Spectrophotometric Determination Of Copper (II) With Schiff Base Derived From 3,4,5 Trimethoxy Benzaldehyde And 2-Amino -6-Methyl Pyridine By Preliminary Adsorption On Polyurethane Foam

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Abstract: Spectrophotometric determination of trace amount of Copper (II) after adsorption of its complex with the Schiff base derived from 3,4,5 trimethoxy benzaldehyde and 2-Amino -6-methyl pyridine is described. Copper (II) formed a stable, water insoluble complex with the above Schiff base. This complex was adsorbed on polyurethane foam and was eluted from foam by two portions of 2.5 ml chloroform. The absorbance of the solution measured at 455 nm. Absorbance obeyed Beer's law over the concentration range 10-130 μg of Copper (11) in 5ml chloroform. The molar absorptivity was found to be 1.3243 x 10⁴ mol cm² at 455 nm and sensitivity being 1.78 x 10² μg Cm² of Cu (11) for absorbance of 0.001.

The optimum conditions of the present study were investigated by examining parameters viz. PH, reagent concentration, number of polyurethane foam pieces used shaking time and diverse ions effect of measurements.

Cu (II) causes water pollution and the permissible limit of Cu (II) in drinking water is 1.0 μ g/ml. Metal plating, industrial and domestic wastes, mining and mineral leaching are the chief sources of copper pollution in water. It is highly toxic to algae and lower group of plants6 at moderate levels. Salvinia natans L., an aquatic plant was a good accumulator of Cu (II), it survives in surface water for 24 hours even at concentration of Cu (II) as high as 100μ g ml of the metal.

Mahapatra et al⁸ synthesized Schiff base from furfuraldehyde and 4-amino-2, 3, dimethy1-1-pheny1 pyrazole-5-5- one and characterized its Cu (II) complex.

Shukla et al⁹ synthesized Cu (II) Complex with Schiff bases derived from 2-pyrrolidone and aromatic amines. Similarly, Cu (II) complex with Schiff base of Salicylaldehyde, Aminophenol's and Anthranilic acids was synthesized by Shukla and Singh¹⁰.

Thakar and Thaker "prepared mixed ligand complexes of Cu (II) derived from Salicylaldehyde with N-Substituted ethylenediamine.

Many spectrophotometric reagents have been reported for the micro-determination of Copper (II)¹².

"Solid-Liquid Extraction" method using microcrystalline naphthalene has been used for a long time for adsorption and separation, but we have, used technique of Hamon using polyurethane foam pieces as adsorbent. Cu (II) was determined, by measuring absorbance at 455nm of the complex, which is extracted from foam with chloroform.

Experimental:

A Standard stock solution of Copper (II) 1000ppm was prepared by dissolving requisite amount of hydrated Copper Sulphate in distilled water. Solutions of different concentrations were prepared from this stock solution, after dilution.

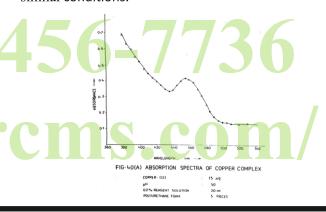
Reagent Solution:

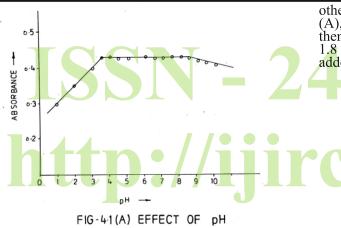
Reagent solution was prepared by dissolving 0.2 gm. Schiff base derived from 3, 4, 5 trimethoxy benzaldehyde and 2-Amino-6-methyl pyridine, in 100 ml ethanol.

Procedure:

To an aliquot (1ml) of standard stock solution containing 2O-110 µg of Copper (I1), in a clean dry beaker 2.0 ml of 0.2% reagent solution was added. The pH of solution was ad adjusted to 5.0 by adding 3.5 ml of acetate buffer solution. The volume of solution vas made 10 ml. All the contents were allowed to stand for 2 minutes for complete development of colour. Now already prepared five foam pieces were added. The flask was stoppered and shaken for sixty seconds to

