroxide was added. The stirring rate was then increased so

that the resin beads produced would be as small as possible. After approximately 45 minutes, the beads became 'sticky' and then solidified. The beads were reacted for a further 18 hours at 88°C. After filtration, the beads were washed with acetone and then with water, and dried at 80°C in an air-oven.

To a mixture consisting of 75 ml of chloromethyl methyl ether and 75 ml of dichloromethane, 26 g of the polystyrene beads, prepared as described, were added. During stirring, 7.3 ml of tin-IV-chloride was added drop by drop to avoid violent reaction. The temperature was maintained at 40°C, and the mixture allowed to react for 6 hours. The reaction was then quenched by the addition of methanol, and the chloromethylated beads were filtered on a suction filter. After washing with methanol, the beads were dried at room temperature. The chloromethylated resin beads were placed in a reaction vessel containing 50 ml of methylal (dimethoxy methane) and the mixture stirred, 60 g of tri-methylamine being added drop by drop. The beads were then allowed to react for 8 hours at a temperature of 40°C. The aminated resin beads were filtered and washed, first with methanol, and then with water. The nitrogen content of the resin was determined by use of an elemental analyser, and the capacity of the resin was calculated as 3.72 meq/g of dry resin.

The resin was then mixed with water and ground for 15 minutes in a Siebtechnik mill. The slurry obtained was transferred to a measuring cylinder and the larger particles allowed to settle by sedimentation. The solution containing the finer particles was decanted, a portion being rotated in a centrifuge for 10 minutes at 4000 r/min. The solution was then decanted from the sediment, and the solution containing the suspended particles was passed through a no. 4 sintered-glass crucible. The filtrate was milky in appearance because of the very fine anion-exchange particles in suspension. These particles were considered small enough to form a thin layer on a surface-sulphonated resin.

## 2.2. Preparation of a Superficial Cation-exchange Resin

An unreacted polystyrene-DVB copolymer consisting of spherical beads of 20 to 40  $\mu\text{m}$  diameter (from Diamond-Schamrock, U.S.A.) was used as the starting material. A surface-sulphonated resin was prepared by the stirring of 2 g of copolymer with approximately 5 ml of concentrated sulphuric acid at room temperature for 5 to 10 minutes. After removal of the sulphuric acid by filtration, the resin was washed with a large quantity of water and transferred to a column (2.5 cm in diameter) with a sintered-glass frit at the bottom.

## 2.3. Preparation of the Pellicular Resin

A very dilute solution of the previously prepared anion-exchange resin in suspension was passed over the resin beads until the solution leaving the column had the same milky appearance as that added. This indicated that the surface-sulphonated (cation-exchange) beads were saturated. After washing with water to remove excess anion-exchange particles and a rinse with an 0.01 M solution of sodium hydroxide, the pellicular anion-exchange resin (designated resin I) was ready for use.

The lengthy preparation time for the strongly basic anion exchanger was later avoided by the use of the commercially available anion-exchange resin Zeolit FF(ip). The Zeolit FF(ip) resin was ground and used for the preparation of the pellicular anion-exchange resin as described in Sections 2.1 and 2.2. The resin obtained in this manner was designated resin II.

Scanning electron micrographs showed that the resin surface consisted of irregularly shaped anion-exchange particles of less than 0.5  $\mu\text{m}$  diameter. It was also observed that these particles occasionally had a tendency to form clusters.

## 3. CHROMATOGRAPHIC PROCEDURE

The apparatus described previously<sup>3</sup> was used for the chromatographic separations. Precision-cut glass columns, 3 mm by 300 mm, were used for anion separation; the columns used for suppression of the eluent were 2 mm wide and 300 mm long and were packed manually by the injection of a resin slurry. The packed columns were connected into the chromatographic system so that the eluent had to pass through the pellicular anion-exchange resin before passing through the suppressor column, which contained a strongly acidic cation-exchange resin (BIO.RAD AG 50W-X8) (Figure 2). Basic eluents containing sodium bicarbonate or sodium carbonate (or mixtures of the two) were pumped through the system at a rate of 1.5 to 2 ml/min until a stable base-line, as indicated on a chromatographic recorder, was obtained. The reactions that take place in the suppressor column (after, e.g., chloride and nitrate have been separated) when sodium bicarbonate is used as the eluent, are as follows:

for the eluent,  $R-SO_3^- H^+ + Na^+ HCO_3^- \rightleftharpoons R-SO_3^- Na^+ + H_2CO_3$ , and

for the anions,  $R-SO_3^- H^+ + Na^+ Cl^-$ ,  $Na^+ NO_3^- \rightleftharpoons R-SO_3^- Na^+ + H^+ Cl^-$ ,  $H^+ NO_3^-$ .

Thus, the chloride and nitrate ions reach the detector as their highly conducting acids in a matrix of essentially molecular carbonic acid (having low conductance). Sensitive signals are obtained for the separated species, which are recorded in the form of a chromatogram. The concentration of a separated anion was gauged by measurement of the peak height in millimetres, and this was then compared with the peak heights obtained for known amounts of the same anion separated under identical conditions.

The resin in the suppressor column was replaced when it was exhausted, i.e. when there was a sharp increase in background conductivity, and the resin was collected for regeneration at a later stage.

