



## International Journal of Advanced Research in Chemistry and Chemical Engineering

### “EXTRACTIVE SPECTROPHOTOMETRIC STUDIES OF ACETOPHEN ONE 2', 4'- DIHYDROXY THIOSEMICARBAZONE WITH URANIUM”

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**ABSTRACT**— *Acetophenone 2',4'-Dihydroxy thiosemicarbazone (APD HTS) is used as a reagent for the extractive spectrophotometric determination of Uranium. The reagent APDHTS gave instantaneous and stable Orange colour with Uranium at pH 7.4. The color reaction in detail has been explored and the possibility of photometric determination of the micro amounts of Uranium is established with necessary conditions. A linear calibration graph over the concentration range 1 ppm to 12 ppm with a 3σ limit of detection of 0.486 ppm was obtained by applying the spectrophotometric method at wavelength 370 nm. The stoichiometry of the complex is established as 1:2 (M: L) by Job's method of continuous variation and confirmed by mole ratio method. The standard deviation and the coefficient of variations are presented. The molar absorptivity and Sandell's sensitivity of the complex is  $0.9881 \times 10^4 \text{ L mol}^{-1}\text{cm}^{-1}$  and  $0.04819 \mu\text{g cm}^{-2}$  respectively. The results of the prescribed procedure applied for the determination of the micro amounts of U (VI) in various synthetic and standard steel samples are presented.*

**Keywords:** Extractive Spectrophotometric Determination, acetophenone 2',4'-dihydroxy thiosemicarbazone (APDHTS), Uranium, Beer's Law, Molar Absorptivity, Sandell's Sensitivity.

### 1, INTRODUCTION

Uranium is found in earth's crust in abundance. The metal is an important alloying element and is present as minor constituent in many industrially important materials. Even small amounts cause tremendous generation of energy during nuclear reaction. The effect to metal



exposure to human is dangerous very minute quantity can cause long term and permanent damage. The increasing use of the metal necessitates development of rapid and sensitive methods for the determination of minute quantities of the metal. Colorimetric and Atomic Emission or Atomic Absorption methods are mostly used for the determination of U (VI). However, colorimetric methods are generally preferred; as they involve less expensive instrumentation and afford better sensitivity when appropriate chromogenic reagents and solvent extraction pre-concentration steps are employed. Most of the extractive spectrophotometric methods developed for U (VI) are based on reactions with suitable colour producing reagents. However, most of the existing methods suffer from limitations such as longer periods of time for phase separations, weak stability of coloured complexes and interferences from metal ions like tungsten, tin, antimony and anions and various complexing agents. In the present paper, extractive spectrophotometric determination of Uranium with Acetophenone 2', 4'- Dihydroxy thiosemicarbazone (A PDHTS) is presented.

## 2, EXPERIMENTAL

Electrochemically and spectrally pure water obtained from triple distillation of deionised feeder water was used for conducting analytical operations. All the chemicals were of Analytical grade quality. The inorganic salt solutions of various metal ions in higher concentrations were prepared by dissolving appropriate salts in requisite quantities in triple distilled water to give a solution containing 200 ppm and few drops of a suitable acid were added before dilution wherever necessary to prevent hydrolysis. A number of buffer solutions of constant ionic strength of 0.2 M covering a wide pH range were prepared. The ligand APDHTS was synthesized, recrystallised, dried and used for preparing a 0.1% solution in methanol. Solutions of ions for interference studies were prepared by dissolving the amount of each compound needed to give 200 ppm of the ion concerned. All the solutions were stable for several weeks.

### 2.1, PROCEDURE FOR THE EXTRACTION

1.0 mL of aqueous solution containing 2 $\mu$ g of Uranium metal and 1 mL of reagent were mixed in a 50 mL beaker. The pH of the solution adjusted to 7.4 It must be noted that the total volume should not exceed 10 mL. The solution was transferred to 100 mL separatory funnel. The beaker was washed twice with n-butanol and transferred to the same funnel. The two phases were shaken for two minutes and allowed to separate. The organic phase was passed through anhydrous sodium sulphate in order to absorb trace amount of water from



organic phase and then collected in 10 mL measuring flask and made up to the mark with organic solvent if required. The amount of Uranium present in the organic phase determined quantitatively by spectrophotometric method by taking absorbance at 370 nm.

### 3, RESULTS AND DISCUSSION

The results of various parameters involved during the extraction is discussed below

#### 3.1, EXTRACTION AS A FUNCTION OF Ph

The optimum pH range in which the metal complex shows maximum and constant absorbance was carried out at different pH range. The maximum extraction was obtained in pH range 6.6 - 8.0 and for all further extractions the pH was maintained at 7.4 (Fig no.1).

