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Original Scientific Paper

Solvent Extraction and Separation of Niobium and Tantalum by Monooctylester of Anilinobenzylphosphonic Acid

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The extraction of niobium and tantalum from aqueous solutions of their oxalato and fluoro complexes has been studied, using the monoctylester of anilinobenzylphosphonic acid (MOABP) as extractant. The dependence of the extraction of niobium and tantalum upon the hydrochloric, sulphuric, nitric, perchloric, phosphoric, hydrofluoric and oxalic acid concentration is described. A procedure for the separation of niobium from tantalum has been established. The back-extraction of niobium and tantalum from the organic phase was carried out with hydrofluoric, phosphoric and oxalic acid. The dependence of the extraction of niobium upon the MOABP concentration in the organic phase, and oxalic or hydrofluoric acid in the aqueous phase was also studied. It was found that the distribution coefficients for niobium from oxalate solution were proportional to the second power of the MOABP concentration. The logarithmic dependence of the distribution coefficients for niobium from fluoride solution *vs.* the MOABP concentration gave a curve whose slope was 1.5. The extraction of niobium from oxalate solution is linearly proportional to the oxalic acid concentration, while the extraction of niobium from fluoride solution depends upon the second power of the hydrofluoric acid concentration. Similar experiments with tantalum were not made because of its very low extraction under the same conditions. The radionuclides ^{95}Nb and ^{182}Ta were used for the determination of the distribution coefficients of niobium and tantalum in all of the experiments described.

INTRODUCTION

There has recently been reported an increasing number of papers dealing with the investigation of the chemistry of zirconium, niobium and tantalum. This group of elements, owing to their exceptional properties, has found a wide application in nuclear as well as in other branches of modern technology. Since their chemical behaviour is very similar, many common methods cannot be successfully applied for their separation. Therefore, many investigators have been looking for new methods for their processing and purification.

Various methods for the separation of zirconium, niobium and tantalum have been described, the most frequently used being extraction procedures utilizing organic compounds. Hardy and Scargil¹ used tributylphosphate for the extraction of niobium from fission products. A mixture of monobutyl- and dibutylphosphates², as well as di-2-ethylhexylphosphoric acid³, was employed

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for the extraction of tantalum and niobium. Djordjević and Goričan⁴ have described the extraction and separation of niobium and tantalum with di-octyl-methylene-bisphosphonic acid. Many different types of amines have also been used for the extraction of niobium and tantalum. Moore *et al.*⁵ applied tribenzylamine and Marchart and Hecht⁶ triisooctylamine for the extraction of niobium and tantalum.

It has been reported that the mono-octyl ester of anilino-benzylphosphonic acid appeared to be very suitable for the extraction of certain metals⁷⁻⁹. This paper describes its application for the extraction of niobium and tantalum from their oxalato and fluoro complexes in solution and the influence of hydrochloric, nitric, sulphuric, phosphoric, perchloric, hydrofluoric and oxalic acid concentrations upon their extraction. The separation of niobium and tantalum, as well as their back-extraction from organic phase is described. From the studies of the dependence of the niobium extraction coefficients upon the MOABP concn. in the organic phase, and upon the oxalic or hydrofluoric acid concn. in the aqueous phase, the probable niobium complexes in the organic phase are discussed.

EXPERIMENTAL

The radionuclide ⁹⁵Nb in the form of oxalato complexes was purchased from the Radiochemical Center Amersham. ¹⁸²Ta was prepared by irradiating its oxalic solution in the Nuclear reactor of the Institute »Boris Kidrič«, Vinča, Yugoslavia.

The standard solutions of niobium and tantalum were obtained from Nb₂O₅ and Ta₂O₅, *Hopkin and Williams* by fusing with KHSO₄ and subsequent extraction with oxalic acid solution. The prepared solutions were then precipitated with ammonia, the precipitate washed with 2% NH₄Cl solution, and dissolved in oxalic or hydrofluoric acid. The concentrations were checked by gravimetric analysis of the metals. The radionuclides were added to the inactive solutions. Following precipitation with ammonia, the precipitate obtained was dissolved in an acid in order to prepare an ionic solution necessary for extraction purposes.

MOABP was synthesized as described earlier¹⁰. Its solution in ligroin *Merck* (b. p. 150–180°) served as the organic phase.

All the other chemicals used were of analytical purity.

Determination of the distribution ratios: The extraction and back-extraction of niobium and tantalum were performed in Erlenmeyer flasks at approximately 20° C, using equal volumes of both the organic and aqueous phase. The shaking time necessary to attain equilibrium was experimentally determined for each system. After equilibrium had been attained, the systems were centrifugated to speed up separation of the phases. Samples were taken from each phase for radioactivity determination. A gamma scintillation counter (NaI/Tl) was used for this purpose. All measurements were taken after a single extraction.