#### 4. SEPARATIONS POSSIBLE ON PELLICULAR RESINS

The separation of fluoride, chloride, nitrate, phosphate, iodide, and sulphate was achieved on resin I when  $5 \times 10^{-4}$  M sodium carbonate was used as the eluent (Figure 3). Separations obtained on resin II (Figure 4) were similar, except that iodide was firmly retained by the resin and could not be determined. Since the active groups are the same in resin I and resin II (quaternary ammonium), it appears that the stronger attraction of iodide to resin II is a function of the hydrocarbon matrix of the resin particles used to form the pellicular layer. However, apart from the behaviour of the iodide, it was found that the elution order for the solutes tested was the same on both resins. Generally, it was observed that the preparation of the pellicular resins was not exactly reproducible, and adjustments had to be made to the eluent concentration each time a new resin was used. The different eluents are placed here in order of increasing displacement potentials: sodium hydroxide, sodium bicarbonate, sodium carbonate, and sodium phenolate. With the exception of the separations shown in Figure 1, sodium phenolate was not used in this investigation because of its tendency to form oxidation products harmful to the performance of the separation column<sup>4</sup>. When a  $10^{-3}$  M solution of sodium bicarbonate, representing a relatively weak eluent, was used, the separation of nitrite from chloride and from nitrate or bromide (which had similar retention times) was achieved (Figure 5). The eluent was too weak to elute species with a lengthy retention time (i.e. phosphate and sulphate) within a reasonable time period. Calibration graphs obtained for fluoride, chloride, nitrite, nitrate, and bromide are shown in Figures 6 and 7, from which it can be seen that the peak heights obtained were proportional to the concentration of these anions in the eluate. The slightly scattered results obtained for chloride were probably because a small negative peak coincided with its elution. The origin of this negative peak is not clearly understood, although it appears to be related to the volume of the sample solution injected, i.e. the amount of water present. The negative peak can be minimized by the addition of eluent salt to the sample solution so that the sample and the eluent contain similar concentrations of eluent salt.

Unsuccessful attempts were made to separate all anions examined so far in one elution and within a reasonable time by the use of mixed bicarbonate-carbonate and hydroxide-carbonate solutions. Invariably, the separation of fluoride and chloride was incomplete, and chloride and nitrite eluted in one peak. However, the use of a mixed bicarbonate-carbonate eluent at an increased flowrate (25 per cent of the pump capacity instead of 15 per cent) decreased the analysis time for fluoride, chloride, nitrate, phosphate, and sulphate from approximately 30 minutes (Figures 3 and 4) to 22 minutes (Figure 8). It can be seen that the increase in flowrate decreased the retention time of the solute anions without appreciably affecting their resolution.

The separation and determination of weak-acid anions, e.g., carbonate, borate, and cyanide, was not possible with the resins and eluents used. This was mainly because these anions were converted to their molecular acids in the suppressor column, which resulted in extremely poor conductimetric detection limits. The determination of some weakly ionized species was achieved by ion-exclusion chromatography in an earlier investigation<sup>5</sup>.

As was to be expected, large amounts of mineral acids in a sample caused overloading of the separation column, with the result that meaningful chromatograms of solute anions were no longer obtainable. A solid sample cannot therefore be dissolved in a mineral acid unless the concentration of anions to be determined is high enough to allow for subsequent dilution with large amounts of water.

Three groups of metals were tested with respect to their effect on the separation of anions. The first of these included those metals likely to form complexes with anions, the second group included metals that are precipitated at an elevated pH value, and the third group comprised those metals present as anionic complexes.

Since aluminium is known to form strong fluoride complexes, tests were done on the effect of this metal on the determination of fluoride. The addition of 100 p.p.m. of aluminium to a standard solution containing 0.3 p.p.m. of fluoride at a pH value of 5 resulted in a slight decrease in the peak height of the latter, indicating incomplete ionization. To induce complete ionization of any aluminium fluoride complexes formed, the pH value of the sample solution was raised to 10. As had been expected, the original peak height was restored, and no interference from aluminium was observed. However, metal ions likely to precipitate in the basic environment of the sample or the eluent, i.e. aluminium, iron, nickel, uranium, etc. (although not immediately affecting the separation of anions) slowly collected at the entrance to the separation column. In time, discolouration of the resin bed, deterioration of the performance of the column, and a build-up of pressure within the chromatographic system occurred, and the separation of anions was no longer possible.

Ficklin<sup>6</sup> demonstrated that trace quantities of metal anions, such as tungstate and molybdate could be determined by a similar ion-chromatographic technique. It was shown in this investigation that relatively large quantities of these metals (i.e. approximately 100 p.p.m.) seriously overloaded the column, and other anions present could no longer be determined. Also, because of the affinity of these metals for the pellicular resins tested, elution was difficult because of extensive tailing. No attempt was made at this stage to overcome interferences caused by, e.g., tungsten. However, metal cations, particularly if present in large amounts, were removed in all further experiments by precipitation of their hydroxides or by a cation-exchange procedure prior to the chromatographic determination of anions.

## 5. APPLICATION OF THE PROCEDURE

The procedure was applied to the determination of various anions in a uranium leach liquor, in effluent water, and in a sample of goethite. Preparation of the aqueous sample solutions involved only appropriate dilution with water so that the anions present would not exceed the range of measurement. To the uranium leach liquor enough sodium hydroxide solution (0.01 M) was added to precipitate the metal cations present. The precipitate was allowed to settle and, after decantation, aliquot portions of the solution were analysed by use of the chromatographic procedures.

A small mass (0.25 g) of the goethite sample ( $Fe_2O_3$ ) was dissolved in a few millilitres of 3 N hydrochloric acid, and the solution evaporated to approximately 1 ml. After dilution to 50 ml, the iron was separated in a small, strongly acidic cation-exchange column at atmospheric pressure. The eluate containing the sulphate was diluted to 400 ml, and aliquot portions of 100  $\mu$ l injected into the chromatographic system. The dissolution of the sample in hydrochloric acid was permissible in this instance because the high concentration of sulphate (approximately 5 per cent) allowed for dilution with large amounts of water. Also, the retention times of chloride and sulphate are sufficiently different to permit a relatively large excess of the former (Figure 9).

Results obtained with the resins and eluents used in this investigation compared favourably with those obtained by wet-chemical procedures and with results from the Dionex Model 10 Ion Chromatograph (Table 1), which at this stage had recently become available in South Africa.

## 6. DISCUSSION

The pellicular resins prepared in this investigation were found to be stable in the alkaline eluents employed and retained their characteristics even after prolonged use. The base-lines obtained were more stable than those achieved with the previously employed surface-aminated resins, when solutions of phthalate, benzoate, and citrate were used as eluents<sup>3</sup>, and five anions could be separated instead of three. Also, presumably because of reduced diffusion paths in the pellicular resin, narrower peaks and, consequently, better resolution were obtained.

