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### Solvent Extraction Separation of Niobium(V), Tantalum(V) or Titanium(IV), and Vanadium(V) by 2'-Hydroxy-4-methoxy-5'-methyl Chalkone Oxime (HMMCO) from Their Binary Mixtures

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## NOTE

### **Solvent Extraction Separation of Niobium(V), Tantalum(V) or Titanium(IV), and Vanadium(V) by 2'-Hydroxy-4-methoxy-5'-methyl Chalkone Oxime (HMMCO) from Their Binary Mixtures**

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#### **Abstract**

2'-Hydroxy-4-methoxy-5'-methyl chalkone oxime (HMMCO) has been used for the solvent extraction separation of Nb(V), Ta(V) or Ti(IV), and V(V) from their binary mixtures. V(V) can be quantitatively extracted with HMMCO from 9 M hydrochloric acid. The green-colored chloroform extractable V(V)-HMMCO complex absorbs at a maximum at 630 nm. Nb(V), Ta(V), and Ti(IV) are not extracted at these conditions. However, these metals can be extracted from strong hydrochloric acid medium in the presence of potassium thiocyanate. The yellow-colored complexes of Nb(V), Ta(V), and Ti(IV) absorb at a maximum at 380, 390 and 390 nm, respectively. The method is simple, rapid, and selective, and affords a clean-cut separation of vanadium from niobium, tantalum, or titanium which are usually present in alloys and ores.

Vanadium is usually associated with niobium, tantalum, or titanium in ores and alloys. Several organic reagents have been used for extraction and colorimetric determination of these metal ions. Various oximes such as formaldoxime (1),  $\alpha$ -benzoin oxime (2), salicyladoxime (3), and other oximes (4) have been used for quantitative extraction of vanadium.  $\alpha$ -Benzoin oxime (5) and cupferron (6) have been tried for niobium and titanium. Various hydroxamic acids have been successfully utilized for the extraction and spectrophotometric determination of these metal ions. *N-m-Tolyl-p*-methoxybenzohydroxamic acid (TMBHA) is a newly synthesized and sensitive reagent used for the extraction of vanadium (7),

niobium (8), tantalum (9), and titanium (10). None of the above-mentioned reagents was found to be suitable for the separation of V(V) from Nb(V), Ta(V), or Ti(IV) when present together in a mixture.

2'-Hydroxy-4-methoxy-5'-methyl chalcone oxime (HMMCO) has been successfully employed for the solvent extraction of several transition elements (11). Extension of these studies revealed that it is possible to quantitatively extract V(V), Nb(V), Ta(V), and Ti(IV) with this reagent. The method is simple, rapid, and selective. It is possible to accomplish clean-cut separation of V(V) from Nb(V), Ta(V), or Ti(IV) in their binary mixtures.

## EXPERIMENTAL

### Apparatus and Reagents

Beckman model, DU 2 spectrophotometer with 10 mm quartz cells. Ganson shaker with to and fro action.

HMMCO was synthesized by the method reported earlier (12). Fresh solutions of the reagent in chloroform were used. A 10 *M* potassium thiocyanate solution was prepared in double distilled water. Vanadium solution was prepared by dissolving its oxide (A.R., Reanal, Hungary) in distilled water containing a sufficient quantity of hydrochloric acid. The solution was standardized by a known method (13). The solutions of niobium, tantalum, and titanium were prepared and standardized by the methods reported earlier (8-10).

## RESULTS AND DISCUSSION

V(V) forms a chloroform extractable green-colored complex with HMMCO at 9 *M* hydrochloric acid concentration. Nb(V), Ta(V), and Ti(IV) are not extracted at these conditions. However, these metals can be extracted from strong hydrochloric acid medium in the presence of potassium thiocyanate. The yellow-colored complexes are extractable in chloroform. The optimum conditions of extraction of these metals and other spectral properties of the complexes extracted are reported in Tables 1 and 2, respectively.