In order to determine the distribution coefficients in the mixture of niobium and tantalum, two parallel systems were prepared, one of which was labelled with radioactive ⁹⁵Nb and the other with radioactive ¹⁸²Ta. The experiments with metal fluorides and hydrofluoric acid were performed in polythene containers.

RESULTS

Systems Nb(V)-Oxalate-Mineral Acids

The extraction of niobium from oxalic acid was found to be very poor. In 0.01 M oxalic acid the distribution coefficient for niobium is practically zero and it remains unaffected by changes in the oxalic acid concentration. The addition of certain mineral acids, however, significantly improves the extraction of niobium. The influence of hydrochloric and oxalic acid concentrations

on the extraction of niobium is shown in Fig. 1. The percent of niobium extracted into the organic phase is plotted vs. the hydrochloric acid concentration in the aqueous phase. The concentration of oxalic acid was constant at either

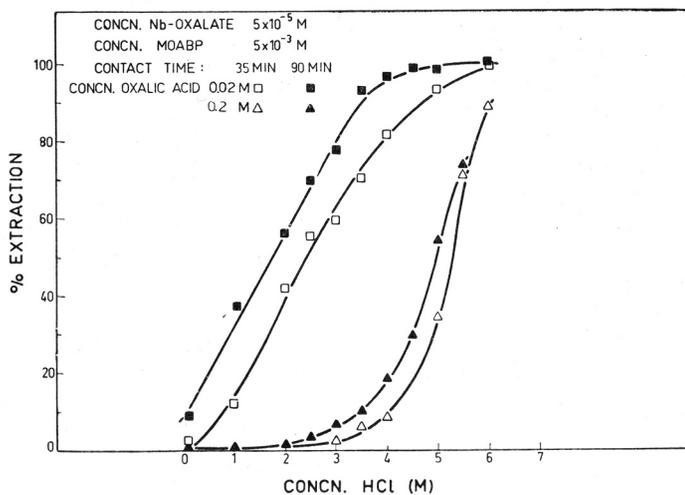


Fig. 1. Extraction of niobium (V) from fluoride solution in the presence of: \square \blacksquare : 0.02 M and \triangle \blacktriangle : 0.2 M oxalic acid as a function of HCl concentration in the aqueous phase. Concentration of niobium 5×10^{-5} M and MOABP 5×10^{-3} M. Shaking time \triangle \square : 35 min., \blacktriangle \blacksquare : 90 min.

0.02 M or 0.2 M. The extraction of niobium increases with an increase in the hydrochloric acid concentration, whereas the concentration of oxalic acid has the opposite effect, causing, a decrease in the extraction of niobium. The extraction of niobium is less dependent upon the oxalic acid concentration in the presence of higher hydrochloric acid concentrations.

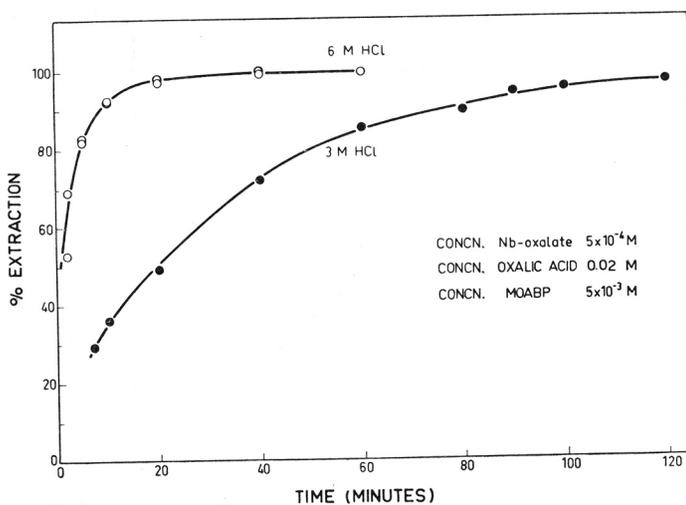


Fig. 2. Extraction of niobium (V) from oxalate solution in the presence of 0.02 M oxalic acid and \bullet : 3 M; \circ : 6 M HCl concentration in the aqueous phase as a function of shaking time. Concentration of niobium 5×10^{-4} M, and MOABP 5×10^{-3} M.

The time necessary to attain equilibrium was experimentally checked for each system. A typical example is given in Fig. 2. Niobium oxalate was extracted in the presence of 3 *M* and 6 *M* HCl. At higher hydrochloric acid concentrations the equilibrium was attained after 20 minutes, *e. g.* 4—5 times faster than with 3 *M* hydrochloric acid. In all of the experiments studied, where different acid concentrations were used, the longest necessary shaking time was always employed in order to equilibrate the system completely. In such a way, reproducible results were obtained.

