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Extraction and Spectrophotometric Determination of Vanadium (V) from *N-m*-Tolyl-o-Methoxybenzohydroxamic Acid

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NOTE

Extraction and Spectrophotometric Determination of Vanadium(V) from *N*-*m*-Tolyl-o-Methoxybenzohydroxamic Acid

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Abstract

The reagent *N*-*m*-tolyl-*o*-methoxybenzohydroxamic acid is proposed for the rapid extraction and simultaneous spectrophotometric determination of vanadium in the presence of commonly occurring metal ions. It forms an intense violet colored complex with vanadium and the complex can be extracted by chloroform from 6–8 *M* HCl. The violet complex obeys Beer's Law at λ_{max} (550 mm) with the molecular absorptivity 6.5×10^3 and sensitivity 0.01 μg of V/cm^2. The effects of acidity, reagent concentration, and diverse ions on the extraction are discussed.

INTRODUCTION

Hydroxamic acids are the selective and sensitive reagents for the determination of vanadium(V) (*1–6*). Molybdate, tungstate, titanium, and zirconium are most likely to interfere in the spectrophotometric determination of vanadium. In the present communication the *N*-*m*-tolyl-*o*-methoxybenzohydroxamic acid (*N*-*m*-T-*o*-MBHA) is proposed to be a selective and sensitive reagent for vanadium(V). The use of *N*-*m*-T-*o*-MBHA for the rapid extraction and simultaneous determination of microgram quantities of vanadium(V) is discussed in detail.

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EXPERIMENTAL

N-m-Tolyl-*o*-methoxybenzohydroxamic acid was prepared freshly from *N-m*-tolylhydroxylamine and *o*-methoxybenzoylchloride [mp 110°, reported 111° (7)]. Its purity was checked by elemental analysis, IR, and UV spectra. A 0.1-*M* reagent solution in ethyl alcohol–free chloroform was used for extraction work. Ethyl alcohol was removed by washing the commercial chloroform five or six times with about half of its volume of distilled water, and it was distilled after drying over fused calcium chloride.

An aqueous solution of vanadium was prepared by dissolving 0.626 g ammonium meta vanadate in a liter of distilled water. Its vanadium content was determined with a standard iron(II) solution.

The absorption spectra were recorded with a recording spectrophotometer (Model SF-10, USSR). Absorbance at a particular wavelength was measured with a single beam spectrophotometer (Model SF-4, USSR) using 10-mm cells.

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Extraction and Spectrophotometric Determination of Vanadium (V)

Five milliliters of vanadium(V) (6.15×10^{-4} *M*) solution was transferred into a 100-ml separatory funnel along with 5 ml of hydrochloric acid (6–8 *M*), followed by 5 ml of 0.1 *M* chloroform solution of the reagent (*N-m*-T-*o*-MBHA). The content was shaken for 10–15 min and the chloroform layer was allowed to separate. The violet extract thus obtained was dried over anhydrous sodium sulfate (A.R., B.D.H.) to remove the moisture and transferred into a 25-ml volumetric flask. To ensure complete recovery of vanadium, the aqueous layer was extracted twice with 5 ml of reagent solution and sodium sulfate was washed with 2 ml (× 3) of chloroform to remove the last traces of violet color. Finally the extracts were diluted to the mark. The absorbance of the violet complex was measured at 550 nm against chloroform as the blank. The results obtained were found to be accurate within ±0.5%.

RESULTS AND DISCUSSION

The absorption spectra (Fig. 1) show that the violet extracts exhibit maximum absorbance at 545–550 nm. The reagent does not absorb between 300 and 600 nm. At 500 nm the absorbance is reproducible and all the measurements were made at this wavelength. The characteristics of the violet vanadium(V)-*N-m*-T-*o*-MBHA complex are given in Table 1.

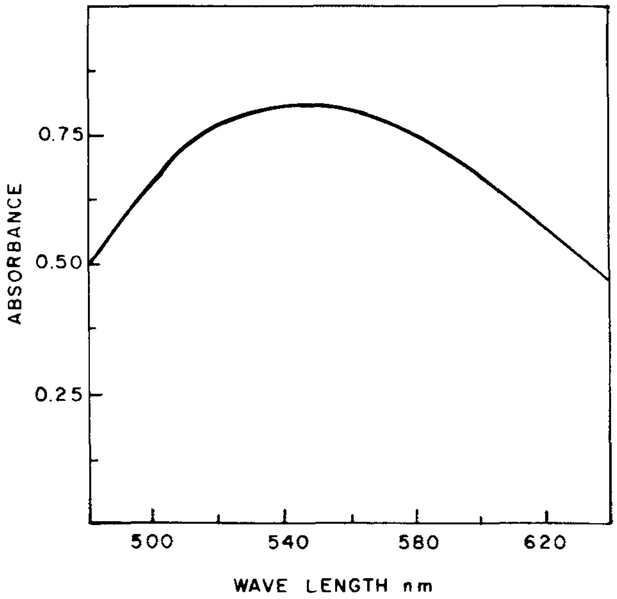

FIG. 1. Vanadium—*N*-*m*-tolyl-*o*-methoxybenzohydroxamic acid complex vs chloroform.