### Procedure for Extraction of Vanadium

Take an aliquot of vanadium solution and add enough concentrated hydrochloric acid to make the acid strength 9.0 *M*. Shake this solution with

TABLE 1  
Optimum Conditions of Extraction of Complexes

Complex system	HCl ( <i>M</i> )	KSCN ( <i>M</i> )	HMMCO ( <i>M</i> × 10 <sup>-2</sup> )	Metal ( <i>M</i> × 10 <sup>-4</sup> )
V(V)-HMMCO	9.0	—	3.50	8.78
Nb(V)-SCN-HMMCO	8.0	0.70	0.57	0.79
Ta(V)-SCN-HMMCO	5.7	2.00	1.80	1.80
Ti(IV)-SCN-HMMCO	7.0	2.00	1.02	5.10

TABLE 2  
Spectral Properties of Complexes

Complex system	$\lambda_{\max}$	Beer's law range ( $\mu\text{g/mL}$ )	Molar extinction coefficient, $\epsilon \times 10^{-3}$ ( $\text{L mole}^{-1}\text{cm}^{-1}$ )	Sandell's sensitivity ( $\mu\text{g/cm}^2$ )
V(V)-HMMCO	630	2.70– 94.0	0.87	0.058
Nb(V)-SCN-HMMCO	380	0.76– 25.0	6.10	0.015
Ta(V)-SCN-HMMCO	390	3.70–108.5	2.50	0.072
Ti(IV)-SCN-HMMCO	390	2.93– 88.0	0.86	0.056

10 mL of reagent solution in chloroform for 10 min. Separate the two layers and measure the absorbance of the colored extract at 630 nm against a similarly processed reagent blank. Compute the amount of metal extracted from an appropriate calibration curve.

### Procedure for Extraction of Niobium(V), Tantalum(V), and Titanium(IV)

To an aliquot of metal ion solution add 0.5 to 2 mL of 10 *M* potassium thiocyanate and enough concentrated hydrochloric acid to make an acid strength to about 5 to 8 *M* in a total volume of 10 mL. The solution is shaken well with 10 mL of HMMCO solution in chloroform for 10 min. The absorbance of the separated organic layer is measured at the respective wavelength of maximum absorption. The amount of the metal extracted is computed from the calibration curve.

### Separation and Determination of Niobium(V), Tantalum(V), or Titanium(IV) from Vanadium(V)

The basis for separation of Nb(V), Ta(V), or Ti(IV) from V(V) with HMMCO is that Nb(V), Ta(V), or Ti(IV) cannot be extracted with HMMCO in the absence of thiocyanate. Therefore, V(V) is first extracted

TABLE 3  
Separation of Metals from Binary Mixtures

Amount taken ( $\mu\text{g}$ )				Amount found ( $\mu\text{g}$ )			
V(V)	Nb(V)	Ta(V)	Ti(IV)	V(V)	Nb(V)	Ta(V)	Ti(IV)
89.6	86.2	—	—	91.2	87.3	—	—
358.4	344.8	—	—	352.2	340.6	—	—
717.8	689.6	—	—	725.8	680.2 <sup>a</sup>	—	—
89.6	—	108.0	—	89.2	—	108.1	—
268.8	—	324.0	—	272.0	—	320.5	—
806.4	—	972.0	—	821.3 <sup>a</sup>	—	960.1 <sup>a</sup>	—
268.8	—	—	283.4	272.5	—	—	280.9
537.6	—	—	566.8	530.8	—	—	560.8
1075.2	—	—	1133.6	1060.2	—	—	1115.2

<sup>a</sup>Extract diluted to 3 times.

in the absence of thiocyanate from hydrochloric acid solution and then Nb(V), Ta(V), or Ti(IV) is extracted by adding thiocyanate to the aqueous phase. The optimum conditions of extraction and the general procedures employed for the separation are the same as those given in their individual determinations. The results (Table 3) show that V(V) can be very well separated and determined from Nb(V), Ta(V), or Ti(IV) present in binary mixtures.

Thus the method is simple, rapid, and selective. It is possible to accomplish clean-cut separation of vanadium from niobium, tantalum, and titanium with which it is usually associated. The method is applicable at tracer concentrations of metal ions.

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