International Journal of Chemistry Studies
ISSN: 2581-348X; Impact Factor: RJIF 5.44
Received: 18-09-2020; Accepted: 22-10-2020; Published: 25-11-2020
www.chemistryjournal.in
Volume 4; Issue 4; 2020; Page No. 34-36

Synthesis and Characterization of Nicel (Ii) Complexes with Thiosemicarbazone Derivaties as Lignads

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Abstract
It is the prime instinct of scientist to be curious to understand the natural phenomena occurring around him. Most researches are outcome of his 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.

Keywords: Nicel (Ii) Complexes, Thiosemicarbazone, Derivaties, Lignads

Introduction

Recovery of Nickel

Nickel was determined as nickel-dimethyglyoxime complex. A know volume of the standard solution as 0.1% …..0.2% was transferred into a 400ml. beaker provided with clock glass and a grass rod. It was treated with 5ml. of (1:1) dilute hydrochloric acid in order to make the medium acidic and then it was diluted to 200ml. it was heated to 70-80 and slight excess of dimethlglyoxime reagent was added and immediately dilute ammonia solution was added directly to the solution with constant stirring until precipitation takes place. It was allowed to stand on steam-bath for one hour and solution was tested for complete precipitation when the precipitate was settled down. It was allowed to stand for one hour. It was filtered through a weighed sintered glass crucible. The precipitate was washed with cold water until it was free from chloride and it was dried at 110-120 C for hours. It was allowed to cool in desiccators and weighed. The process of drying and weighing was repeated till a constant weight was obtained. Thus the percentage of Ni in Ni salt was calculated.

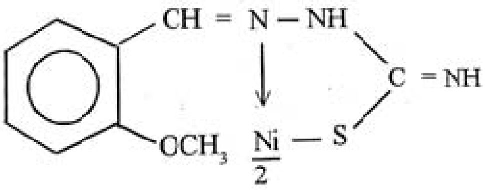
Prepration of O-Methoxybenzalidinethiosemicarbazone Nickel (Ii)

0.1 gram of nickel sulphate (B.D.H.) was dissolved in water and the solution made alkaline with strong ammonia solution. 0.2 gram of the ligand dissolved in acetone under reflux was added to the above solution and refluxed for 1.5 hours on stream-bath. The whole mass was then digested on water-bath for an hours. The brown precipitate was filtered off, dried and analysed.

Found :	Ni……………………..	12.38%
	C……………………..	45.40%
	H……………………..	4.00%
	N……………………..	17.50%
Required for	$[Ni(C_9H_{10}N_3OS)_2]$:-	
	Ni……………………..	12.42%
	C……………………..	45.47%
	H……………………..	4.21%
	N……………………..	17.68%

The complex is insoluble in water, soluble in acetone, ethanol any pyridine. The compound does not lose any weight up to 150c, beyond which the decomposition of the complex begins.

The complex is diamagnetic which shows that it is square planner. The i.r. spectra[26] have shown that the complex has the structure as below.



Estimation of Ni (Ii) With O-Methoxbenzalidinethiosemicarbazone

I gram of the ligan was dissolved in 10ml. of hot actone and cooled. The reagent was used for the estimation.

Procedure

Nickel (II) solutions of different concentration were prepared. In actual practice, we used the concentration ranging from 0.001 gram to 0.01 gram per 100ml. Nickel (II) solution was slightly acidified with dilute hydrochloric acid solution and warmed on steam-bath The solution was then treated with reagent solution (1%) followed with dilute ammonia. The whole mass left for some time to settle the precipitate. Test for completer precipitation was made and the precipitate warmed on steam-bath for half and hour. Filtered through a weighed sintered glasses crucible, washed with hot water till completely free from CI. The precipitate was then heated in an air oven at 120-130 C to a constant weight. Three such experiments were performed for every concentration.