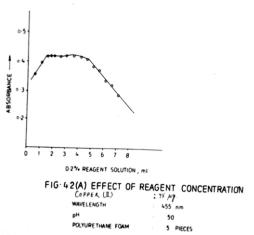
allow the metal complex to be adsorbed on the foam. Polyurethane foam pieces were squeezed by plungers during the shaking time. The complex was eluted from foam by squeezing with two portions of 2.5 ml chloroform. 2.0 gm. anhydrous sodium sulphate was added to remove traces of water. Absorbance was measured in a one cm cell in the wave length region 380-540 nm. Calibration curve was constructed under similar conditions.





other conditions constant. Results are given in Fig. 4.2 (A), Table 4.2 (A). Absorbance Increased up to 1.5 ml then achieved a maximum constant value over range 1.8 to 4.0 ml. Therefore, 2.0 ml of 0.2% reagent was added for all absorbance measurements.

COPPER (11)	:	75 Jug
WAVELENGTH	:	455 nm
02% REAGENT SOLUTION	:	2·0 ml
POLYURETHANE FOAM	:	5 PIECES



Results and Discussion Absorption Spectra:

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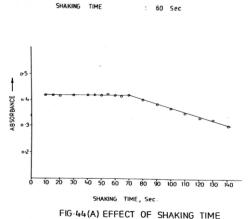
A sample solution of Copper (II) containing 75 µg of Copper (II), 2.0 ml of 0.2% reagent solution and 3.5ml of acetate buffer solution was mixed to adjust the pH of solution at 5.0 The Copper (II) complex formed was adsorbed on prepared five polyurethane foam pieces with frequent squeezing. The complex was then eluted and absorbance was measured in the range 380-540 nm of wave length. Results have been shown in Fig. 4.0 (A). The curve of complex has maxima at 455nm so this wave length was chosen for absorbance measurements.

Effect of pH:

In order to study this effect absorbance of Copper (II) solution containing 75 μg of Cu (II) was measured at 455nm in the pH range 1-10. The results are shown in Fig. 4.1(A), Table 4.1(A). Accordingly the absorbance first increases with pH, attains maximum constant value in the range 3.5 to 9.0. Therefore, pH of solution was adjusted to 5.0 for all1 absorbance measurements.

Effect of Reagent Solution:

The amount of reagent solution varied maintaining the



NO OF POLYURETHANE FOAM USED -FIG-43 (A) EFFECT OF ADSORBENT

ەيد 75

455 nm 50

2-0 m!

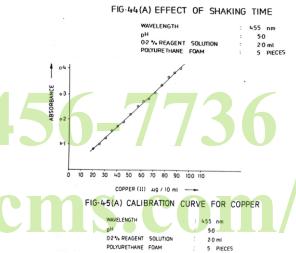
5 PIECES

COPPER (II)

WAVELENGTH

0-2 % REAGENT SOLUTION

POLYURETHANE FOAM



Calibration Curve for Copper (II):

With the optimum conditions described above, the calibration curve for Cu (II) was constructed at wave length 455 nm. The linearity was tested over the concentration range 20-110 µg of Cu (II) present. Results are shown in Fig. 4.5(A), Table 4.5(A). The molar absorptivity was found to be 1.3243 x 10⁴ mol⁻¹ cm⁻¹ at 455 nm and sensitivity being 1.78 x 10⁻² µg cm⁻² of Cu (II) for absorbance of 0.001.

Effect of Diverse ions:

The interference due to various diverse alkali metal salts and diverse metal ions was examined. The possible interference , due to their presence has been shown in Tables 4.6(A) and 4.7(A).

Precision:

Precision of the proposed method was estimated with ten samples of copper (II) complex solutions containing 75 µg of Copper, which provided a mean absorbance of 0.421 which a standard deviation of 0.32%

Table 4.1 (A): Effect of pH

pH	Absorbance at 455 nm
1.0	0.300
2.0	0.350
3.0	0.400
3.5	0.426
4.0	0.426
4.5	0.425
5.0	0.425
5.5	0.427
6.0	0.425
6.5	0.425
7.0	0.426
7.5	0.426
8.0	0.426
8.5	0.424
9.0	0.405
9.5	0.410
10.0	0.405
Cu (II)	: 75ug,
0.03% Reag	gent. 2.0 ml,
Polyurethane I	Foam : 5 Pieces,
	me : 60 Sec.

Table 4.2 (A): Effect of Reagent Concentration.

