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Chemistry

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Abstract

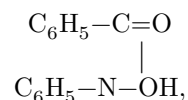
Full Text

Chemistry

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SEPARATION OF NIOBIUM AND TANTALUM BY EXTRACTION OF NIOBIUM N-BENZOYLPHENYLHYDROXYLAMINATE

The separation of niobium and tantalum is rightly regarded as one of the most difficult problems in analytical chemistry ⁽¹⁾. The most promising method for separating these two elements is the extraction of inner-complex compounds with organic solvents. For this purpose an analogue of cupferron, N-benzoylphenylhydroxylamine (BPHA), proved very suitable,



which forms with niobium a complex compound soluble in chloroform. This reagent has recently been used for the quantitative determination of Zr, Th, and Sc ⁽²⁾, and also for the gravimetric determination of niobium and tantalum ⁽³⁻⁵⁾. In one review on extraction ⁽⁶⁾ there is a brief indication of the possibility of extracting tantalum with a solution of BPHA in chloroform in the presence of fluoride ion. It should be noted that the gravimetric method has a number of well-known disadvantages caused by the large phase-interface area and, consequently, by considerable coprecipitation, which makes at least double precipitation necessary. Extraction from F⁻-containing solutions requires the use of polyethylene vessels. In one of our studies ⁽⁷⁾ it was shown that extraction of the inner-complex compound of niobium with BPHA is possible in the presence of tartaric acid, which keeps niobium and tantalum in solution and at the same time does not hinder extraction.

Fig. 1. Extraction with chloroform of Nb and Ta compounds with BPHA as a function of the pH of the solution.

To monitor the extraction, radioactive isotopes of niobium and tantalum were used (Nb^{95} , $T_{1/2}$ 35 days; Ta^{182} , $T_{1/2}$ 115.1 days). To exclude incomplete isotopic exchange, the radioactive isotopes were added to the stable isotopes before dissolution of niobium and tantalum pentoxides. The solutions were prepared by dissolving weighed portions of Nb_2O_5 and Ta_2O_5 in a mixture of conc. H_2SO_4 and $(\text{NH}_4)_2\text{SO}_4$, followed by dissolution of the melt in tartaric acid. The resulting solutions contained 0.35 mg/ml Nb_2O_5 and 0.30 mg/ml Ta_2O_5 , and were 3% in tartaric acid. Four ml of solution were placed in a test tube with a ground-glass stopper, then 1 ml of a 10% alcoholic solution of BPHA was added. After precipitate formation, 5 ml of chloroform were introduced and the mixture was shaken for 3 min. In all experiments the pH of the aqueous phase after extraction-

...did not change. Then 2 ml of solution was taken from the organic phase, and the activity of the solution was measured on a counting apparatus equipped with a γ -counter. From the measurement results, extraction curves for niobium and tantalum were constructed (Fig. 1).

In the pH range 4-6, niobium is extracted by a single extraction to the extent of 98-100%, whereas tantalum is not extracted at all. During extraction of tantalum at pH 0.5-3.0, the tantalum compound partially floats at the phase boundary. This is apparently explained by the fact that tantalum forms polynuclear compounds that precipitate but do not dissolve in the organic layer. In the pH range 6-9, the data on tantalum extraction are poorly reproducible.

The new separation method was tested on artificial mixtures of niobium and tantalum. Standard solutions of niobium and tantalum, prepared as indicated above, were mixed before separation (pH 4.5-5.0). In each case one of the elements contained a radioactive isotope. The results of the experimental test (Table 1) confirm the possibility of successful and rapid separation of niobium and tantalum up to Nb : Ta ratios of 100 : 1 and 1 : 100.

Table 1

Separation of niobium from tantalum by extraction of niobium N-benzoylphenylhydroxylamine

Taken, mg	Taken, mg	$\frac{\text{Nb}_2\text{O}_5}{\text{Ta}_2\text{O}_5}$	Isotope	Imp/min after extraction	Imp/min after extraction	Extracted %	Extracted, %
Nb_2O_5	Ta_2O_5	$\frac{\text{Nb}_2\text{O}_5}{\text{Ta}_2\text{O}_5}$	Isotope	organic phase	aqueous phase	Nb_2O_5	Ta_2O_5
10.0	1.0	10:1	Nb^{95}	1164	—	160.0	—
10.0	0.1	100:1	Nb^{95}	1024	—	98.1	—

Taken, mg	Taken, mg	$\frac{\text{Nb}_2\text{O}_5}{\text{Ta}_2\text{O}_5}$	Isotope	Imp/min after extrac- tion	Imp/min after extrac- tion	Extracted %	Extracted, %
10.0	0.1	100:1	Ta ¹⁸²	0	1152	—	0
1.0	10.0	1:10	Nb ⁹⁵	1536	—	98.8	—
1.0	10.0	1:10	Ta ¹⁸²	—	2624	—	1.8
0.1	10.0	1:100	Nb ⁹⁵	1797	—	97.9	—
0.1	10.0	1:100	Ta ¹⁸²	32	—	—	2.5

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