The preparation of the pellicular resin and the packing of a column, as described in this report, were not always reproducible, and the concentration of the eluent used invariably had to be adjusted to give similar retention times for the various anions on a new column. This difficulty makes the application of the method to routine analytical work somewhat complicated, particularly if the analyst is unfamiliar with the principles involved in the technique. The preparation of 'home-made' pellicular resins is further complicated by the fact that the unreacted spherical polymer beads used as the matrix are not readily available in South Africa.

It is felt that, for practical purposes, the lengthy time needed for preparation of the strongly basic anion-exchange resin (Section 2.1) from a monomer mixture can be omitted, and a commercially available resin used instead for the formation of the pellicular layer. The analysis time for the determination of chloride, nitrate, and sulphate in a uranium leach liquor is 12 hours if wet-chemical procedures are used. The chromatographic procedure (NIM-IC) requires approximately 2.5 hours for the first sample, which

TABLE 1

*The determination of anions in samples*

	Method		
	Wet chem.	NIM-IC	Dionex-IC
<i>Uranium leach liquor</i>			
Chloride, p.p.m.	184	175	170
Nitrate, p.p.m.	-	170	175
Sulphate, g/l	25	23,3	23,3
<i>Effluent water</i>			
Fluoride, p.p.m.	-	3,5	3,5
Chloride, p.p.m.	-	50,0	49,0
Nitrate, p.p.m.	-	21,0	20,5
Sulphate, p.p.m.	-	595	610
<i>Goethite</i>			
Sulphate, %	4,7	4,80	4,82

Wet chem. = Wet-chemical procedure  
 NIM-IC = The ion-chromatographic procedure investigated at this laboratory  
 Dionex-IC = Dionex Ion Chromatography

includes the analysis of three standard solutions for calibration purposes. All further samples require only an additional 30 minutes each. Unfortunately, double the analysis time is required if fluoride (or nitrite) is to be determined, because this anion is not completely separated from chloride (Figure 4) and a different, weaker eluent, i.e. bicarbonate, has to be employed. This is particularly important if very small amounts of fluoride are to be accurately determined.

By comparison, use of the Dionex Ion Chromatograph allows the complete separation, with only one eluent, of fluoride, chloride, nitrite, phosphate, bromide, nitrate, and sulphate well within 25 minutes in a highly reproducible manner (Figure 10). This is achieved by the use of highly efficient resins with a pellicular layer of extremely small, perfectly spherical, anion-exchange particles instead of the layer of irregular and sometimes lumpy particles employed in this investigation. The Dionex system, which evolved from the work of Small *et al.*<sup>1</sup>, therefore offers greater ease of operation, and routine anion analysis can be carried out even by an inexperienced analyst. Also, replacement of the suppressor column, which is required in the NIM-IC system, is not necessary because chromatographs are equipped with an automatic regeneration system that can be put into operation after working hours. In addition to anion analysis, the Dionex system allows the determination of ammonium, alkali metals, alkaline earths, and certain amines on low-capacity cation-exchange resins, which has not been attempted in this investigation.

7. CONCLUSION

The fast and accurate determination, by an inexpensive ion-chromatographic system of various anions on 'home-made' pellicular anion-exchange resins, was demonstrated. Application of the technique to the analysis of actual samples showed that the results obtained were in good agreement with other methods of determination. It was shown that the pellicular resins were more efficient than the previously used surface-aminated resins, and that the number of anions that could be analysed increased from three to five.

However, it is also apparent that these resins are not as efficient and reproducible as those used in commercially available ion chromatographs, which became available in South Africa while this work was in progress. Therefore, although the investigation undertaken at NIM was of value in the absence of a more-advanced system, further work on the simulation of existing techniques appears to be unjustified.

8. ACKNOWLEDGEMENT

Thanks are due to Afronix (Pty) Ltd for the loan of the instrument used in this investigation.

