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### SYNTHESIS AND STUDIES OF SOME FIRST-ROW TRANSITION METAL COMPLEXES WITH SCHIFF BASE DERIVED FROM 2-HYDROXY-1-NAPHTHALDEHYDE AND *m*-ANISIDINE

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RECENT studies have thrown light on the synthesis of first-row transition metal complexes<sup>1-3</sup> of some new Schiff bases. However, no work seems to have been done on the metal complexes of Schiff bases derived from 2-hydroxy-1-naphthaldehyde and anisidines. This work is aimed at studying some metal complexes of Schiff bases from the point of their structural characteristics and deals with the isolation of Cu(II), Ni(II), Co(II), Zn(II), Mn(II) and Cr(III) complexes with the bidentate Schiff base derived from 2-hydroxy-1-naphthaldehyde and *m*-anisidine.

**Preparation of the Schiff base:** It was synthesised<sup>4</sup> by mixing equimolar amounts of 2-hydroxy-1-naphthaldehyde (3.44 g *i.e.*, 0.02 mol) and freshly distilled *m*-anisidine (2.2 ml *i.e.*, 0.02 mol) in absolute ethanol and refluxing the mixture on water bath in the presence of piperidine (2-3 drops) as the condensing agent for 2 hr. On ice-cooling shining orange-coloured Schiff base was crystallized out. It was filtered, washed, dried and recrystallized from ethanol solution. The Schiff base was analyzed and characterized by its elemental analysis (table 1) and IR spectral studies. On the basis of these studies, the structure as shown in figure 1 is proposed.

**Preparation of the metal complexes:** Ethanolic solution of the Schiff base and the metallic chlorides were mixed in 1:2 molar ratio and the mixture refluxed on water

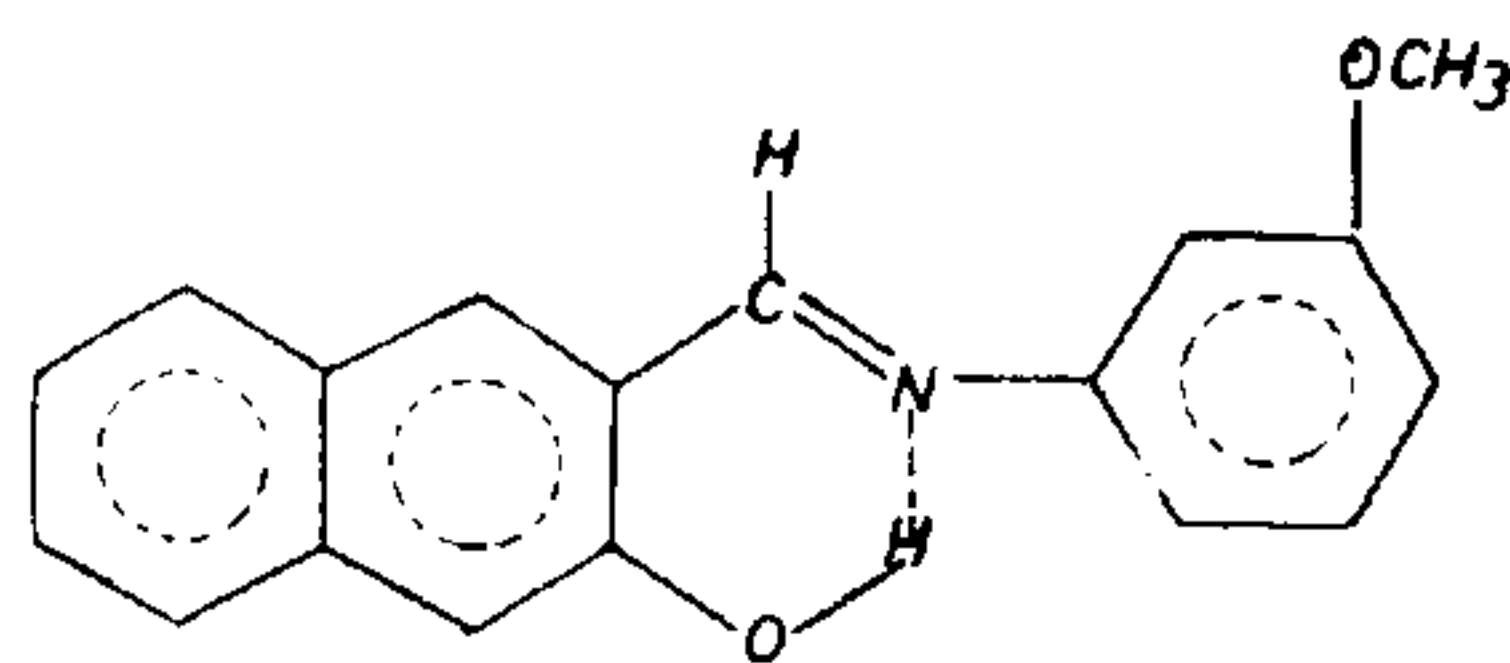


Figure 1.

bath for 2-3 hr. The reaction mixture was concentrated and then cooled. On adjusting the pH of the medium between 4 and 5 in the case Cu(II) derivative and about 8 in the other cases, solid derivatives were separated out by adding dilute ammonia solution. These were filtered, washed and finally air-dried.

