



RESEARCH ARTICLE

ANALYTICAL CHEMISTRY

**SOLVENT EXTRACTION AND SPECTROPHOTOMETRIC  
DETERMINATION OF Cu(II) BY USING ACETOPHENONE 2',5'-  
DIHYDROXY SEMICARBAZONE AS AN ANALYTICAL REAGENT**

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### ABSTRACT

A spectrophotometric method has been developed for the determination of Cu(II) using Acetophenone 2',5'-dihydroxy, semicarbazone<sup>1</sup> as an extractive reagent. The reagent forms a colored complex which has been quantitatively extracted into n-Butanol at pH 5.6. The method obeys Beer's law over a range of 1 to 10 ppm. The molar absorptivity is  $2564.1 \text{ L mol}^{-1}\text{cm}^{-1}$  and Sandell's sensitivity is  $0.02484 \mu\text{g cm}^{-2}$  respectively. The proposed method is very sensitive and selective. This method has been successfully applied to synthetic and commercial samples.



## KEY WORDS

Copper, Spectrophotometric determination, n-Butanol, Acetophenone 2', 5'-dihydroxy, semicarbazone derivative.

## INTRODUCTION

The cursory look at the literature survey reveals the fact that Copper reacts with many organic reagent. It also indicates that some of the reagents suffer from limitations such as interference of Ni(II)<sup>2</sup>, Co(II)<sup>2</sup>, W(VI)<sup>3</sup>, Zn(II)<sup>3</sup> etc. In some cases, complex formation takes place after several minutes<sup>5,4</sup>. Some of the reagents are not selective<sup>6,7</sup> and sensitive. Some are less stable<sup>8</sup>. In this paper, a new method has been developed using Acetophenone 2', 5'-dihydroxy, semicarbazone [ADHS] for extraction and Spectrophotometric determination of Copper, Cu (II), which is simple, selective and sensitive.

## EXPERIMENTAL

The reagent Acetophenone 2', 5'- dihydroxy semicarbazone was synthesized by the given procedure. The stock solution of Cu (II) was prepared by dissolving a weighed amount of ammonium copper sulphate in double distilled water and then diluted to the desired volume with double distilled water and standardized by Diethyldithiocarbamate method. The absorbance and pH measurements were carried out on a Shimadzu UV-Visible 2100 spectrophotometer with 1 cm quartz cells and digital pH meter with combined glass electrode respectively.

### **PROCEDURE FOR THE EXTRACTION:**

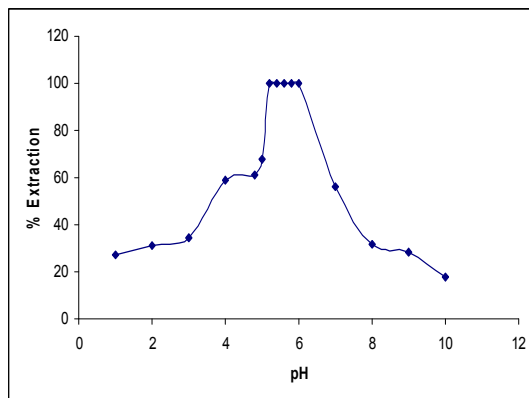
0.1 ml of aqueous solution containing 1µg of Copper metal and 1 ml of reagent was mixed in a 50 ml beaker. The pH of the solution was adjusted to 5.6. It must be noted that the total volume should not exceed 10 ml. The solution was transferred to 100 ml separatory funnel. The beaker was washed twice 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 Copper present in the organic phase was determined quantitatively by spectrophotometric method by taking absorbance at 380 nm and that in the aqueous phase was determined by diethyldithiocarbamate method.

## RESULTS AND DISCUSSION

The results of various studies are discussed below.

### **Extraction as a function of pH:**

The extraction of Copper with Acetophenone 2',5'-dihydroxy ,semicarbazone has been studied over the pH range 1-10 and has been observed that percentage extraction of Cu (II) is maximum at pH 5.6.(Fig 1).



**Extraction as a function of pH  
( Fig 1)**

**Absorption spectrum:**

The absorption spectrum of Cu (II): Acetophenone 2',5'-dihydroxy semicarbazone in n-butanol shows the maximum absorption at 380 nm. The absorption due to reagent at this wavelength is nearly negligible. Hence, the absorption measurements are carried out at 380nm.