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PELLICULAR RESINS

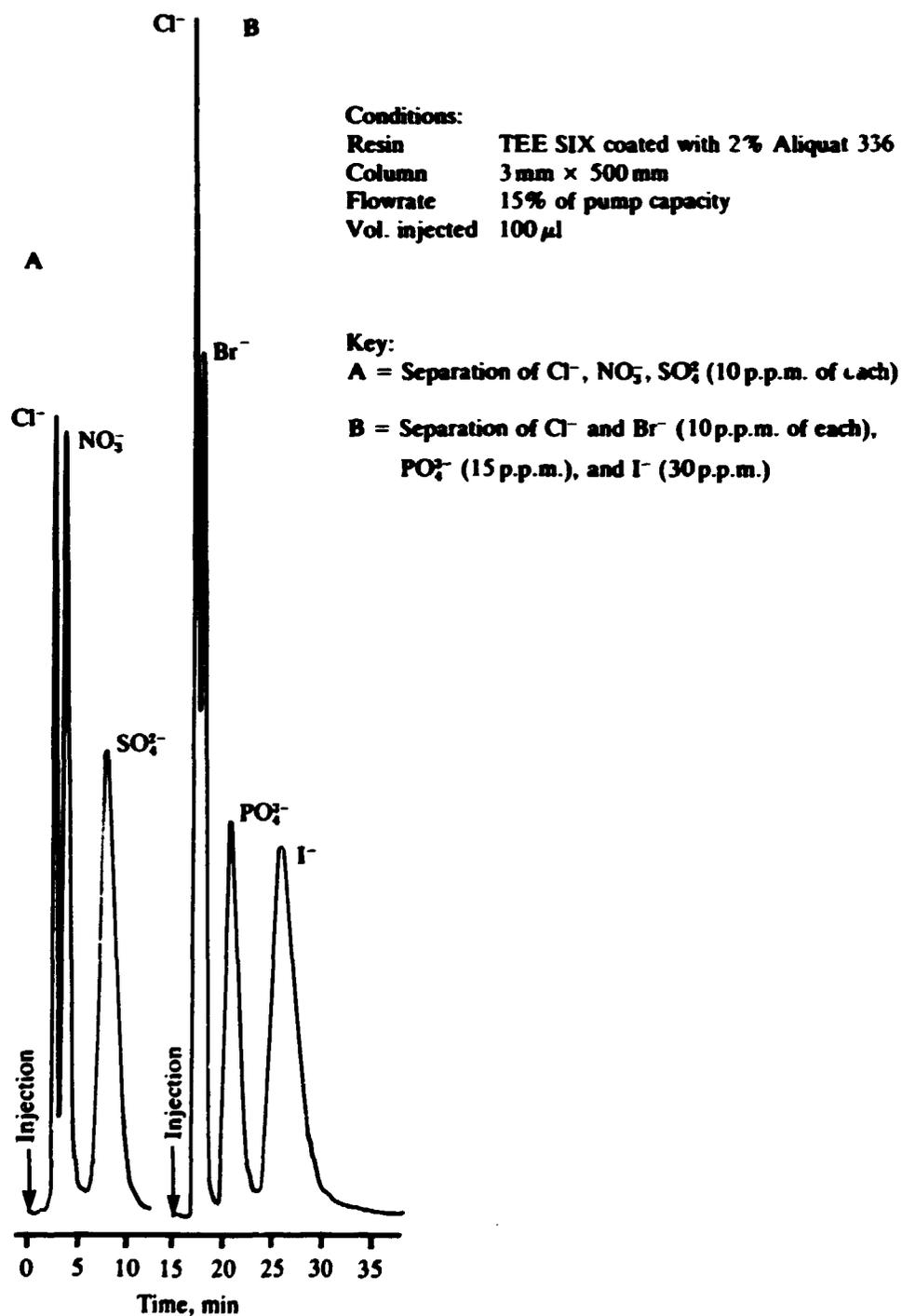


FIGURE 1. Separation of some anions on TEE SIX treated with Aliquat 336

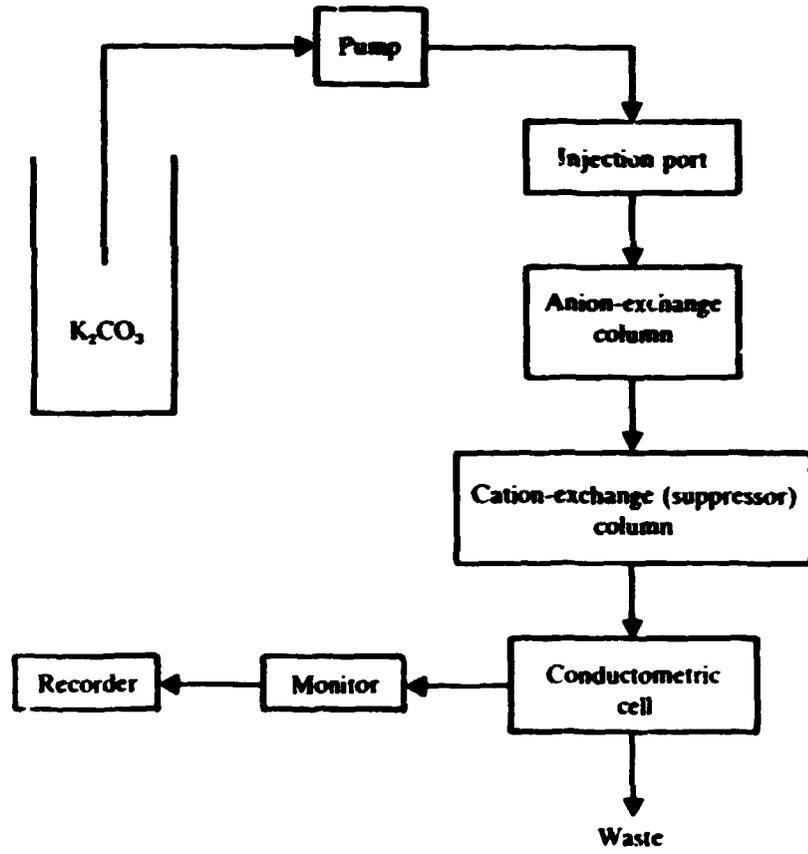


FIGURE 2. System for the determination of anions

PELLICULAR RESINS

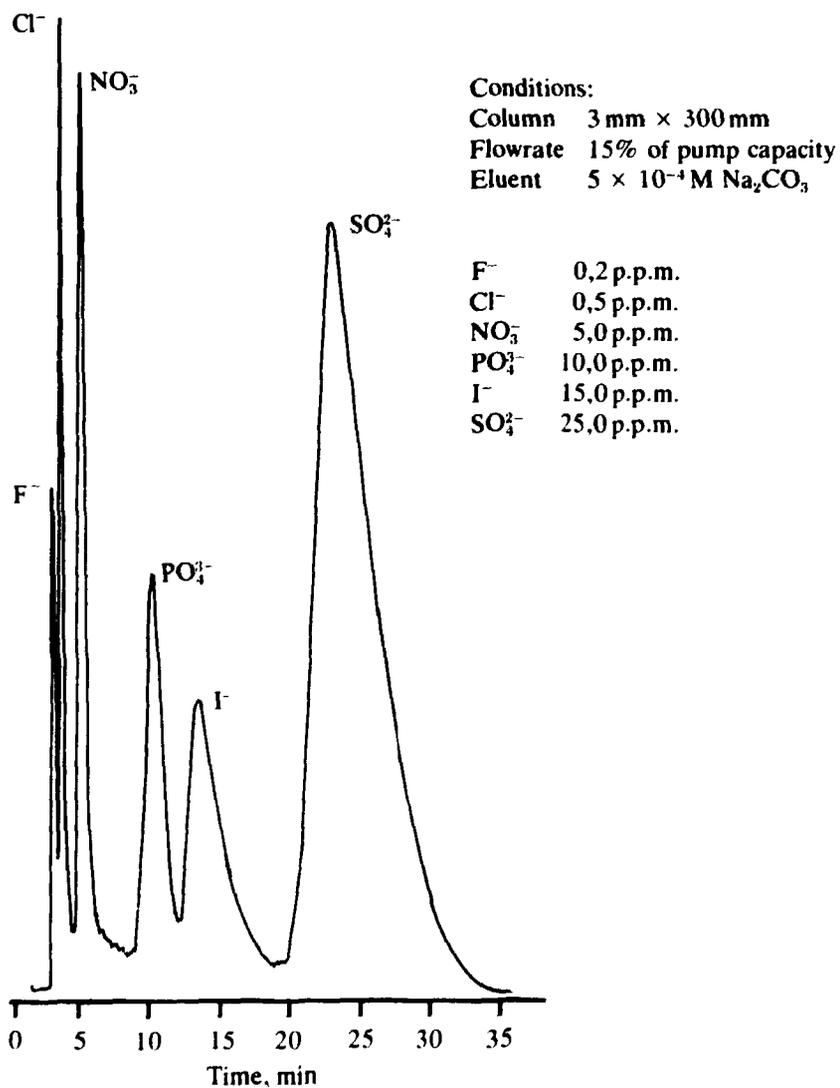