The influence of sulphuric acid upon the extraction of niobium from oxalate solution closely resembles that of hydrochloric acid. Therefore these results are not separately presented.

Nitric and perchloric acid, to some extent, improve the extraction of niobium oxalate. However, they cannot be used in concentrations higher than 3 *M*, because at higher concentrations some precipitation takes place during the extraction.

Phosphoric acid, if present in low concentrations, only slightly improves the extraction of niobium. Higher concentrations decrease the extraction⁹. Depending upon the phosphoric acid concentration (0.001—6 *M*), the distribution coefficients for niobium amounted to between 0.02 and 0.0005.

Extraction Dependency of Nb(V) on Oxalic Acid Concentration

The extraction dependence of niobium upon oxalic acid concentration in the aqueous phase is shown in Fig. 3. The experiments were carried out at

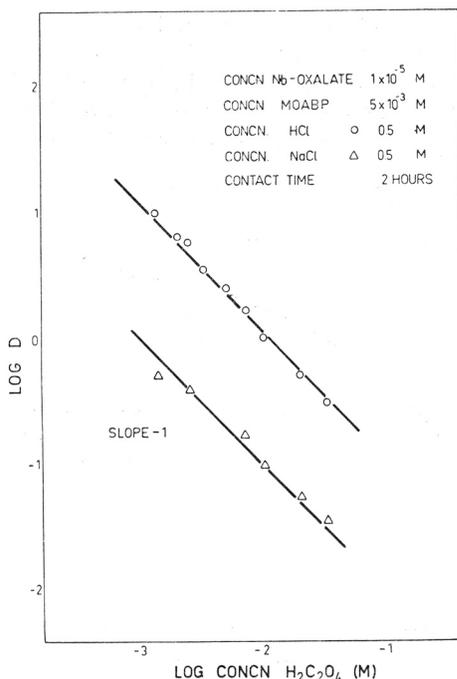


Fig. 3. Distribution ratio *D* of niobium (V) from oxalate solution in the presence of ○ : 0.5 *M* HCl; △ : 0.5 *M* NaCl, as a function of oxalic acid concentration in the aqueous phase. Concentration of niobium 1×10^{-5} *M* and MOABP 5×10^{-3} *M*.

a constant ionic strength in the aqueous phase, in the presence of 0.5 M HCl or 0.5 M NaCl (niobium concentration 10^{-5} M; MOABP concentration 5×10^{-3} M). Under the described experimental conditions, linear curves were obtained. Their slope of -1 indicates that during the extraction of niobium only one oxalate ion is, on the average, released per one niobium.

Extraction Dependency of Nb(V) on MOABP Concentration

The extraction dependence of niobium from oxalate solution upon ligand concentration in the organic phase is shown in Fig. 4. An excess of MOABP

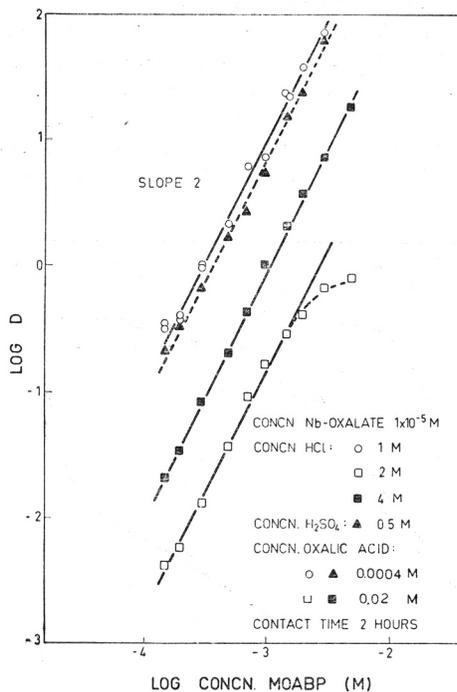


Fig. 4. Distribution ratio D of niobium from oxalate solution in the presence of: \circ \blacktriangle : 0.0004 M, \square \blacksquare : 0.02 M oxalic acid and \circ : 1 M, \square : 2 M, \blacksquare : 4 M, HCl and \blacktriangle : 0.5 M H_2SO_4 , as a function of MOABP concentration in the organic phase.

was used; therefore its total concentration was plotted. The experiments were carried out at constant acid concentrations of 1 M HCl; 2 M HCl; 4 M HCl; 0.5 M H_2SO_4 ; 0.0004 M oxalic acid and 0.02 M oxalic acid. In all systems studied, the linear dependence of the distribution coefficients upon the reagent concentration was determined. The slope of the logarithmic curve was 2, indicating that, regardless of the niobium complex present in the aqueous phase, there is always a complex in the organic phase which contains 2 molecules of MOABP per niobium atom.

