MICRODETERMINATION OF Nb(V) AND Ta(V) USING THYMINE

Shashi Lata and G.C. Shivahare

Department of Chemistry, University of Rajasthan, Jaipur - 302004.

Niobium and Tantalum have been determined by gravimetric spectrophotometric, colorimetric and potentiometric methods using various reagents (1-7). In the present communication thymine (2,4-dihydroxy 5-methyl pyrimidine) has been used as a reagent for potentiometric determination of Ta (V) in mixture. Niobium and tantalum are very similar in their chemical properties and their estimation in mixture by potentiometric method is of interest since other methods take long time.

EXPERIMETNAL

E.M.F measurements were carried out with Toshniwal CL 43 pH meter having combined glass calomel electrode. Microburette with 0.01 subdivisions was used for volume measurements.

Preparation of Solution

Solutions of Ta(V) and Nb(V) were prepared by fusing their oxides (Fluka) separately with potassium pyrosulphate and extracting the melt with 10 percent aqueous solution of oxalic acid [8]. The solutions were standardised by precipitating Nb(V) and Ta (V) with tannin and weighing them as their pentaoxides. These solutions were used as stock solutions and diluted to required concentrations.

0.01 M solution of thymine was prepared by dissolving calculated amount in double distilled water. This solution was further diluted as required.

Titrations were carried out using 0.01 M, 0.02 M and 0.03 M solutions of Nb(V) and Ta(V) in direct titrations and 0.001 M, 0.002 M and 0.003 M respectively in reverse titrations. In the direct titrations, the metal solution was added from the burette to the cell containing the ligand solution In the reverse titration, the ligand solution was added from the burette to the metal solution in the cell. The concentration of the solution in the burette was kept 10 times more than that in the cell. The end point was obtained by plotting the curve representing $\Delta E/\Delta V$ against the volume added. The composition of the complex formed in solution corresponds to 1:2 and 1:1 metal to ligand ratio for the Nb(V) and Ta(V) complex respectively.

While titrating the mixture of Nb(V) and Ta(V), the titration curve shows two distinct inflexions and two peaks are obtained when $\Delta E/\Delta V$ is plotted

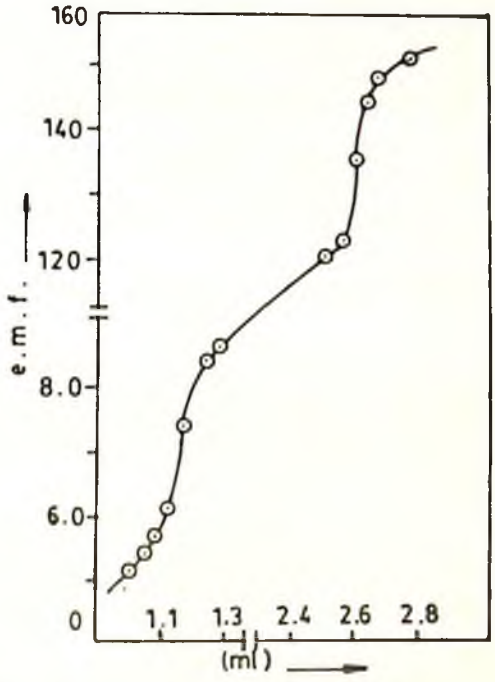


Fig. 1a Direct titration mixture of Nb(V) & Ta(V)

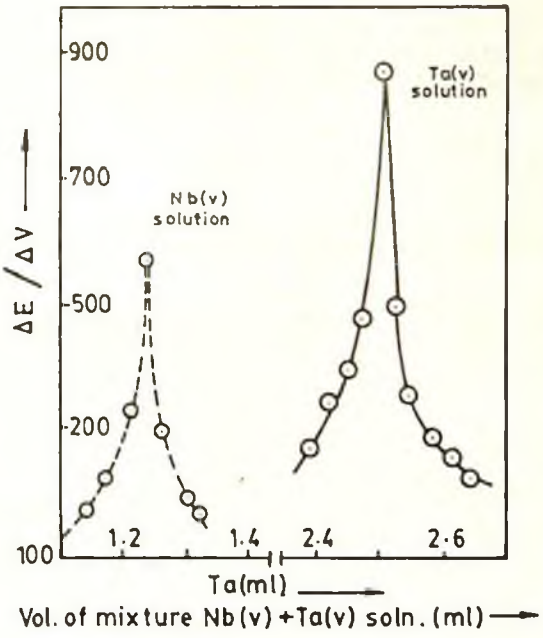


Fig. 1b Direct titration of mixture of Nb(V) & Ta(V) with thymine

0013 466 x (35) SN 13

TABLE 1

ESTIMATION OF NIOBIUM AND TANTALUM

Direct Titration Nb(V) solution vs Thymine solution in cell			Direct Titration Ta(V) solution vs Thymine solution in cell		
Nb(V) present (µg)	Nb(V) found (µg)	Error %	Ta(V) present (µg)	Ta(V) found (µg)	Error (%)
23.25	23.16	0.39	45.25	45.43	0.39
11.63	11.67	0.34	22.63	22.67	0.18
7.75	7.73	0.26	15.08	15.12	0.27

TABLE 2

ESTIMATION OF Nb(V) AND Ta(V) IN MIXTURE

Nb(V) present (ug)	Nb(V) found (ug)	Error (%)	Ta(V) present (ug)	Ta(V) found (ug)	Error (%)
23.25	23.15	0.43	45.25	45.42	0.37
11.63	11.65	0.17	22.63	22.69	0.26
7.75	7.72	0.39	15.08	15.12	0.26

against volume which shows that presence of the other metal does not interfere when each one is estimated potentiometrically using thymine.

Typical graph for the titration of the mixture is given in Fig.1a&b. Results of some estimations are summarised in Table 1-2

Interference of Foreign Ions

Mg^{2+}, Cd^{2+}, Ca^{2+}, Fe^{3+}, Ta^{5+}, Mo^{6+}, Mn^{6+}, W^{6+}, Cl^-, NO_3^-, citrate, oxalate and tartrate do not interfere in the estimation of Nb(V)by this method but Pb^{2+}, Sr^{2+}, Zr^{4+} V^{5+}, U^{6+}, Br^-, I^- and BO_3^{3-} interfere, Mg^{2+}, Ca^{2+}, Cd^{2+}, Fe^{3}, Sr^{2}, V^{5+}, Mo^{6+}, Cl^-, Br^-, I^-, NO_3^- citrate, tartrate, and oxalate do not interfere in the estimation of Ta(V) by this method but Pb^{2+}, Zr^{4+}, W^{6+}, BO_3^{3} and PO_4^{3-} interefere.

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