FIGURE 3. The separation of six anions on resin I

PELLICULAR RESINS

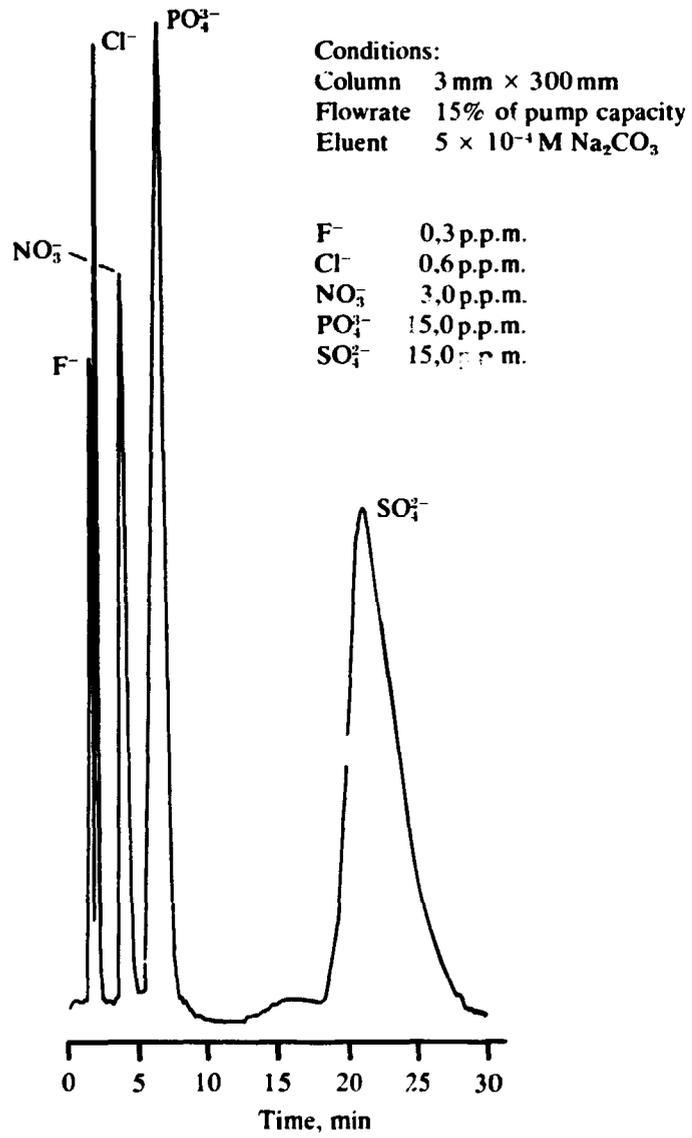


FIGURE 4. The separation of anions on resin II

PELLICULAR RESINS

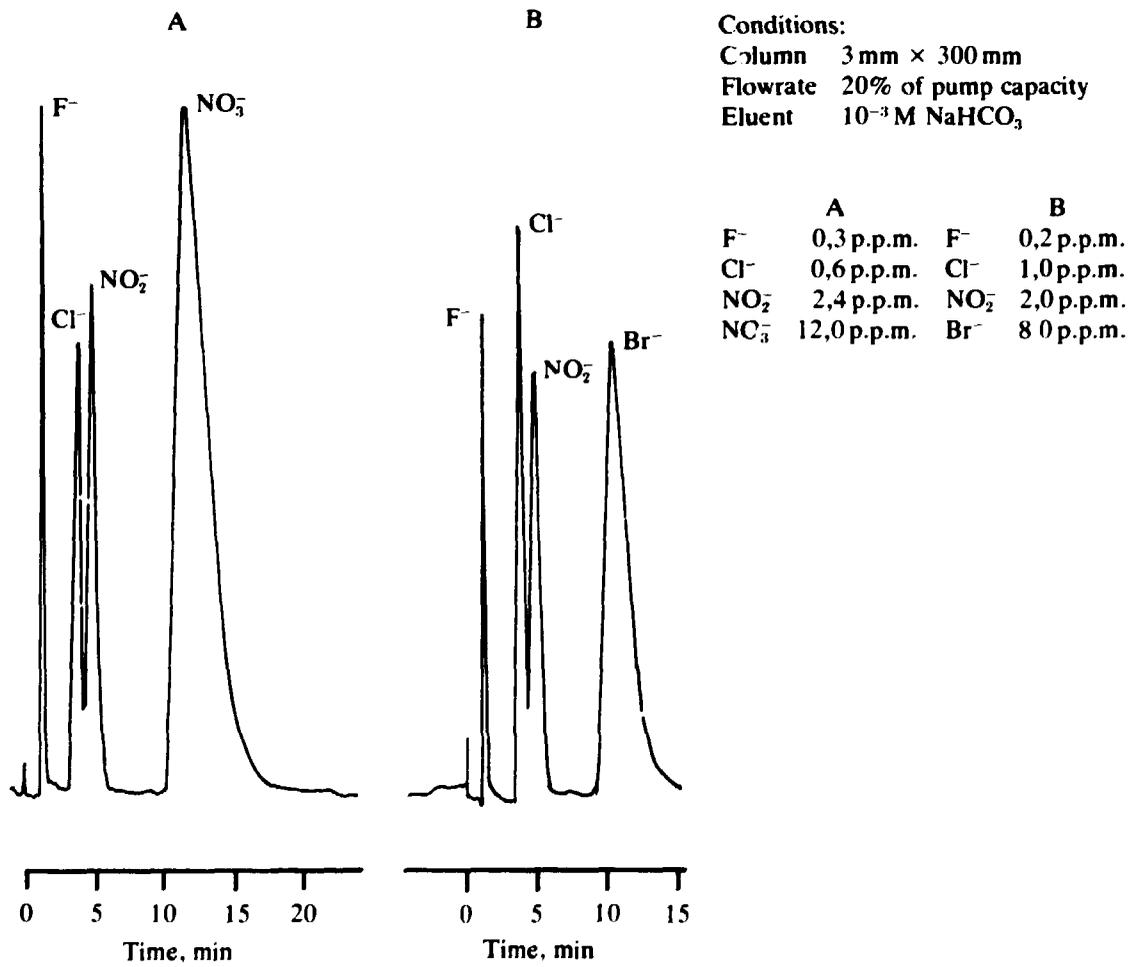


FIGURE 5. The separation of nitrate or bromide from nitrite, chloride, and fluoride

