

## Extractive Spectrophotometric Determination Of V (V) By Using 2-Hydroxy-3-Methoxy Benzaldehyde Thiosemicarbazone As An Analytical Reagent.

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### Abstract:-

2-Hydroxy-3-methoxy benzaldehyde thiosemicarbazone (2H3MBTS) is proposed as a new photometric reagent for the extractive spectrophotometric determination of V(V). 2H3MBTS reacts with V(V) and form a stable colored complex in the pH range 6.2 to 7.6. This was well extracted in n-butanol. The absorption spectrum of V(V) and (2H3MBTS) complex in n-butanol shows maximum absorbance at 390 nm. The system obeyed Beer's law up to  $1.8 \mu\text{g} / \text{cm}^3$ . The molar extinction coefficient was found to be  $18.20 \times 10^2 \text{L mol}^{-1} \text{cm}^{-1}$  and the sensitivity of the method as defined by Sandell's sensitivity was  $0.02797 \mu\text{g cm}^{-2}$ . The Composition of the extracted species was determined by Job's Continuous variation method, Mole ratio method and slope ratio method and it was found to be 1:2. The proposed reagent is satisfactorily applied for the determination of trace amount of V(V) from synthetic and commercial samples.

**Keywords:** Solvent Extraction, Sandell's sensitivity, Vanadium, n-butanol, 2-Hydroxy-3-methoxy benzaldehyde thiosemicarbazone (2H3MBTS) etc.

**Introduction:-** Vanadium is a hard, ductile, silver-gray metal. Some sources describe vanadium as "soft", perhaps because it is ductile, malleable and not brittle. Vanadium is harder than most metals and steels. It has good resistance to corrosion and it is stable against alkalis, sulfuric and hydrochloric acids. Approximately 85% of vanadium produced is used as ferrovanadium or as a steel additive. Vanadium forms stable nitrides and carbides, resulting in a significant increase in the strength of the steel. From that time on vanadium steel was used for applications in axles, bicycle frames, crankshafts, gears, and other critical components. There are two groups of vanadium containing steel alloy groups. Carbon steel alloys contain 0.15% to 0.25% of vanadium. Vanadium is compatible with iron and titanium, therefore vanadium foil is used in cladding titanium to steel. All Vanadium compounds should be considered toxic. Vanadium compounds are poorly absorbed through the gastrointestinal system. Inhalation exposures to vanadium and vanadium compounds result primarily in adverse effects on the respiratory system.

**Experimental Procedure for the Extraction:** An

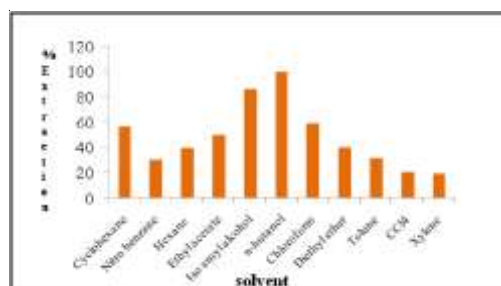
aliquot of solution containing 1mL of 20ppm of vanadium was taken. To this 1mL of (2H3MBTS) reagent is mixed. The pH of the solution adjusted to 7.0, & noted that the total volume should not exceed than 10mL. The solution was transfer to the 125mL of separating funnel & equilibrated with 10mL of n-butanol solution. The separating funnel was shaken vigorously and allowed to stand for some time to separate the two phases. The aqueous phase is separated and the organic phase is passed through anhydrous sodium sulphate in order to absorb water and then collected in 10mL volumetric flask and dilute up to the mark with n-butanol. The absorbance was measured at  $\lambda_{\text{max}} = 390 \text{nm}$  on a Shimadzu UV-Visible 2100 Spectrophotometer with 1cm quartz cells.

### Result and Discussion:

The results of various studies were discussed as given below:

#### Effect of solvent on extraction:

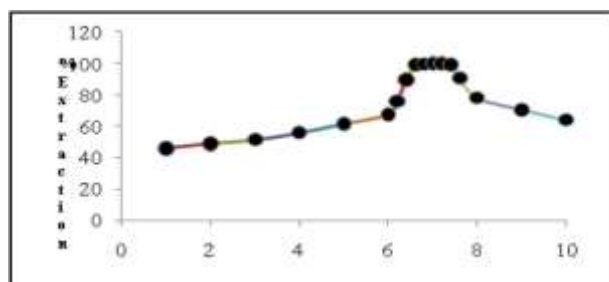
n-Butanol is chosen as solvent, since it was found that the metal complex V(V)(2H3MBTS) complex in n-butanol shows maximum absorbance at 460nm.



**Effect of pH on extraction:**

The absorbance of the complex V (V)(2H3MBTS) was measured as a function of pH of the aqueous phase. The complexation of V (V) was

carried out at pH 1-10. From which pH range is 6.2-7.6 is selected. The data obtained shows maximum absorbance at pH 7.0.

**Effect of reagent concentration**

The effect of variation in the concentration of OVTS in the range of 0.2 to 2.0 cm<sup>3</sup> of 0.1% OVTS on the extraction and on colour development was studied. It was observed that 1.0 cm<sup>3</sup> of 0.1 % OVTS was sufficient for complete extraction and for colour development. Hence, for subsequent studies 1.0 cm<sup>3</sup> of 0.1 % OVTS was employed.

**Equilibration time**

The absorbance by V (V): OVTS complex was checked by varying the time equilibration from 30 sec. to 40 min. It was observed that the equilibration time of 1 min. was sufficient for quantitative extraction of vanadium.

