

## A NEW SELECTIVE TEST FOR DETECTION OF MICRO- QUANTITIES OF VANADIUM IN MIXTURE OF INORGANIC COMPOUNDS

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### **Abstract**

A highly selective and sensitive test for the detection of micro-quantities of Vanadium (IV, V) employing mixed ligands N-o-tolylbenzohydroxamic acid (O-TBHA) and tri-n-butyl phosphate (TBP) is reported. In concentrated hydrochloric acid medium (> 9N) a bluish-violet extract is obtained in TBP which acts both as solvent and ligand. The limit of identification in terms of vanadium (V) is 40  $\mu\text{g}\cdot\text{ml}^{-1}$ . Various ions including Ag (II), Al (III), Au (III), Bi (III), Cd (II), Ce (IV), Fe (III), La (III), Mg(II), Mo (IV), NI (II), Pb (II), Pd (II), Pr (III), Sb (III), Sn (II), Th (IV), Ti (IV), U (VI), W (VI), Zr (II), Zr (IV) etc. do not interfere. Interferences due to Cu (II), Co (II) and Tl (I) are eliminated by performing test in sulphuric acid media

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### **Keywords:**

Ligand;

N-o-

tolylbenzohydroxamic  
acid;

Tris-n-butyl phosphate;

Vanadium;

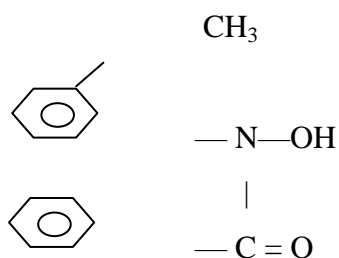
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## 1. Introduction

Among a number of procedures in the literature for detection of vanadium (V) [1-13] those using hydroxamic acids are fairly sensitive. In present communication a new test is developed for vanadium. The vanadium (V) forms a bluish-violet to blue colored complex with N-o-tolylbenzohydroxamic acid (o-TBHA) in presence of tris-n-butyl phosphate (TBP) from concentrated acid (> 9N HCl) solution.



o-TBHA

o-TBHA forms violet complex with vanadium (V). TBP also acts as a complexing agent for vanadium (V) and solvent for extraction of resultant mixed complex. The extracted species is intensively colored and stable hence it has been used for the development of selective test for detection of micro-quantities of vanadium (V).

## 2. Experimental

### Reagents:

**o-TBHA:** N-o-tolylbenzohydroxamic acid (o-TBHA) was synthesized by the method developed by Tandon and Priyadarshini [14], [15] and used by many workers including Sharma and Rajput [16].

**Reagent Solution:** 0.1 % w/v solution of o-TBHA in TBP (LR, BDH) was used.

**Vanadium Solution:** 0.00854 M solution of ammonium metavanadate (AR, Reanal-Hungary) was prepared in glass distilled water, and the content was determined volumetrically [17]. Oxidation of vanadium (IV) is not necessary because in both tetravalent and pentavalent states vanadium gives the test.

**Diverse Ions Solution:** The solutions of diverse ions were prepared following the procedure of West [18].

### 3. Procedure

Placed a drop of test solution in ignition tube (0.4 x 5 cm) and then added 3 to 4 drops of concentrated hydrochloric acid followed by 1-2 drops of reagent solution. A bluish-violet to blue color appears in organic phase depending upon the amount of vanadium present.

In presence of Co (II), Cu (II) and Tl (I) test must be performed by using sulphuric acid instead of hydrochloric acid.

### 4. Results and Discussion

The test is given by both vanadium (IV) and vanadium (V) hence no oxidation of vanadium (IV) to vanadium (V) is necessary. Limit of Identification in terms of vanadium (V) is  $40 \mu\text{g}, \text{ml}^{-1}$ .

Metal ions either commonly associated with vanadium (V) or usually interfering in the detection of vanadium (V) by reported procedures (1-9) do not interfere in this test. Thus large quantities of Ag (II), Al (III), Au (III), Bi (III), Cd (II), Ce (IV), Fe (III), La (III), Mg(II), Mo (IV), Ni (II), Pb (II), Pd (II), Pr (III), Sb (III), Sn (II), Th (IV), Ti (IV), U (VI), W (VI), Zr (II), Zr (IV) etc. did not interfere in this test.

If Co (II) is present in the test solution, addition of hydrochloric acid produces blue color [19] which partly goes in to TBP and therefore in presence of Co (II), the test loses its reliability. Cu (II), if present, is extracted in TBP as orange colored species under experimental conditions. Hence, partial interference is experienced. Tl (I), forming precipitate with hydrochloric acid interferes in the test. Therefore, it is recommended that in presence of Co (II), Cu (II) and Tl (I) test must be performed using sulphuric acid instead of hydrochloric acid. Ag (I) also forms precipitate with hydrochloric acid but does not interfere in the detection of vanadium (V).

The test is highly selective and sensitive for vanadium (IV, V). Besides, it does not require any rapid control of experimental conditions.

## 5. Acknowledgement

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## 6. Conclusion

A highly selective and sensitive test for the detection of micro-quantities of Vanadium (IV, V) employing mixed ligands N-o-tolylbenzohydroxamic acid (O-TBHA) and tri-n-butyl phosphate (TBP) has been developed. This test may be used for detection of vanadium (IV) and vanadium (V) present in micro-quantities in inorganic mixtures even in presence of many other metal ions.

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