PELLICULAR RESINS

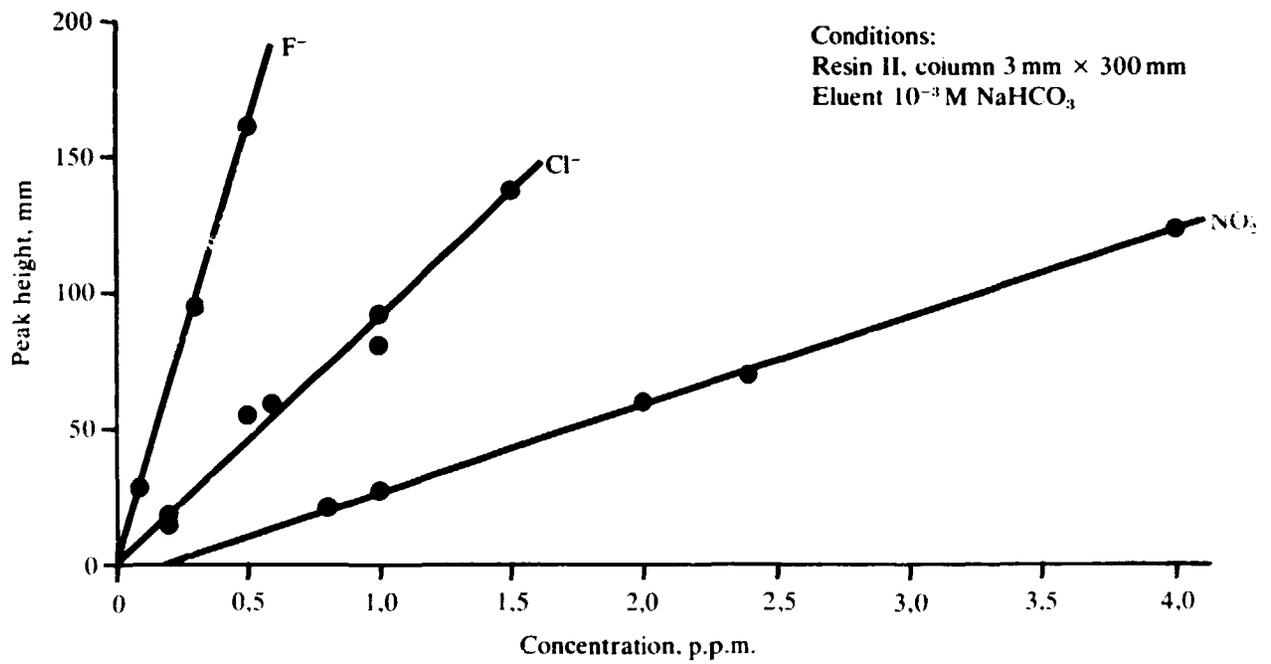


FIGURE 6. Calibration graphs for fluoride, chloride, and nitrate

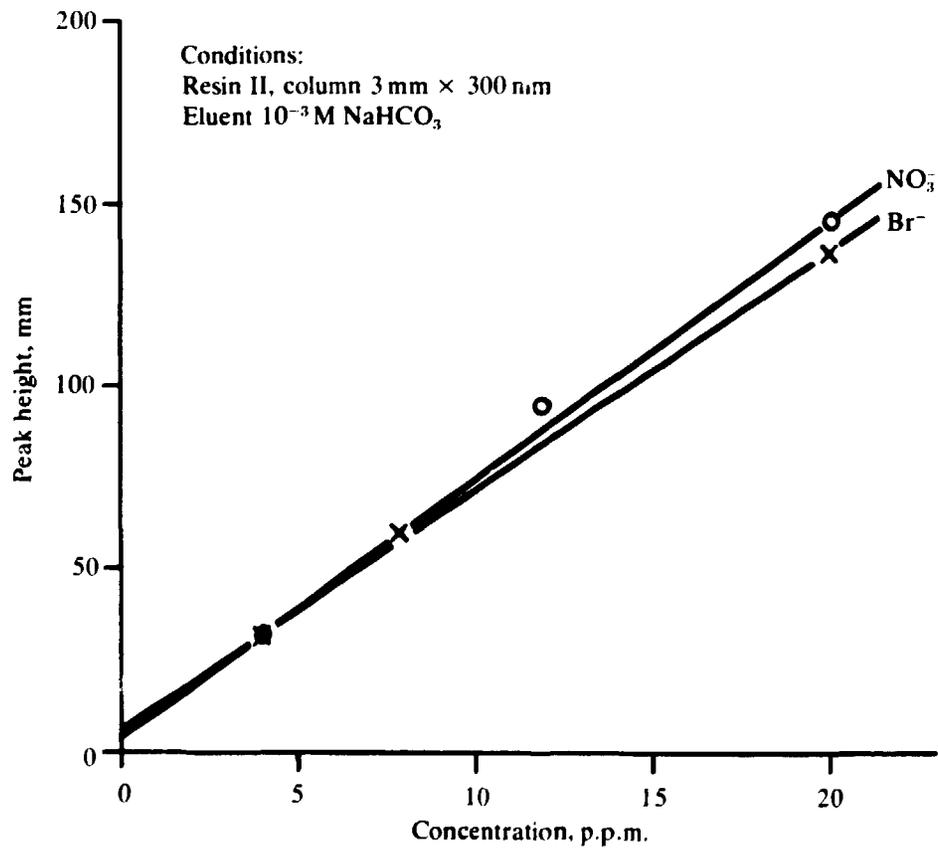


FIGURE 7. Calibration graphs for nitrate and bromide