TABLE 1

Spectral Characteristic of Vanadium–*N*-*m*-Tolyl-*o*-methoxybenzohydroxamic Acid Colored System in Chloroform

Concentration of acid (*M*)	Color of chloroform extract	Wavelength of maximum absorption (nm)	Molecular absorptivity (ε)
0.01	Amethyst	480	4100
0.10	Amethyst	480	—
1.00	Reddish violet	510	5200
2.00	Reddish violet	530	—
4.00	Reddish violet	530	—
6.00	Violet	550	6500
7.00	Violet	550	6500
8.00	Violet	550	6500

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The Effect of Acidity, Reagent, and Time

The maximum color intensity was obtained with 6–8 *M* HCl and was found to be stable for several days (*8*, *9*). The reagent had no effect on the color system; hence even a large excess of it could be used for extraction purposes. The effect of acidity on the extraction system is shown in Fig. 2, and the distribution data at different pH are given in Table 2. It can be seen that extraction is complete at pH 1 or <1. The distribution ratio is infinite (Fig. 2, Table 2) at pH 1 or <1.

Beer's Law

The plot of absorbance (at 550 nm) vs concentration of vanadium gives a straight line which passes through the origin. Beer's law is obeyed

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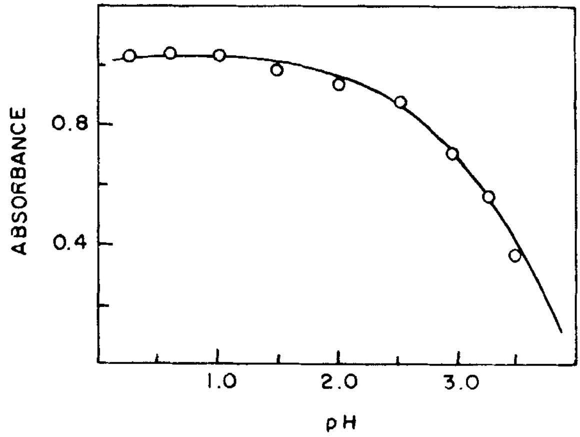

FIG. 2. Effect of acidity in extraction of vanadium(V).

TABLE 2

Extraction of Vanadium(V)–*N*-*m*-T-*o*-MBHA Complex as a Function of pH

pH	Extraction (%*E*)	Distribution ratio (*D*)
0.2	100	∞
0.3	100	∞
0.5	100	∞
1.0	100	∞
1.5	90	30.0
2.0	60	10.0
2.5	30	0.8
3.0	15	0.2

over the range of 0.05 to 15 μg/ml vanadium at 550 nm. The sensitivity of he reagent defined by Sandell (*10*) was found to be 0.01 μg of V/cm^2 and the molecular extinction coefficient worked out to be 6.5 × 10^3 at 550 nm.

Effect of Diverse Ions

The present method was followed to study the interference due to various diverse ions in direct spectrophotometric determination of vanadium(V) with *N*-*m*-T-*o*-MBHA: Be^{2+} (30 mg), Mg^{2+} (30 mg), Ca^{2+} (30 mg), Sr^{2+} (30 mg), Ba^{2+} (30 mg), Ti^{4+} (20 mg), Zr^{4+} (20 mg), Fe^{3+} (25 mg), Co^{2+} (30 mg), Ni^{2+} (25 mg), Cu^{2+} (30 mg), Zn^{2+} (30 mg), Cd^{2+} (30 mg), Hg^{2+} (30 mg), Al^{3+} (30 mg), Pb^{2+} (30 mg), As^{3+} (30 mg), rare earths (30 mg), and 50–100 mg of chloride, fluoride, citrate, tartrate, oxalate, and phosphate did not interfere with the determination of 75 μg V in 25 ml.

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Analytical Data

To test the reliability of the present method, different amounts of vanadium were extracted and determined. The data given in Table 3 show that there is good agreement in the experimental values.

TABLE 3

Analytical Data on Extraction of Vanadium(V)

Vanadium(V) (μg/25 ml of chloroform)	Vanadium(V) found (μg)	Error	Standard deviation, σ
2	1.99	−0.01	0.01
4	3.99	−0.01	0.01
8	8.01	+0.01	0.02
10	10.00	0.00	0.01
50	50.00	0.00	0.01
100	99.98	−0.02	0.03
200	200.03	+0.03	0.02

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