Systems Ta(V)-Oxalate-Mineral Acids

The extraction of tantalum has been studied from the following acid solutions: 0.01–6 M HCl; 0.01–6 M HNO_3 ; 0.01–6 M H_2SO_4 ; 0.001–2 M H_3PO_4 ; 0.1–2 M HF and 0.02–0.4 M oxalic acid. For all of these systems the extraction

was very poor, the distribution coefficients of tantalum varying between 0.0005 and 0.03, depending upon the acid concentration. During the extraction, a precipitate was always formed which contained practically all of the tantalum present. In order to avoid this precipitation, different initial oxalic acid concentrations, as well as more polar organic solvents (other than ligroin) were investigated, but without success.

Systems Nb(V)-Ta(V)-Fluoride-Mineral Acids

The extraction of niobium and tantalum from hydrofluoric acid solution is quite unsatisfactory. The presence of mineral acids significantly increases the extraction of both metals, especially if the hydrofluoric acid concentration is low. The extraction dependence of niobium and tantalum upon hydrochloric, hydrofluoric and MOABP concentration is shown in Fig. 5. (Metal concentration 10^{-4} M; MOABP concentration 10^{-3} M and 5×10^{-3} M). Niobium was extracted using the following constant hydrofluoric acid concentrations: 0.004 M, 0.01 M and 0.1 M HF. The tantalum experimental data in Fig. 5 are for 0.004

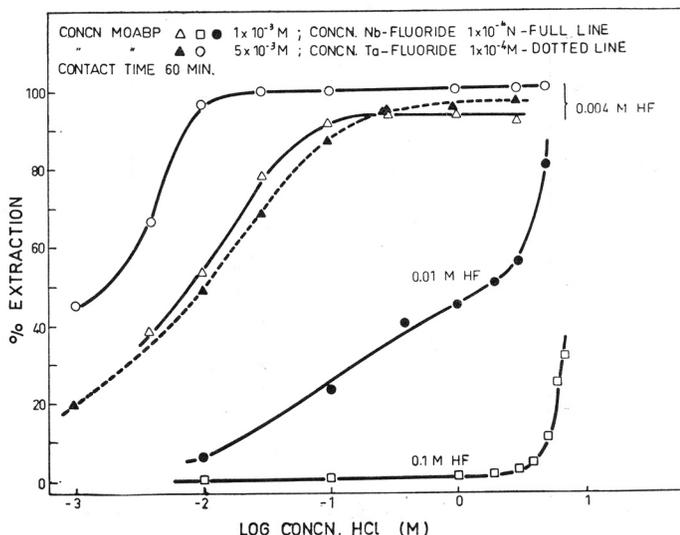


Fig. 5. Extraction of niobium (V) and tantalum (V) from fluoride solution in the presence of : \circ \triangle \blacktriangle : 0.004 M, \bullet : 0.01 M, and \square : 0.1 M HF as a function of HCl concentration. Concentration of niobium and tantalum 1×10^{-4} M and MOABP : \triangle \square \bullet : 1×10^{-3} M, \blacktriangle \circ : 5×10^{-3} M.

M HF. The quantitative extraction of niobium was achieved by using an aqueous solution containing 0.004 M HF and 0.01 M HCl, with an excess (5×10^{-3} M) of MOABP. Increasing the hydrofluoric acid concentration to 0.01 M significantly diminishes the extraction of niobium. To improve the extraction it was necessary to increase the hydrochloric acid concentration. With 0.1 M HF the extraction was very low even in the presence of concentrated hydrochloric acid. Tantalum behaves similarly. Therefore, only one experimental result is presented in Fig. 5. for comparison. In general, the extraction of tantalum from hydrochloric acid solution is slightly lower than that of niobium under the same conditions.

Extraction Dependency of Nb(V)-Fluoride on HF Concentration

The dependence of niobium extraction upon hydrofluoric acid concentration in aqueous phase is shown in Fig. 6. Two systems are plotted in the diagram.