PELLICULAR RESINS

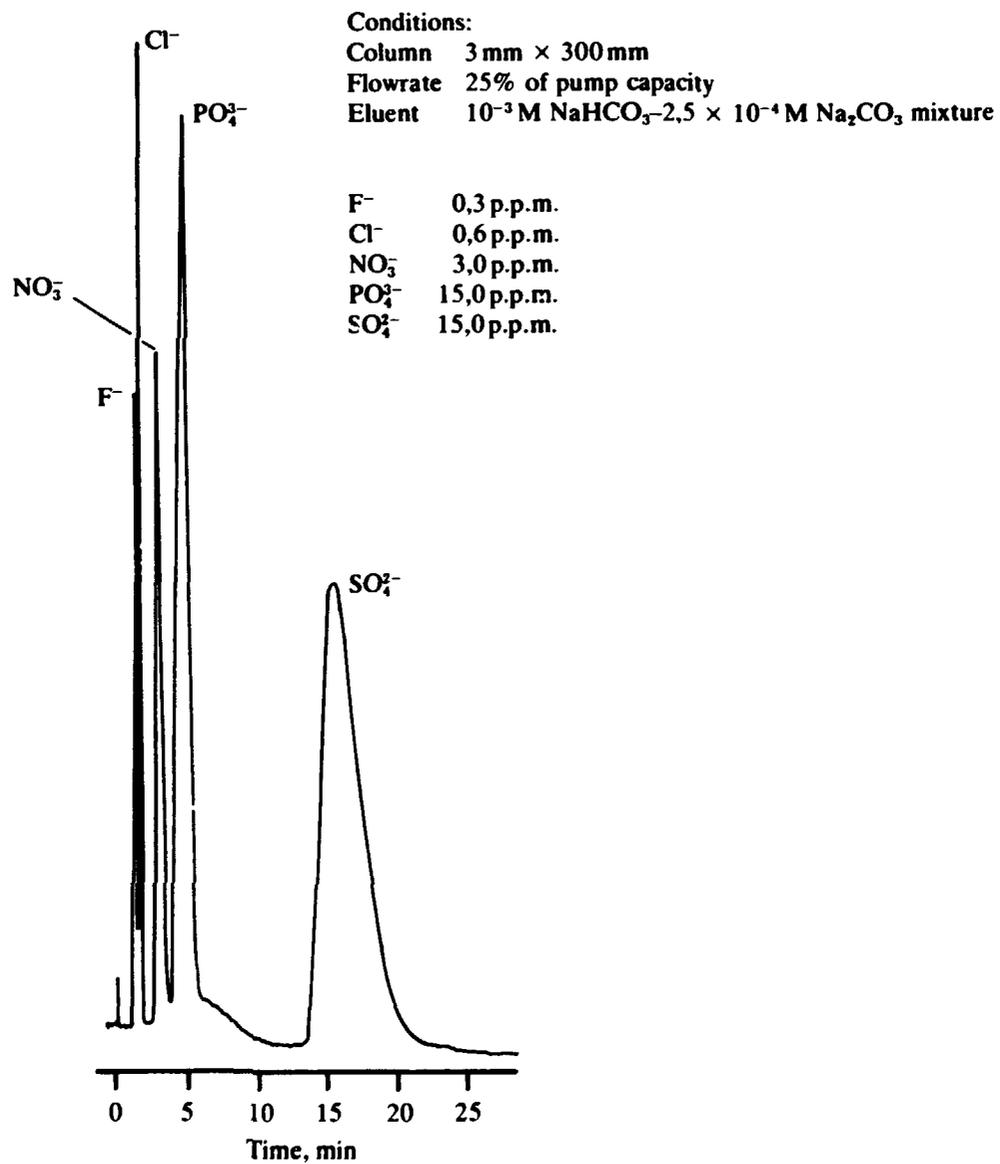


FIGURE 8. The separation of some anions at an increased flowrate

PELLICULAR RESINS

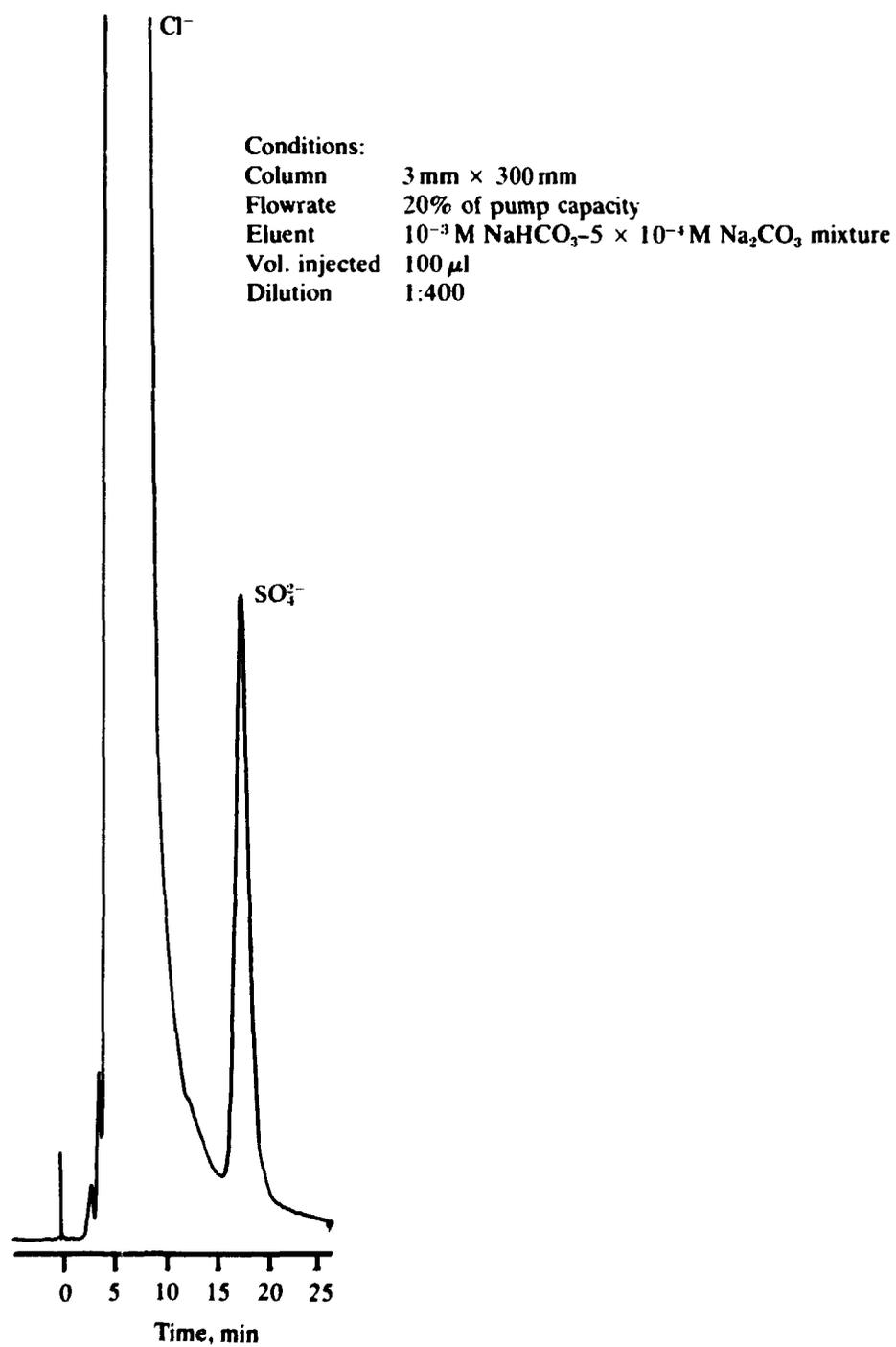