0.2 % Reagent M.I.	Absorbance at 455 nm
0.5	0.360
1.0	0.390
1.5	0.420
1.8	0.420
2.0	0.420
CM3.0 S. (0.415 0.420
3.5	0.425
4.0	0.420
4.5	0.415
5.0	0.390
5.5	0.375
6.0	0.349
6.5	0.325
7.0	0.290
Cu (II) : 75ug,
рН	0.325 0.290 1): 75ug, H: 5.0 Foam: 5 Pieces,
Polyurethane	Foam : 5 Pieces,
Shaking T	ime : 60 Sec.
	ime : 60 Sec.
ble 4.3 (A) : Effect of Absorbent.	<u> </u>
No. of Polyurethane	Absorbance at 455 nm
Foam Pieces Used	

Table 4.3 (A): Effect of Absorbent.

No. of Polyurethane	Absorbance at 455 nm
Foam Pieces Used	
1	0.421
2	0.420
3	0.420
4	0.422
5	0.430
6	0.430
7	0.425
8	0.420
9	0.398
	0.375
Cu (I	i) : 75ug,
0.2% Rea	1 : 5.0 gent : 2.0 ml, ime : 60 Sec.

	Table 4.6 (A) : Effect of Diverse Alkali Metals.			
Table 4.4 (A): Effect of Shaking Time.		Alkali Metal Salts	Amount Added	Cu (II) Found
Shaking Time (Seconds) Absorbance at 455 nm		KC1	(mg)	(ug)
10	0.424	Kel	100	74.2
15	0.424	K₂SO₄	40	72.3
20	0.415		100	72.0
30	0.420	Na ₂ SO ₄	30	75.8
40	0.422		100	75.2
45	0.422	KNO ₃	50	73.9 74.1
11 C 50	0.422	NaNO ₃	50	76.2
55	0.423		150	75.8
60	0.420	Na ₂ CO ₃	40	76.3
65	0.420		100	74.8
70	0.420	Na₂HPO₄	50	74.1
80	0.400		100	74.8
		Sodium Oxalate	50	74.9
90	0.385		300	74.6
100	0.370		Cu (II) : 75 ug,	
110	0.350	pH:5.0,		
120	0.325		0.2% Reagent : 2.0ml, Polyurethane Foam : 5 Pieces, Shaking Time : 60 Sec.	
130	0.325	P		
140	0.300			
Cu (II) : 75ug,		Table 4.7 (A): Effect of Diverse Metals lons.		
pH:	5.0	Diverse Metal Ions	Amount Added	Cu (II) Found

•		pH:5.0		Diverse metal lons	Alliount Audeu	ca (ii) i caila
	0.2% Reagent : 2.0 ml,			(mg)	(ug)	
		Shaking Time : 60 Seconds Table 4.5 (A) : Calibration Data for Cu (II)		Co (II)	50	74.9
(•				100	74.3
2	Cu (II) Concentration	Absorbance at 455 nm		Cd (II)	50	75.0
4	(ug)				100	75.3
1	20	0.085		Ca (II)	40	74.2
	25	0.100			100	73.9
	30	0.125		Pb (II)	40	74.4
	35	0.160		Ni (II)	150	73.3
	40	0.175			50	73.9
	45	0.190		,	100	74.1
	50	0.225		Mn (II)	50	73.9
	55	0.260		.v ()	100	73.6
	60	0.275				
	65	0.280		Sn (II)	50	75.0
	70	0.315			100	74.8
	75	0.340		Bi (III)	30	73.8
	80	0.375		5 6	80	73.2
	85	0.365		Fe (III)	40	74.8
	90	0.400			150	74.6
	95	0.435		Cr (III)	50	74.1
REF	ZE	100 0.450 105 0.480 110 0.500 pH:5.0, 0.2% Reagent: 2.0 ml,		Mo (VI)	100	73.9
	103				50	74.2
			irams	200	74.8	
				7 1 1 1	Cu (II) : 75 ug,	
		Polyurethane Foam : 5 Pieces, Shaking Time : 60 Sec.			pH : 5.0, 0.2% Reagent : 2.0ml,	
	Shaking T			0.2% Reagent : 2.0ml, Polyurethane Foam : 5 Pieces,		
					Shaking Time: 60 Sec.	

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