



## DETERMINATION OF TRACE AMOUNT OF Cu(II) USING 3', 5'-DINITRO SALICYALDEHYDE SEMICARBAZONE AS AN ANALYTICAL REAGENT BY SOLVENT EXTRACTION AND SPECTROPHOTOMETRIC METHOD

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### ABSTRACT

A simple precise spectrophotometric method is coupled with solvent extraction technique used for determination of Cu(II) with 3',5'-Dinitro Salicylaldehyde Semicarbazone [3',5'DNSAS] as an analytical reagent. Reagent forms a complex with Copper metal producing a yellow coloured stable complex which can be quantitatively extracted into n-butanol at pH 7.8. The absorption spectrum of Cu(II) : 3',5'DNSAS complex in n-butanol exhibits an intense absorption peak at 350 nm. The study of changes of colour intensity of copper with varying concentration of reagent showed that 1.0 cm<sup>3</sup> of 0.1% reagent is sufficient for full colour development of extraction of 100 ppm copper solution. The extraction was observed to be quantitative for equilibrium time even less the 60 seconds and absorbance of extracted species showed stability upto 28 hours. Beer's law is obeyed. Stoichiometric ratio of complex studied by job's continuous method, mole ratio and slope ratio method. The composition observed was 1:1. The molar absorptivity and Sandell's sensitivity are also calculated. The newly developed method has been successfully applied for the analysis of copper in synthetic samples and alloys.

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### INTRODUCTION

Copper is very important metal because it has wide applications in industry, pharmaceuticals and in preparation of different type of alloys. It is one of the essential elements for human metabolism. It is generally stored in cell in the form of metalloproteins. It is usually used in organic synthesis for the reduction of nitro compounds. copper has been used for deoxygenating of amine oxides., reduction of aldehydes and the desulfurization<sup>10</sup>, Hence owing to the significance of Copper, it's determination from associated elements by extractive spectrophotometry has been of considerable importance. Several compounds are known to react with the metal ions to give coloured complexes and have been employed for the quantitative extraction and spectrophotometric determination of metals at trace level<sup>7</sup>. Many methods have been reported for the determination of Copper<sup>11</sup>. Therefore, precise knowledge of the Copper present in a various samples is required for an accurate assessment of the Copper, which has resulted in an increasing need of analytical methods for determination of micro-trace or ultra-trace level.

### Experimental

The reagent 3', 5'-Dinitro Salicylaldehyde Semicarbazone was synthesized by the given procedure<sup>9</sup>. The stock solution of Cu(II) was prepared by dissolving a weighed amount of Copper sulphate pentahydrate [CuSO<sub>4</sub>.5H<sub>2</sub>O] in double distilled water containing dilute sulphuric acid and then diluted to desired volume using double distilled water. The Copper solution was standardized by EDTA method<sup>2,4</sup>. 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<sup>8</sup> respectively.

### Procedure for the Extraction

1 mL of aqueous solution containing 0.5µg of copper metal and 1 mL of reagent was mixed in a 50 mL beaker. The pH of the solution adjusted to 7.8, 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<sup>5</sup> 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

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if required. The amount of copper present in the organic phase determined quantitatively by spectrophotometric method by taking absorbance at 350 nm and that in the aqueous phase was determined by EDTA method.

## RESULTS AND DISCUSSION

The results of various studies are discussed below.

### Extraction as a function of pH

The extraction of Cu (II) with (3',5'-DNSAS) was carried out over the pH range of 1 to 10. 1ml of aqueous solution containing 100µg of copper and 1mL of 0.1 % solution of the reagent were used. It reveals that 99.99 % and above of the metal is extracted into organic phase in the pH range 7.2 to 8.0. So the analytical work for the estimation of copper is carried out at pH 7.8. (Fig 1).