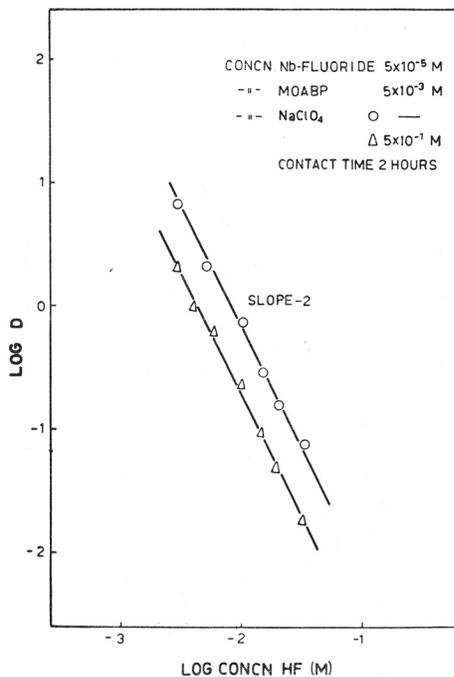


Fig. 6. Distribution ratio D of niobium (V) from fluoride solution in the presence of : ○ : — and Δ : 0.5 M NaClO₄ as a function of HF concentration in the aqueous phase. Concentration of niobium 5×10^{-5} M and MOABP 5×10^{-3} M.

One curve was obtained in the presence of 0.5 M NaClO₄ and the other without NaClO₄. In both cases a linear dependence was found, and the slope of the logarithmic curve was -2 . The experiments were carried out with a large excess of MOABP, therefore its concentration was assumed to be constant during the extraction.

Extraction Dependency of Nb(V)-Fluoride on MOABP Concentration

In Fig. 7 the extraction dependence of niobium upon MOABP concentration in the organic phase is given. A large excess of MOABP was used and its total concentration is plotted on the abscissa of the diagram. Two experiments were conducted, one with the addition of hydrochloric acid (0.01 M) and the second without hydrochloric acid. The extraction of both systems was carried out in the presence of a constant hydrofluoric acid concentration (0.013 M). Regardless of the different experimental conditions, parallel curves were obtained and in both cases a metal-ligand ratio of $1:1.5$ was observed.

Systems Nb(V)-Ta(V)-Fluoride — H₃PO₄

The extraction of niobium and tantalum from orthophosphoric acid solutions is shown in Fig. 8. The initial hydrofluoric acid concentration was

$5 \times 10^{-4} M$ and $9 \times 10^{-4} M$; the metal concentration $1 \times 10^{-4} M$ and MOABP $5 \times 10^{-3} M$. It was found that niobium can be quantitatively extracted from

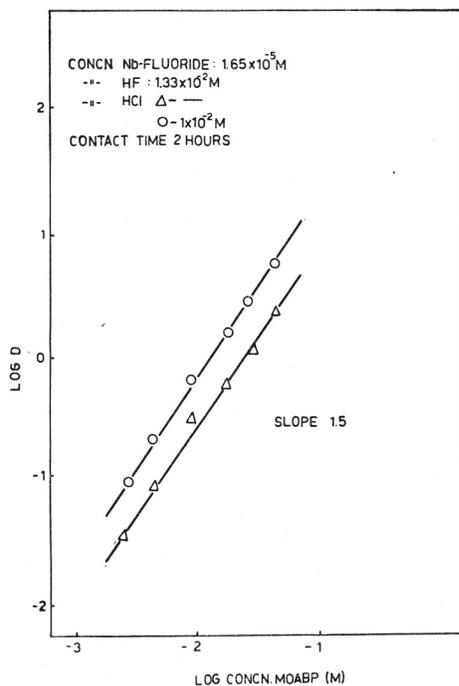


Fig. 7. Distribution ratio D of niobium (V) from fluoride solution in the presence of: $1.33 \times 10^{-2} M$ HF and \triangle —, \circ : $1 \times 10^{-2} M$ HCl, as a function of MOABP concentration in the organic phase. Concentration of niobium $1.65 \times 10^{-5} M$.

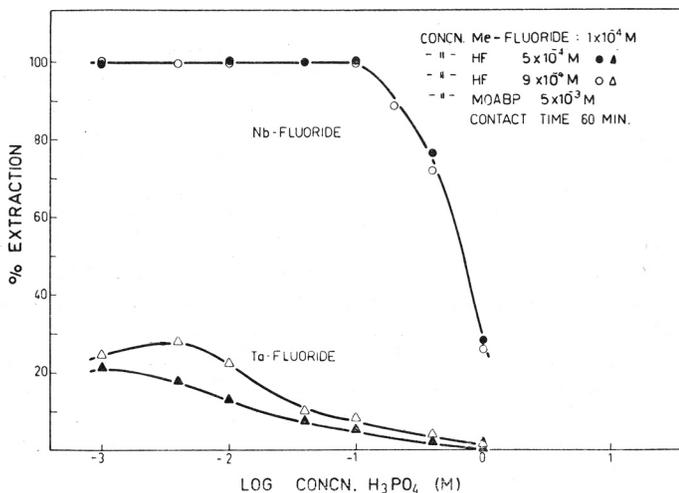


Fig. 8. Extraction of niobium (V) and tantalum (V) from fluoride solution in the presence of: \bullet \blacktriangle : $5 \times 10^{-4} M$ and \circ \triangle : $9 \times 10^{-4} M$ HF as a function of H_3PO_4 concentration in the aqueous phase. Concentration of niobium and tantalum $1 \times 10^{-4} M$.