**Stability of complex with time**

For the study of 1μg V (V) was extracted with the reagent in n-butanol and absorbance of n-butanol extract was measured at different intervals of time. The study of stability of complex with variation in time shows that the complex was stable up to 36 hours after which the absorbance decreases slowly.

**Effect of salting out agent**

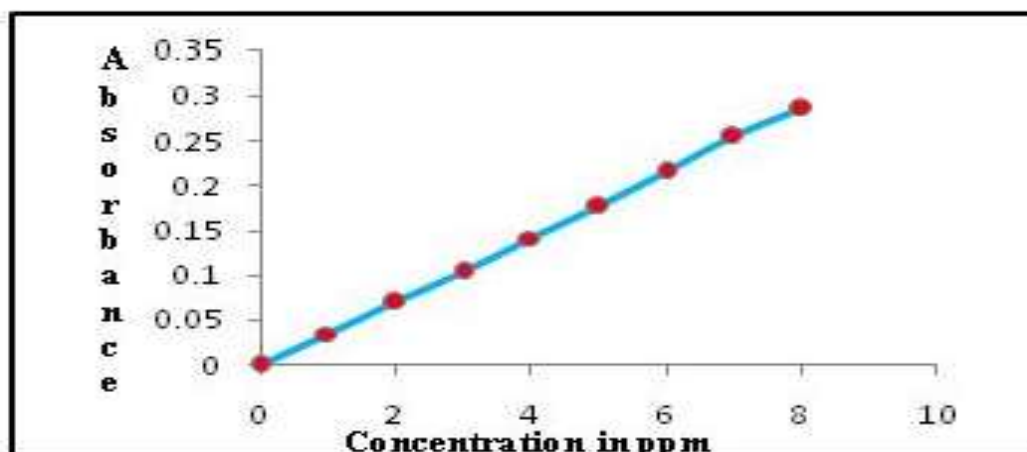
Different salting out agents as sulphates, chlorides, carbonates, and nitrates of sodium, potassium, barium, magnesium, ammonium, and calcium were used in the extraction of V (V). It was observed that there was no effect on extraction.

**Calibration curve**

Different amounts of the V (V) from 1 μg/cm<sup>3</sup> to 8 μg/cm<sup>3</sup> were extracted quantitatively under optimum experimental conditions and the plot of absorbance against concentration of V (V) gave a straight line indicating that the Beer's law is obeyed in this range. The molar absorptivity calculated on the basis of total V (V) taken was found to be  $18.20 \times 10^2 \text{ Lmol}^{-1} \text{ cm}^{-1}$  and sensitivity of the method as defined by Sandell's sensitivity which is  $0.02797 \mu\text{g cm}^{-2}$ .

**Effect of divalent ions and foreign ions:**

The effect of diverse ions on the V (V) was studied, in the presence of foreign ions. The ions which show interference in the spectrophotometric determination of Vanadium were overcome by using appropriate masking agent as given

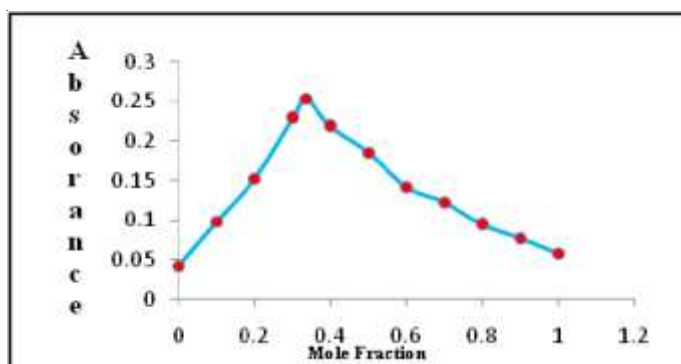


Sr.no.	Interfering ions	Masking agent
1	Fe(III)	Thiourea
2	Ce(IV)	Sodium fluoride.
3	Cu(II)	Sodium Thiosulphate
4	Ni(II)	DMG
5	CN <sup>-1</sup>	Boiled with concentrated HNO <sub>3</sub> and formaldehyde.
6	EDTA	Boiled with concentrated HNO <sub>3</sub>

**Nature of extracted species:**

The composition of extracted V(V)(2H3MBTS) complex has been determined by Job's continuous variation method, Slope method & Mole ratio

method. It shows that the composition of V (V) (2H3MBTS) complex is 1:2

**Application:**

The proposed method was successfully applied for the determination of Vanadium from various synthetic mixtures, industrial waste and alloys and commercial samples etc. The results obtained

were found to be in good agreement with those obtained by the standard method as given in below table.

Observation Table For Determination of V (V) Using (2h3mbts) From Different Samples

Synthetic Samples			
Composition of Sample (mg)	Amount of Vanadium (µg)	Standard method (µg)	Present method (µg)
Al+ Mn+ Cd+ V	8	7.98	7.975
Ti+Fe +V	6	5.8	5.76
Pharmaceutical samples			
Tablet	9.8	9.78	9.70
Steel Alloys			
Carbon steel	5	4.9	4.85
Ferro Vanadium	1.0	0.98	0.96

**Conclusions:**

The method required simple apparatus which have low cost. This method offer several silent features such as rapidity, selectivity and simplicity. The other associated elements do not interfere in the determination. Hence the proposed method is recommended for the determination of V (V) with (2H3MBTS) by spectrophotometric method, at trace level analysis

of various alloys, synthetic mixture.

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