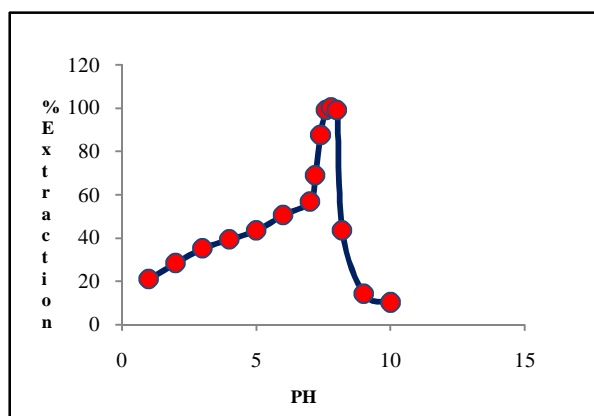


Fig 1 Effect of pH on extraction of Cu (II) : (3',5'-DNSAS) complex

### Absorption spectrum

The absorption spectrum of Cu(II): 3',5'-DNSAS in n-Butanol shows the maximum absorption at 350 nm. The absorption due to reagent at this wavelength is nearly negligible. Hence the absorption measurements were carried out at 350 nm.

### Limit of detection

LOD (Limit of detection) of the present method was calculated at 95 % confidence level by analyzing blank solution with reference to solvent. Standard deviation of blank solution and slope of calibration curve is use for calculating limit of detection, which found to be 0.114 µg / mL.

**Effect of reagent concentration:** The effect of variation in the concentration of (3',5'-DNSAS) in the range of 0.2 to 2.0 mL of 0.1% (3',5'-DNSAS) on the extraction and on colour development was studied. It was observed that 1mL of 0.1 % (3',5'-DNSAS) was sufficient for complete extraction and for colour development. Hence, for subsequent studies 1.0mL of 0.1 % (3',5'-DNSAS) was employed.

### Effect of equilibration time and stability of the complex

The equilibration time of 60s is sufficient for the quantitative extraction of Copper. The stability of colour of the Cu (II): 3',5'-DNSAS complex with respect to time shows that the absorbance due to extracted species is stable up to 28 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 were calculated to be is 2500 L mol<sup>-1</sup>cm<sup>-1</sup> and 0.02542 µg cm<sup>-2</sup> respectively (Fig 2).

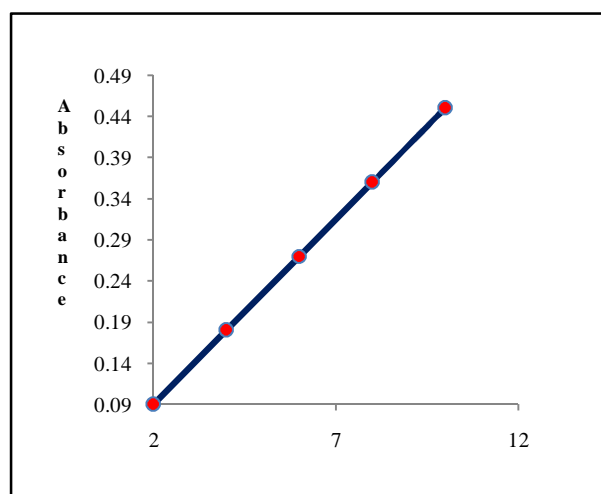


Fig 2 Calibration plot of Cu (II): (3',5'-DNSAS) complex.

### Solvent Study

Various solvents were tried to get the maximum extraction of copper, Fig 3, n-Butanol was found to be the most suitable solvent as it showed the maximum extraction<sup>3</sup>. The extraction of copper varied from maximum to minimum for the solvent in the order of: n-Butanol > Chloroform > Isoamyl alcohol > Ethyl acetate > Toluene > Diethyl ether > Hexane > Carbon tetrachloride > Xylene > Cyclohexanone > Nitro benzene .