0.001—0.1 M H_3PO_4 . A further increase in the phosphoric acid concentration sharply decreases its extraction. A change in the hydrofluoric acid concentration from 5×10^{-4} M to 9×10^{-4} M HF does not effect the niobium extraction.

Under the same conditions, tantalum is extracted to a lesser degree, its extraction being more dependent upon the hydrofluoric acid concentration.

In order to obtain more information about the extraction of these two metals from a mixed hydrofluoric and phosphoric acid solution, the following experiment was made. Niobium and tantalum were extracted for constant phosphoric acid concentrations (0.1 M and 0.01 M H_3PO_4) while the concentration of hydrofluoric acid was varied, as shown in Fig. 9. From a solution

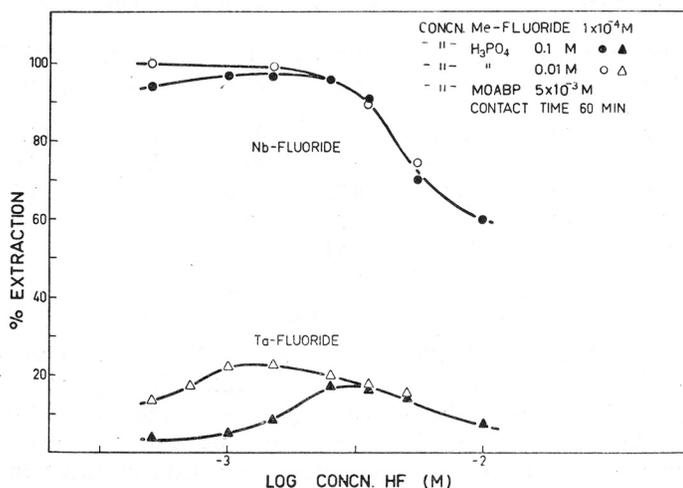


Fig. 9. Extraction of niobium (V) and tantalum (V) from fluoride solution in the presence of: ● ▲ : 0.1 M and ○ △ : 0.01 M H_3PO_4 as a function of HF concentration in the aqueous phase. Concentration of niobium and tantalum 1×10^{-4} M.

of 0.001 M HF and 0.01 M H_3PO_4 , niobium is quantitatively extracted. With the HF concentration between 5×10^{-4} M and 3×10^{-3} M HF, for both 0.1 M and 0.01 M H_3PO_4 , more than 90% of niobium can be extracted. A further increase in the hydrofluoric acid concentration decreases its extraction. From Fig. 8 and 9 one can see that the tantalum extraction is lower, and depends more upon the change in concentration of HF or H_3PO_4 . The maximum extraction of tantalum occurs at 0.01 M H_3PO_4 and 0.001 M HF. If the concentration of phosphoric acid increases to 0.1 M, the maximum is shifted to 0.003 M HF. Increasing the hydrofluoric acid concentration above 0.003 M steadily decreases the tantalum extraction. From the results obtained, it is obvious that the extraction behaviour of niobium and tantalum from mixtures of phosphoric and hydrofluoric acid of varying concentrations differs sufficiently to permit satisfactory separation. The best separation can be achieved from a mixture of 0.1 M H_3PO_4 and 0.001 M HF.

Back-extraction of Nb- and Ta—MOABP Complexes from the Organic Phase

Back-extraction of niobium and tantalum from the organic phase has been studied in oxalic, hydrofluoric and phosphoric acid solutions. Niobium and tantalum were previously transferred to the organic phase by the extraction

of their oxalato or fluoro complexes in the presence of HCl or H_3PO_4 , by MOABP, under the most favourable conditions.

The back-extraction was carried out by shaking the prepared organic phase with an equivalent volume of aqueous oxalic, hydrofluoric or phosphoric acid solution. The results obtained are shown in Table I. It can be seen that niobium and tantalum are easily recovered from the organic phase using any of these acids.

TABLE I

Back-extraction of Nb- and Ta-MOABP complexes from the organic phase, with oxalic, HF and H_3PO_4 acids. The distribution coefficient refers to the equilibrium concentration of metal in the organic and aqueous phase. Shaking time 60 minutes.

