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.

Synthesis and Characterization of Copper (II) Complexes With Thiosemicarbazone Derivaties as Lignads

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INTRODUCTION

It is the prime instinct of scientist to be curious to understand the natural phenomena occurring around him. Most researches are outcome of this curiosity. Coordination compounds such as chlorophyll hemoglobin, vitamin B12 etc. are acting as metalloenzyme. These have metal ions coordinated through nitrogen and oxygen so in order to understand a large amount of work has been appeared in literature on coordination compounds with nitrogen-oxygen donar ligands. Recently sulphur is the third element playing its role in natural phenomena.

RECOVERY OF COPPER

Copper was determined with salicyladoxime. 2n sodium hydroxide was added to the copper solution (100ml.), which contained a known weight of Cu compound, until a slight permanent precipitate was formed. It was dissolved in little dilute acetic acid. Salicyldoxime regent was added in slight excess at room temperature. Precipitated complex was filtered off on a weighed

sintered glass crucible, washed with water until the washing give no colour with ferric chloride, and dried to constant weight at 100-105 C (about one hour). It was weighed as $Cu(C_7H_6O_2N)_2$.

PREPRATION OF O-METHOXYBENZALIDINETHIOSEMICARBAZONE COPER (II)

I gram $CuSo_4.5H_2O$ was dissolved in distilled water and treated with 1.85 gram of the ligand dissolved in acetone.

The whole mass was digested on steam-bath for half an hour. The red-brown precipitate was filtered off and dried in air.

Found : Cu……13.20%

C……45.00%

H……4.02%

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N……..17.70%

Required for $[Cu(C_9H_{10}N_3OS)_2]$:-

Cu…….13.24%

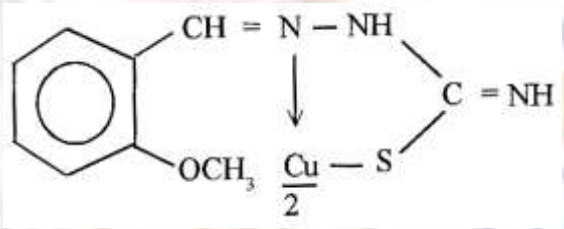
C……45.05%

H……4.17%

N……..17.52%

The complex is insoluble in water but dissolves in acetone, ethanol and methanol. The compound does not loss any weight upto 120C, above this temperature the complex begins to decompose.

The complex is square planar as indicated by its μβ which is equal to 1.73[27] B.M. The i.r. spectra[26] of the complex indicate the following structure of the complex (Fig.1)



(Fig.1)

ESTIMATION OF CU(II) WITH O-METHOXYBENZALIDINETHIOSEMICRABAZONE The reagent was prepared as in the case of Ni(II) estimation. The solutions of Cu(II) with varying concentration were used for the Iodometric determination of the metal.

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results obtained in this case are tabulated below:-

Volume of Cu(II) solution (ml.)	Concentration of Cu(II) in 100ml. of the solution (gm.)	Expected weight of precipitate (gm.)	Experimental weight of precipitate (gm.)
100	0.001	0.0059	0.0058
100	0.002	0.0118	0.0117
100	0.003	0.0177	0.0175
100	0.004	0.0236	0.0235
100	0.005	0.0295	0.0295
100	0.006	0.0354	0.0355
100	0.007	0.0413	0.0413
100	0.008	0.0472	0.0472
100	0.009	0.0531	0.0532
100	0.01	0.0590	0.0590

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Volume of Cu(II) solution (ml.)	Concentration of Cu(II) per 100ml. of the solution (gm.)	Expected volume of $Na_2S_2O_3$ (N/100) solution	Experimental volume of $Na_2S_2O_3$ (N/100) solution
100	0.003	4.38	4.40
100	0.004	5.84	5.95
100	0.005	7.30	7.40
100	0.006	8.76	8.80
100	0.007	10.22	10.25
100	0.008	11.68	11.70
100	0.009	13.14	13.10
100	0.01	14.60	14.60

Burettes having the graduation of 0.1 ml. were used. The end points were determined by using starch solution.

The results can be compared with the results obtained in the gravimetric estimation of Cu(II) with the new reagents. The results show that these new reagents have all the properties of quantitative reagents. The precipitates are quite stable at normal temperature. They can be very easily precipitated and dried to constant weight.

We have also determined Cu (II) gravimetrically with α -benzoionoxime (cupron) reagent using the same Cu (II) solution. The

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100 ml. of Cu(II) solutions containing 0.001 gram copper to 0.01 gram Cu(II) were used. The solution was slightly acidified with dilute hydrochloric acid and warmed on the steam-bath. The solution was then treated with the

Concentration (100ml.)	Experimental weight of the complex	Expected weight on the basis of the formula $Cu(C_9H_{10}N_3OS)_2$
0.001	0.0075	0.0076
0.001	0.0074	
0.001	0.0073	
0.002	0.0150	0.0152
0.002	0.0151	
0.002	0.0149	
0.003	0.0226	0.0228
0.003	0.0225	
0.003	0.0225	
0.004	0.0302	0.0304
0.004	0.0303	
0.004	0.0303	
0.005	0.0381	0.0380
0.005	0.0381	
0.005	0.0381	
0.006	0.0457	0.0456
0.006	0.0457	
0.006	0.0456	
0.007	0.0532	0.0532
0.007	0.0533	
0.007	0.0532	
0.008	0.0609	0.0608
0.008	0.0609	
0.008	0.0608	
0.009	0.0684	0.0684
0.009	0.0685	
0.009	0.0685	
0.01	0.0760	0.0760
0.01	0.0761	
0.01	0.0761	

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prepared reagent followed by dilute solution. The whole m-ass was dije-sted on steam-bath for about half an hour and then filtered through a weighed sintered glass crucible. The precipitate was washed with hot ter ill from the acid ion and dried in an air oven to a constant weight at 120-130 C. Three such periments were performed for each concentration.

RESULT & DISCUSSION

Result are tabulated below:-

Thus the result obtained with the reagent are not so good in the low concentrations. Besides the reagents cannot be used in neutral medium. There agent is specific for copper only in ammoniacal medium.

On the other hand our new reagents are very susceptible for the precipitation of Cu(II) from the low concentration. The precipitates are very easy to filter and they can be dried at 110-130^{0}C to constant weight.

The precipitate with α – benzoinoxime is green which may be confused with Cu(II) hydroxides. The precipitate obtained with the new regents are brown to black and hence the confusion is eliminated.

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