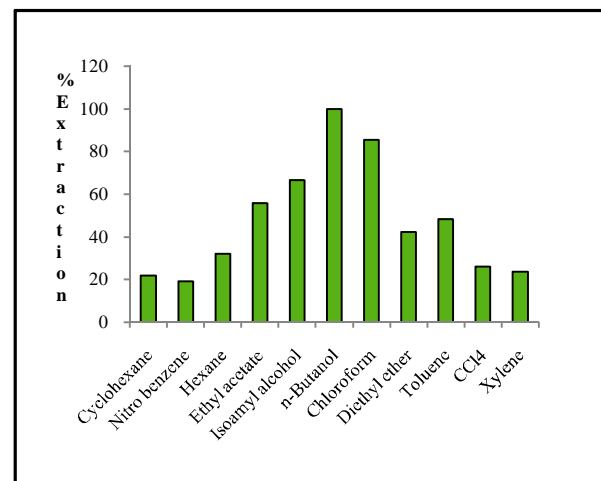


Fig 3 Effect of various solvents on Cu (II): (3',5'-DNSAS) complex

### Precision and accuracy

The precision and accuracy of the spectrophotometric method were tested by analyzing the solution containing known amount of copper (II). Average of ten determination 4.0 µg Cu (II) in 10 mL solution is 3.9312 µg which varies between 3.8001 µg to 4.1001µg at 95% confidence limit.

### Nature of extracted species

The composition of extracted Cu (II): 3',5'DNSAS complex has been determined by Job's continuous variation method, Slope ratio method and Mole ratio method. It shows that the composition of Cu(II): 3',5'DNSAS complex is 1:1 (Fig 4).

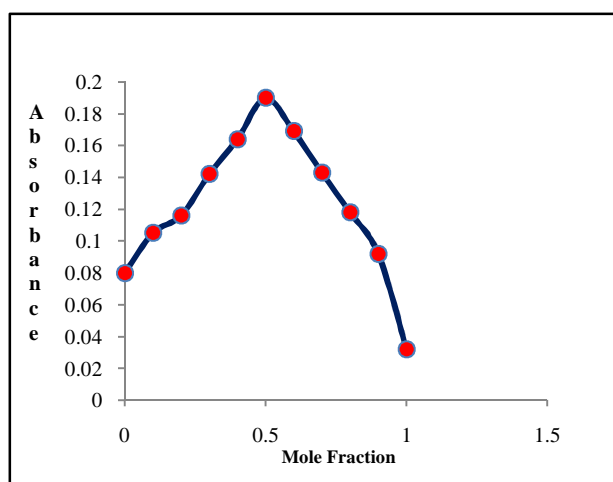


Fig 4 Job's continuous variation curve Nature of extracted species

The proposed method was successfully applied for the determination of copper from various alloys and synthetic samples. The results found to be in good agreement with those obtained by the standard known method. Each result is average of three independent determinations. shown in table-1.

**Table 1** Observation Table For Determination of Cu (II) Using (3',5'-Dnsas) From Different Samples

Sr.No.	Sample	Certified value	Present method
Copper alloys:			
1	Brass	60%	59.8%
Synthetic mixture:			
1	Cu+ Zn	5 ppm	4.8 ppm
2	Cu+ Zn+ Cd	4 ppm	3.8 ppm

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#### References

1. R.S.Lokhande, S.P.Janwadkar, S.M, Pitale, S.K. Patil, S.W.Kulkarni *Ijpbs* (2011).
2. Murthi M.V.R., Khopkar S.M., *Talanta*, 23, 243 (1976).
3. More P.S., Sawant A.D., *Ind. J. Chem.*, 31 A, 984 (1999).
4. Lokhande R.S., Srinivasan N., Chaudhary A.B., *J. of Ind. Council of Chem.*, 19 (1) 12 18 (2002).
5. R.S. Lokhande and A.S.Jayant, *Asian J. Chem.*, 11, 1040 (1999).
6. Vogel, A. I. *Textbook of Quantitative Inorganic Analysis*, 3<sup>rd</sup> edi.; Longman Green and Co. Ltd., London, 1961.
7. Ross, S. D. and Wilson, D.W. *Spectrovision*, 1961, 4, 10.
8. Wilfred, L.; Armarego, E.; Christina, Li, L. C. *Purification of laboratory chemicals*, 5<sup>th</sup> edi.; Butterworth Heinemann, 2003.
9. Ahluwalia, V.K.; Bhagat, P.; Aggarwal, R.; Chandra, R. *Intermediates for organic synthesis*, I.K. International Pvt. Ltd., 2005.
10. Vogel, A. I. *Textbook of Organic synthesis*, 3rd ed.; ELBS, London, 1957.
11. Robinson and shah; *J. Chem. Soc.*, 1934, 1, 491.

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