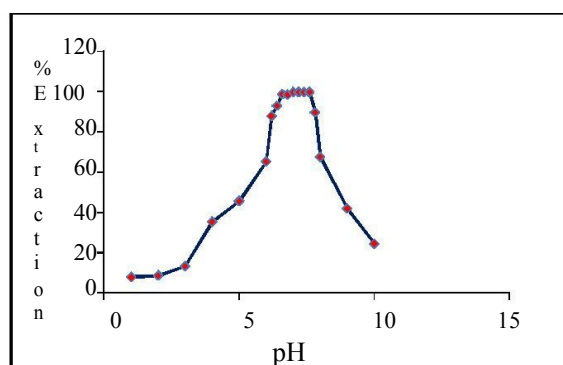


Fig No.1

#### 3.2, ABSORPTION SPECTRUM

The absorption spectra of U (VI): Acetophenone 2', 4'- Dihydroxy thiosemicarbazone (APDHTS) complex was recorded from 200-800 nm against reagent blank. From the spectra, the absorption maximum ( $\lambda$  max) of the complex is observed between the range 360-370 nm at which, the absorbance of the metal ion and the ligand are negligible or nil. Hence for the spectrophotometric study, all the measurements were carried out at 370nm.

#### 3.3, EFFECT OF SOLVENTS AND EQUILIBRATION TIME

The absorbance value and the percentage of extraction of U (VI) - APDHTS was done with various solvents and it was observed that n-butanol gave the quantitative extraction in just 1 min.



### 3.4, EFFECT OF REAGENT CONCENTRATION AND STABILITY OF THE COMPLEX

The minimum amount of reagent to acquire maximum colour intensity with a given amount of U (VI) ion was found that 2 ml of the reagent is sufficient for colour development. The absorbance of the complex is stable even up to 36 hrs.

### 3.5, APPLICABILITY OF BEER'S LAW

A linear plot was obtained when the measured absorbance values are plotted against the amount of U (VI) in the concentration range of 1-12 ppm between 370 nm (Fig.no.2). The molar absorptivity and Sandell's sensitivity of the complex is  $0.4881 \times 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$  and  $0.04819 \mu\text{g cm}^{-2}$  respectively.

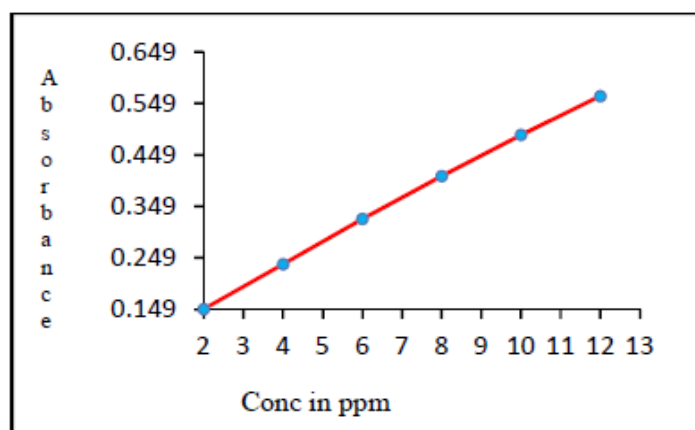


Fig.No.2

### 3.6, THE NATURE OF THE EXTRACTED SPECIES

The composition of the complex responsible for the observed Orange colour in the reaction between the metal ion and the APDHTS is established from the Job's method, mole ratio method and slope ratio method was found to be 1:2 (M:L).

### 3.7, EFFECT OF DIVALENT IONS AND FOREIGN IONS

The effect of other ions present in various amount indicated no interference in the spectrophotometric determination of 10 ppm of Uranium. The ions which show interference in the spectrophotometric determination of Uranium were overcome by using appropriate Masking agents. (Table no 1).



**Table No. 1. MASKING AGENTS**

Sr. No.	Interfering Ion	Masking Agent
1	Ni (II)	Sodium cyanide
2	Fe(III)	Sodium Fluoride
3	Cu (II)	Sodium thiosulphate
4	Mo (VI)	Citrate
5	Ag (I)	Potassium iodide
6	V (V)	Thiourea

### 3.8, PRECISION AND ACCURACY

The precision and accuracy of the developed spectrophotometric method have been studied by analyzing ten solutions each containing 10  $\mu\text{g}$  of Uranium in the aqueous phase. The average of ten determinations was 9.9813 and variation from mean at 95% confidence limit was  $\pm 0.0313$ .

### 3.9, APPLICATION

The proposed method was successfully applied for the determination of Uranium from various alloys, ores and pharmaceutical samples. The results found to be in good agreement with those obtained by the standard known method (Table 2).

**Table No. 2.**

Synthetic Samples			
Composition of Sample (mg)	Amount of U(VI) (mg)	Standard method	Present method
Zn(4), W(6), Co(8), U(6)	6	5.97	5.85
Zr(5), Ti(2), U(4)	4	4	3.93
Ores/Minerals			
Monazite Sand	10	9.9	9.85
lignite	6	5.8	5.74



#### 4, CONCLUSION

The proposed method permits the determination of trace amounts of Uranium without any prior separation. The major advantage of the proposed method is that the colour development is instantaneous at room temperature and stable. The proposed method has higher sensitivity and selectivity than that of the methods reported earlier.

#### ACKNOWLEDGEMENT

I take this opportunity to thank my research guide Dr. R. S. Lokhande and Co-guide Dr. S.M. Pitale for their valuable and checkered guidance. I am also thankful to my sir Dr. Suhas Janwadkar who helped me to enter the research world. I thank my colleague Dilip Yadav who helped me in many ways. Last but not the least I thank my family member who stood behind me in each and every situation.

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