The prepared metal derivatives were analyzed for metal, C, H, N and chlorine. C and H were analyzed by Leibigs method and nitrogen by Dumas's method.

Chlorine was estimated as AgCl gravimetrically<sup>5</sup>. For the assay of chlorine, the weighed amount of metal derivative was fused with Na<sub>2</sub>CO<sub>3</sub> and the melt, cooled and extracted with boiling distilled water, and filtered. The extract was acidified with concentrated HNO<sub>3</sub> and chlorine precipitated as AgCl. The magnetic susceptibilities were measured on a Gouy magnetic susceptibility balance by employing Gouy's method at room temperature. Diamagnetic corrections were made with Pascal's Constants<sup>6</sup>.

The analytical and magnetic moment data of the complexes are given in table 1. The experimental values of the analyses for C, H and chlorine in the metal derivatives, agreed with the calculated values within the limits of experimental error.

**Infrared spectra:** The site of coordination of the Schiff base was inferred from IR studies. The Schiff base acts as a bidentate ligand, with the hydroxyl oxygen and the imino nitrogen atom as the principal bonding sites. The assignments of the IR bands of ligand and complexes, are based on earlier studies<sup>7,8</sup>. The ligand shows a band at 2800 cm<sup>-1</sup> which is assigned to ν<sub>OH</sub> involved in intramolecular H-bonding<sup>9</sup> with basic azomethine nitrogen. This band is absent in the complexes indicating the breaking of H-bonding leading to deprotonation during coordination to the metal ion resulting in the formation of the O-M bond<sup>10</sup>. Further, the spectrum of the Schiff base shows a strong sharp band at 1610 cm<sup>-1</sup> assigned to ν(C=N), which in the complexes was shifted towards the lower

Table 1 Analytical and magnetic moment data

Compound (Mol. wt.)	Colour	M.P. °(C)	Metal	Analysis % Found			Cl	u eff. B.M.
				C	H	N		
Schiff base (hydrox-MA) (277)	Orange	108	—	77.81	5.32	5.09	—	—
[Cu (hydrox-MA) <sub>2</sub> ] (615.54)	Chocolate brown	222	10.46	70.38	4.67	4.72	—	1.78
[Ni (hydrox-MA) <sub>2</sub> · 2H <sub>2</sub> O] (646.71)	Apple- green	231	9.18	66.88	4.98	4.51	—	3.00
[Co (hydrox-MA) <sub>2</sub> · 2H <sub>2</sub> O] (646.94)	Pink	140	9.36	66.92	4.96	4.64	—	4.87
[Zn (hydrox-MA) <sub>2</sub> ] (617.38)	Light- yellow	218	10.64	69.86	4.72	4.88	—	—
[Mn (hydrox-MA) <sub>2</sub> · 2H <sub>2</sub> O] (642.94)	Yellow	190	8.66	67.36	5.02	4.62	—	5.96
[Cr (hydrox-MA) <sub>2</sub> · Cl · H <sub>2</sub> O] (657.51)	Dark- green	209	7.98	65.86	4.90	4.36	5.12	3.82

hydrox = 2-hydroxy-1-naphthaldehyde; MA = m-anisidine

frequency region<sup>11,12</sup> indicating the involvement of azomethine nitrogen during coordination to the metal ion.

A broad trough near 3400–3000 cm<sup>-1</sup> in all complexes except those of Cu(II) and Zn(II) indicates the presence of coordinated water molecules<sup>13,14</sup>. The peaks in the region 580–250 cm<sup>-1</sup> are assigned to  $\nu_{M-O}$ ,  $\nu_{M-N}$  and  $\nu_{M-Cl}$  vibrations<sup>15</sup>.

**Magnetic susceptibilities:** All the complexes were paramagnetic except Zn(II) complex which was diamagnetic. The magnetic moment data given in (table 1) suggest high spin octahedral geometry<sup>16</sup> for Ni(II), Co(II), Mn(II) and Cr(III) complexes. The magnetic moment of Cu(II) complex was normal<sup>17</sup> corresponding to one unpaired electron giving no specific information about stereo-chemistry.

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