FIGURE 9. The determination of sulphate in goethite

PELLICULAR RESINS

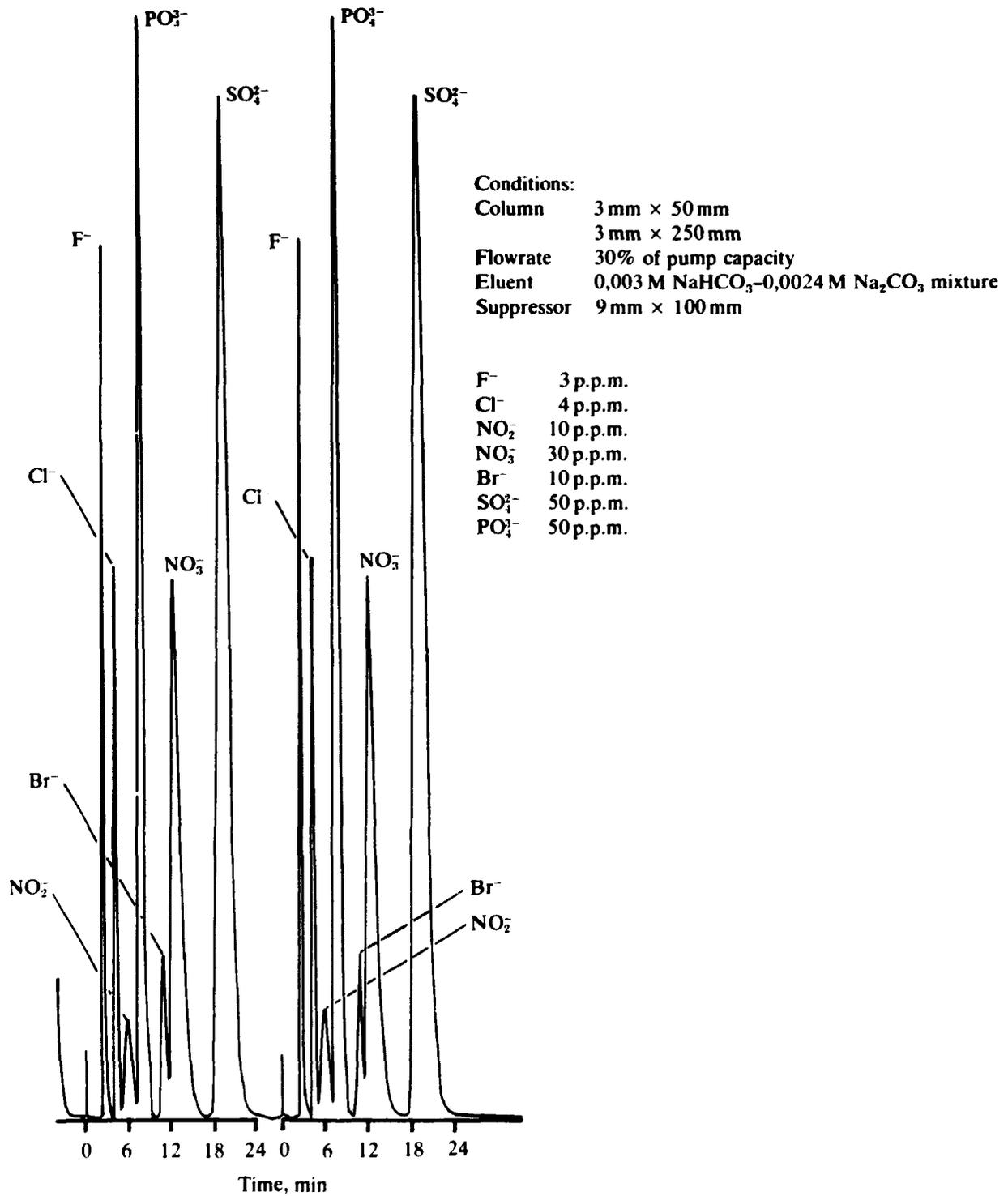


FIGURE 10. The reproducibility test with a Dionex Model 10 Ion Chromatograph