

## COMPLEXES OF Cu(II), Ni(II) AND Pd(II) WITH 1-HYDROXY-2-ACETONAPHTHONE PHENYLHYDRAZONE\*

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## ABSTRACT

Hydrazones, characterised by the presence of triatomic grouping,  $\text{>C=N-N<}$  have been found to be of biological and analytical importance<sup>1-5</sup>. In continuation of our work on the preparation and structural characterisation of metal complexes of phenylhydrazones of *o*-hydroxyaldehydes and ketones, we incorporate, in this paper, the preparation of Cu(II), Ni(II) and Pd(II) complexes with 1-hydroxy-2-acetonaphthone phenylhydrazone (HANP) and their characterisation based on analytical, spectral, magnetic susceptibility and thermogravimetric data.

## EXPERIMENTAL

THE ketone, 1-hydroxy-2-acetonaphthone was prepared by the method reported in literature<sup>6</sup>.

HANP was prepared by the general procedure of preparing phenylhydrazone. The compound was recrystallised from alcohol. M.P. 138–39°.

The Cu(II) complex was prepared in sodium acetate medium by treating the solution of  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  (one mole) in water with the reagent (two moles) in methanol. The methanol concentration was maintained at 60–70%. Similarly Ni(II) complex was prepared in ammoniacal medium and Pd(II) complex in 0.03–0.04 N HCl medium. The precipitate obtained in each case was digested on water bath, filtered and washed thoroughly with hot water and then with methanol until the washings were free from the reagent. The complexes were then vacuum dried.

The physico-chemical data were obtained as described previously<sup>7</sup>.

## RESULTS AND DISCUSSION

The analytical data indicate that the complexes of HANP can be represented as  $\text{M}(\text{C}_{18}\text{H}_{15}\text{N}_2\text{O})_2$  where  $\text{M} = \text{Cu}, \text{Ni}$  or  $\text{Pd}$  and are brown, yellow and green in color respectively. The complexes are insoluble in water, dilute mineral acids and common organic solvents such as benzene, chloroform, etc.; they are sparingly soluble in DMF and 1,4-dioxan.

Cu(II) complex is found to be paramagnetic with an effective magnetic moment of 1.85 B.M. and Ni(II) and Pd(II) complexes are diamagnetic. The complexes are considered non-electrolytes as their molar conductance values in DMF at the concentration  $10^{-3}$  M are low.

The thermograms show that the complexes start decomposing with the loss of ligand at 200° C. The final products of decomposition of the complexes above 660° C correspond to metallic oxide in each case.

Some important IR and electronic absorption frequencies are given in Table I.

TABLE I

*IR and electronic absorption data*

HANP	Cu-complex	Ni-complex	pd-complex	Assignment
<i>IR (<math>\text{Cm}^{-1}</math>)</i>				
3320	..	..	..	Intramolecularly hydrogen bonded-O-H
3270	3210	3210	3210	$\nu\text{N-H}$
1585	1560	1560	1570	$\nu\text{C=N}$
1240	1235	1235	1235	$\nu\text{C-N}$
1165	1200	1200	1200	$\nu\text{C-O}$
<i>Electronic (nm)</i>				
..	620*	500	500**	* $^2\text{B}_{1g} \rightarrow ^2\text{B}_{2g}$
			670 +	** $^1\text{A}_{1g} \rightarrow ^1\text{B}_{1g}$ + $^1\text{A}_{1g} \rightarrow ^1\text{A}_{2g}$
365	365	365	365	Internal charge-transfer ligand
295	295	295	295	vibrations.

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Based on the foregoing data, square planar geometry has been proposed for the complexes.

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## A NOTE ON MAGNETISATION OF MAGNETITE-ORE BAND AT ADDATIGALA IN ANDHRA PRADESH

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#### ABSTRACT

Remanent magnetisation of seven oriented samples from the magnetite-ore band at Addatigala (Lat.  $17^{\circ} 30' 38''$  N, Long.  $82^{\circ} 3' 20''$  E), East Godavari District, Andhra Pradesh, is studied. The NRM intensities of the samples range from  $1.6 \times 10^{-3}$  to  $8.7 \times 10^{-3}$  e.m.u. The samples are subjected to a.c. demagnetisation. The directions, after demagnetisation, are distributed around two different mean directions. The Declination (D) and Inclination (I) of the two mean directions are  $D = 310^{\circ}$  (E),  $I = +25^{\circ}$  and  $D = 36^{\circ}$  (E),  $I = +9^{\circ}$ . Samples with differing directions are found to be different mineralogically also. In the samples with the first mean direction, the major ore mineral is found to be magnetite, while in those with the second mean direction, magnetite is found to be extensively martitised.

#### INTRODUCTION

**M**AGNETITE occurs in the gabbroic rocks traversing the Eastern Ghat suite of rocks, garnet sillimanite gneisses, quartzites, leptynites, and charnockites (Sriramadas<sup>1</sup>) along the NE-SW shear zones in the margins of the charnockite belts (Karunakaran<sup>2</sup> and Narayanaswamy<sup>3</sup>). The magnetite ore at Addatigala is intimately associated with a sequence of ultramafic rocks comprising variants of pyroxenite, hornblende and plagioclase. It occurs as layers and lenses. The country rocks comprise garnetiferous granite gneisses, garnet-feldspar-sillimanite gneisses, reconstituted quartzfeldspar gneisses and schists, feldspar-sillimanite gneisses, charnockites and pink granites. The ore band trends at  $N 60^{\circ} E$  with a dip of  $60^{\circ} SE$ . The area is surveyed with a magnetometer for delineating the ore band and finding its extent and dimensions (Lakshmipati Raju<sup>4</sup>). Measurements to estimate the Natural Remanent Magnetisation (NRM) and magnetic susceptibility are made on collected samples of the ore. The results of NRM measurements and subsequent demagnetisation studies are reported here.

#### ORIGIN OF THE ORE

The occurrence of different ultramafic bodies and their associated rocks in the southern Peninsular India along major planes of weakness suggests that they may represent mantle material injected into the crust. According to Narayanaswamy<sup>3</sup> the ultramafics (including the magnetite concentrations) along NE-SW shear zones in the Eastern Ghats probably represent late tectonic intrusives during the Eastern Ghat Orogeny. According to Karunakaran<sup>2</sup> they are emplaced into the Eastern Ghats prior to the uplift and are older than the enclosing charnockites.

From a study of the field relations and chemical analysis of the ore, Sriramadas<sup>1</sup> deduced that the magnetite ore may be taken as connected with mafic magmas and magmatic looking rocks like gabbros. Minor and trace assemblages of the ore also suggest (Narasayya<sup>5</sup>) an original magmatic origin. The metamorphic episode, after the culmination of Eastern Ghats Orogeny, was placed at 500 Myr (Aswathanarayana<sup>6</sup> and Sarkar<sup>7</sup>).