**Influence of diluents:**

The suitability of solvent was investigated using various organic solvents and the extraction of Cu (II): ADHS was quantitative in n-butanol. Hence, n-butanol was used for further extraction studies as it gave better and quicker phase separation.

**Effect of reagent concentration:**

It was found that 1 ml of 0.1% reagent is sufficient for the colour development of the

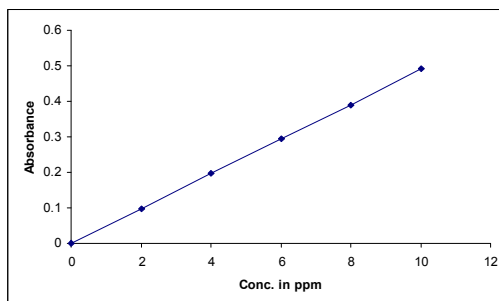
metal Cu (II) in 10 ml of aqueous solution at pH 5.6.

**Effect of equilibration time and stability of the complex:**

The equilibration time of 1 minute is sufficient for the quantitative extraction of Copper. The stability of colour of the Cu (II): ADHS complex with respect to time shows that the absorbance due to extracted species is stable up to 32 hours, after which slight decrease in absorbance is observed.

**Calibration plot:**

The Beer's law is obeyed from 1 to 10 ppm. The molar absorptivity and sandell's sensitivity are calculated to be is  $2564.1 \text{ L mol}^{-1}\text{cm}^{-1}$  and  $0.02484 \mu\text{g cm}^{-2}$  respectively. (Fig 2).



**Calibration plot  
(Fig 2)**

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

The effect of other ions present in various amount indicated no interference in the spectrophotometric determination of 10ppm

of Copper. The ions which showed interference in the spectrophotometric determination of Copper were overcome by using appropriate masking agents. (Table 1)

**Table 1**  
**Use of masking agent**

Sr. No.	Interfering Ion	Masking agent
1	Ce(IV)	Sodium fluoride
2	Fe(III)	Thiourea
3	U (VI)	8-Hydroxy quinoline
4	Cr ( II)	Ammonium acetate
5	Mo (VI)	Citrate
6	EDTA	Boiled with concentrated HNO <sub>3</sub>
7	CN <sup>-</sup>	Boiled with concentrated HNO <sub>3</sub> and formaldehyde

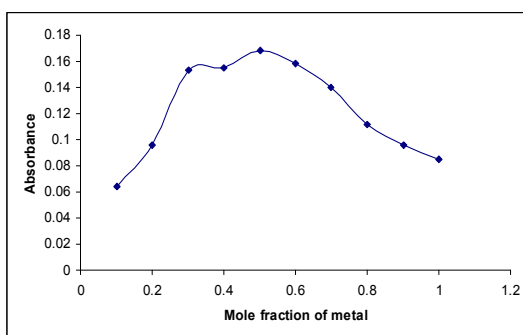
**Precision and accuracy:**

The precision and accuracy of the developed spectrophotometric method was studied by analyzing ten solutions each containing 10μg of Copper in the aqueous phase. The average of ten determinations was 10.0019 and variation

from mean at 95% confidence limit was ± 0.01007.

**Nature of extracted species:**

The composition of extracted Cu (II): ADHS complex was determined by Job's continuous variation method, Slope ratio method and Mole ratio method. It showed that the composition of Cu (II): ADHS complex is 1:1.(Fig 3).



**Nature of extracted species**  
**(Fig 3)**

**Application:**

The proposed method was successfully applied for the determination of Copper 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 2**  
**Applications in pharmaceutical samples**

Sr. No.	Sample	Certified value	Present method
<b>Copper alloys:</b>			
1	Brass	60%	59.98%
2	Cupra – Nickel	35%	34.89
<b>Milk Sample:</b>			
1	Raw Milk	4.3 x 10 <sup>3</sup>	4.3 x 10 <sup>3</sup>
<b>Pharmaceutical Sample</b>			
1	Multi vitamin capsule	5.0mg	4.89mg
<b>Synthetic mixture:</b>			
1	Cu(55)+Zn(45)	55 µg	54.51µg
2	Cu(100)+Zn(100)+ Cd(100)	100µg	99.10µg
<b>Industrial Waste:</b>			
1	Water at Boisar Creek	4.2 ppm	4.1ppm
1.	<b>Each result is average of three independent determinations.</b>		
2.	<b>Each result is compared with diethyldithiocarbamate method.</b>		

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