Concentration	Weight of precipitate obtained	Expected weight of precipitate with the formula $Ni(C_9H_{10}N_3OS)_2$
0.001	0.0080	0.0081
0.001	0.0078	
0.001	0.0080	
0.002	0.0161	0.0162
0.002	0.0163	
0.002	0.0161	
0.003	0.0242	0.0243
0.003	0.0242	
0.003	0.0241	
0.004	0.0325	0.324
0.004	0.0323	
0.004	0.0325	
0.005	0.0406	0.0405
0.005	0.0404	
0.005	0.0406	
0.006	0.0486	0.0486
0.006	0.0486	
0.006	0.0486	
0.007	0.0567	0.0567
0.007	0.0568	
0.007	0.0568	
0.008	0.0648	0.0648
0.008	0.0649	
0.008	0.0648	
0.009	0.0729	0.0729
0.009	0.0730	
0.009	0.0730	
0.01	0.0811	0.0810
0.01	0.0811	
0.01	0.0811	

Result & Discussion

The experimental data have been tabulated above. Similar experiments were carried out with the common regents for for Ni (II) for comparisons. The results obtained with these reagents are discussed in this above.

The same solutions of Ni(II) with varying concentrations were taken and 1% solution of diethyl glyoxime was used to precipitate the metal. The result are tabulated below:

Volume of solution (ml.)	Concentration of Ni(II) per 100ml. of the solution in gram.	Weight of Ni (Lmg)2 expected (gm.)	Weight of Ni(Dmg)2 obtained (gm.)
100	0.001	0.0039	0.0039
100	0.002	0.0079	0.0078
100	0.003	0.0118	0.0118
100	0.004	0.0158	0.0159
100	0.005	0.0197	0.0197
100	0.006	0.0237	0.0237
100	0.007	0.276	0.0276
100	0.008	0.316	0.0316
100	0.009	0.355	0.0355
100	0.01	0.0395	0.0395

Thus comparing the results obtained with the new reagents with those obtained with dimethylglyoxime, we are in a position to say that our new reagents are no inferior to dimethylglyoxime so far the quantative preciption is concerned.

Howereve, the new reagents may be considered inferior to dimethylglyoxime in the respect that they cannot be used in the presence of Cu(II). Dimethylglyoxime cab be used for the precipitation of Ni(II) in the presence of Cu(II), provided ammonia in used in excess, but in that case the precipitate itself dissovels to some extent.

Dimethylglyoxime cannot be used as quantitative regent for Ni(II) if Co(II) is present in excess. Our new regents have superiority over this reagent in this respect that Ni(II) can be precipitated even in the presence of Co (II) because Co(II) is not precipitated so readily by these reagents.

Besides, the Ni(II) dimethylgyoxime has the tendency to absord other substances as post- precipitate and the precipitate sticks to the glass hence filtration is sometimes very difficult. In the case of new reagents, such difficulties are not met.

The Ni (II) dimethlglyoxime is deep-red while the precipitate obtained with these regents are brown to red.

The same solutions of Pd(II) with varying concentrations were taken and the metal was precipitated with 1% dimethylglyoxime solution in the strong acid medium. The precipitate was dried between 120-140 ^{0}C to a constant weight. The result obtained are tabulated below:-

Valume of the Pd(II) solution (ml.)	**Concentration Pd(II) per 100 ml. solution (gm.)**	**Expected weight of the precipitate (gm.)**	**Experimental weight of the precipitate (gm.)**
100	0.001	0.0026	0.0027
100	0.002	0.0053	0.0054
100	0.003	0.0079	0.0078
100	0.004	0.0105	0.0105
100	0.005	0.0132	0.0131
100	0.006	0.0158	0.0158
100	0.007	0.0184	0.0184
100	0.008	0.0211	0.0211
100	0.009	0.0237	0.0238
100	0.01	0.0264	0.0264

Conclusion

Thus the result obtained with the dimethylglyoxime and those obtained with the new reagents are comparable.

The experimental details in these cases indicate that while Pd (II) can be quantitatively precipitated with dimethylglyoxime even in strong acid medium, the quantitative precipitation of Pd (II) with the new reagents takes place only in the range of P^H 2-3.

Both the new reagents and the dimethylglyoxime quantitatively precipitate the metal from low concentration and the precipitates are thermally stable.

The Pd (II) dimethylglyoxime is difficult to precipitate due to its adhesive nature. The new regents give precipitates which are very easy to filter throught sintered glass crucible.

Like dimethlyglyoxime, the new reagents cab be used to precipitate Ni (II) and Pd (II) separately in the presence of one another, because the former is quantitatively precipitated in the ammonia cal medium while the latter can be precipitated only in the acidic medium.

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