$H_2C_2O_4$ (M)	Distribution ratio		(M) HF	Distribution ratio		H_3PO_4 (M)	Distribution ratio	
	D_{Nb}	D_{Ta}		D_{Nb}	D_{Ta}		D_{Nb}	D_{Ta}
0.008	44.6	59.6	0.01	95.0	42.1	0.5	61.7	67.3
0.04	1.9	17.3	0.04	6.8	14.2	1.0	4.0	5.5
0.08	1.1	9.8	0.1	3.75	6.2	1.5	2.1	3.1
0.2	0.8	2.7	0.2	2.25	2.1	2.0	1.8	1.9
0.4	0.4	1.6	0.4	0.64	0.5			

DISCUSSION

The purpose of the experiments described in this paper was to ascertain whether the mono-octyl ester of anilinobenzylphosphonic acid, $C_6H_5NHCH-(C_6H_5)PO(OC_8H_{17})OH$, would be useful for the extraction of niobium and tantalum. As far as we know, this is the first published procedure utilizing an aminophosphonic acid monoester for the extraction of these metals. Since the oxalato and fluoro complexes of both metals are soluble in water and, in addition, are easily accessible, the investigation of the extraction of niobium and tantalum from oxalic and hydrofluoric acid solutions seemed to us to be of particular importance. Our experiments show that the extraction of niobium and tantalum with MOABP from both oxalic and hydrofluoric acid solutions is very poor. This is not surprising, since these metals form stable complexes with oxalate and fluoride anions. The addition of mineral acids to an aqueous solution of these metal complexes, increases, to a certain extent, their extraction. An exception is tantalum oxalate, in which case there was always a precipitate formed during the extraction. It was found that this precipitate contained nearly all of the original tantalum, and this explains the reason why the tantalum extraction remained very low, even upon the addition of mineral acids.

It is known that niobium and tantalum form different oxalato and fluoro complexes, depending upon the pH of the solution and the ligand concentration. These complexes contain water molecules or hydroxyl groups as ligands around the metal atoms. The niobium and tantalum complexes are easily hydrolyzed at relatively low pH values, which is probably due to the substitution of water molecules by hydroxyl groups in the metal co-ordination sphere. An increase in the distribution coefficient of niobium and tantalum upon the addition of mineral acids is mainly the result of a change in their oxalato or fluoro complexes in aqueous solution. The influence of the hydrochloric acid

concentration on the extraction of niobium is shown in Fig. 1. One explanation could be that with the increased hydrogen ion concentration, the hydroxyl ions, placed around the central metal ions in their oxalato or fluoro complexes, are replaced by water molecules or other ions present, which, in addition, can be more easily substituted by MOABP than by hydroxyl groups.

The influence of the sulphuric acid concentration on the extraction of niobium is similar to that of hydrochloric acid and is therefore not presented. In Fig. 1 it can be seen that the niobium extraction decreases when the oxalic acid concentration is increased. This is understandable, since oxalic acid competes with the reagent. The plot of the dependence of the niobium distribution coefficient upon the oxalic acid concentration gave a curve with a slope of -1 (Fig. 3). This result indicates that on the average one molecule of oxalic acid is released during the extraction. After plotting the dependence of the niobium distribution coefficient upon the MOABP concentration in the organic phase (Fig. 4), the curves with slope 2 are obtained, indicating that, following the release of one oxalic acid molecule, 2 molecules of the reagent are bound to one atom of niobium. Since it was found that in the niobium oxalato complexes existing in aqueous solution, only one oxalate ion is bound to niobium¹¹, the complex which is extracted into the organic phase does not contain oxalate. In spite of the different experimental conditions, (summarized in Figs. 3 and 4), the same results were always obtained. This led us to propose the following reaction mechanism:



This is only a simplified presentation of the actual extraction mechanism which, as is to be expected, is more complex. In this complex, niobium probably has a co-ordination number of 6, since the reagent may wet as a bidentate ligand.

A decrease in the niobium extraction, caused by an increase in the oxalic acid concentration can be compensated by the addition of hydrochloric acid, as shown in Fig. 1. From 0.02 *M* oxalic acid and 3 *M* hydrochloric acid, niobium is nearly quantitatively extracted. If the oxalic acid concentration is increased to 0.2 *M*, it is necessary to add 6 *M* hydrochloric acid in order to attain the same percentage of extracted niobium.

During the extraction of niobium and tantalum by means of MOABP, equilibrium is achieved slowly. The rate of substitution of oxalic or hydrofluoric acid by reagent ligands is dependent upon the mineral acid concentration. A typical example is shown in Fig. 2., where niobium was extracted in the presence of 3 *M* and 6 *M* hydrochloric acid. With higher acid concentrations the extraction equilibrium was achieved 4—5 times faster.

Nitric and perchloric acid, to a degree, also increase the extraction of niobium. However, if the concentration of these acids is higher than 3 *M*, some precipitate is formed during the extraction. This precipitate consists mainly of niobium and MOABP and is probably present as the polymeric or oxo complexes.

A similar investigation on tantalum was not undertaken, because of its precipitation and poor extraction.

Extraction of niobium and tantalum in the presence of hydrochloric acid is shown in Fig. 5. Here again, an increase in the hydrochloric acid concentration improves the extraction. Hydrofluoric acid has the opposite effect, causing

a very sharp decrease in the extraction as the acid concentration is increased. This decrease of extraction can be, to some extent, compensated with higher hydrochloric acid concentration. The dependence of the niobium distribution coefficient in fluoride media upon the hydrofluoric acid concentration in aqueous phase (Fig. 6) and the MOABP concentration in the organic phase (Fig. 7), indicates that, during the extraction, 2 molecules of hydrofluoric acid on the average are released and subsequently substituted with 1.5 molecules of MOABP. These results show that the extraction of niobium from fluoride solution is a complex phenomenon and for its complete understanding it will be necessary to have more experimental data. Similar experiments with tantalum were not carried out because of its low extraction from hydrofluoric acid solution in the absence of hydrochloric acid.

The results discussed above show that oxalate complexes cannot be used for the separation of niobium and tantalum because of the precipitation of the latter. From fluoride media the tantalum extraction is less than for niobium but the difference is not sufficient to warrant their separation. It was found, however, that the influence of orthophosphoric acid on the extraction of niobium and tantalum from fluoride solution is significantly different. The results presented in Figs. 8 and 9 show that niobium is extracted much better than tantalum. The extraction of niobium is quantitative from solutions containing 0.001–0.1 *M* phosphoric acid. An increase in the phosphoric acid concentration from 0.1 *M* to 1 *M*, decreases the percentage of extracted niobium to about 30%. The maximum tantalum extraction occurs in the presence of 0.001 *M*–0.01 *M* phosphoric acid. A further increase in the phosphoric acid concentration decreases the extraction. Such an influence of phosphoric acid can be explained if one assumes that at a lower acid concentration, the metal fluoro complexes are converted into more reactive forms which can react with MOABP. At higher concentrations, phosphoric acid itself forms stable phosphate complexes in the aqueous phase. The results plotted in Figs. 8 and 9 indicate that the extractions of niobium and tantalum in the presence of phosphoric acid differ sufficiently for it to be possible to separate these two metals. Similar results were obtained by repeated experiments with a mixture of these metals. The best separation can be achieved from a solution containing a mixture of 0.001 *M* hydrofluoric and 0.1 *M* phosphoric acid, whereby niobium extracts into the organic phase while tantalum remains in the aqueous phase.

The advantage of this procedure is that both metal fluoride solutions can be easily prepared. These solutions, stored in polythene containers, are stable even if the concentration of hydrofluoric acid is only 0.001 *M*. Reproducible results were obtained even if solutions several months old were used.

Table I shows that niobium and tantalum, extracted into the organic phase, can be easily re-extracted into the aqueous solution. Equally satisfactory results for their back-extraction were obtained by means of the oxalic, hydrofluoric or phosphoric acid solutions.

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IZVOD

Ekstrakcija i separacija niobija (V) i tantala (V) pomoću mono-oktilnog estera anilinobenzilfosfonske kiseline

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Studirana je ekstrakcija niobija i tantala iz otopina oksalata i fluorida pomoću mono-oktilnog estera α -anilino-benzilfosfonske kiseline. Ispitana je ovisnost ekstrakcije niobija i tantala o koncentraciji HCl, HNO₃, H₃PO₄, HF i H₂C₂O₄. Opisana je mogućnost odjeljivanja niobija od tantala kao i reekstrakcija ovih metala iz organske faze pomoću HF, H₃PO₄ ili H₂C₂O₄.

Određena je ovisnost koeficijenta ekstrakcije o koncentraciji ekstraktanta u organskoj fazi i o koncentraciji oksalne odnosno fluorovodične kiseline u vodenoj fazi. Dobiveni su odnosi metal-ligand 1 : 2 za niobij iz otopine oksalata, a 1 : 1,5 iz otopine fluorida. Ekstrakcija niobija prikazana u ovisnosti o koncentraciji oksalne odnosno fluorovodične kiseline daje pravce s nagibom —1 za oksalnu kiselinu a —2 za fluorovodičnu kiselinu.

Određivanje koeficijenta ekstrakcije vršeno je radiometrijskom metodom primjenom γ -scintilacionoga brojača (NaJ/Tl) a upotrijebljeni su radionuklidi ⁹⁵Nb i ¹⁸²Ta.

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