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Syntheses, characterization and x-ray crystal structures of copper(II) and nickel(II) complexes of tridentate monocondensed diamines

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SYNTHESES, CHARACTERIZATION AND X-RAY CRYSTAL STRUCTURES OF COPPER(II) AND NICKEL(II) COMPLEXES OF TRIDENTATE MONOCONDENSED DIAMINES

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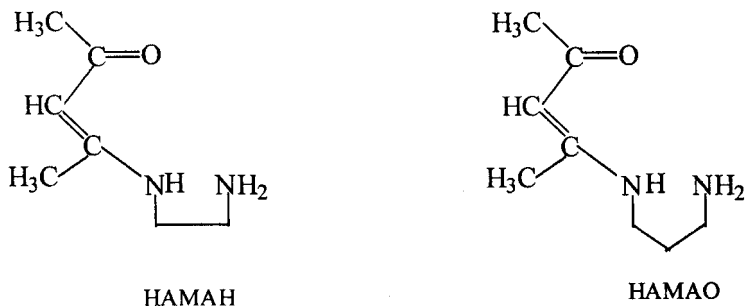
The syntheses and characterization of three compounds involving tridentate “half-units” 7-amino-4-methyl-5-aza-3-hepten-2-one (HAMA) and 8-amino-4-methyl-5-aza-3-octen-2-one (HAMA) are described. Cu(II) and Ni(II) complexes with HAMA have been isolated as four-coordinate complexes, the fourth coordination site being taken by imidazole, and have been structurally characterized. A Cu(II) complex involving HAMA has been isolated as a highly insoluble polymeric species. Hydroxo bridging between the metal centres is indicated.

Keywords: NO Donor; Tridentate; Schiff base; Cu(II); Ni(II); X-ray structure

INTRODUCTION

In our continued interest [1] with unsymmetrical ligands and their metal complexes, we describe the synthesis and characterization of complexes having tridentate “half-units”. The tridentate units can be used as precursors for synthesis of unsymmetrical Schiff bases involving the free amino group [2]. Such singly condensed “half-units” have been prepared by several routes [2–5]. Reaction of constituents under high dilution was employed in the present study as in our earlier work [1]. The units (Scheme 1) chosen for this study are 7-amino-4-methyl-5-aza-3-hepten-2-one (HAMA) and 8-amino-4-methyl-5-aza-3-octen-2-one (HAMA), resulting from condensation of one molecule of acetylacetone with either 1,2-ethanediamine (HAMA) or 1,3-propanediamine (HAMA).

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SCHEME 1

The complexes (imidazole)(7-amino-4-methyl-5-aza-3-hepten-2-onato)copper(II) perchlorate [(AMAH)CuImH]ClO₄, (**1**), and (imidazole)(7-amino-4-methyl-5-aza-3-hepten-2-onato)nickel(II) perchlorate [(AMAH)NiImH]ClO₄, (**2**), have been isolated, where imidazole occupies the fourth coordination site. These two compounds have been structurally characterized. Attempts to isolate a similar imidazole complex with HMAO resulted in the separation of a polymeric copper(II) complex with extensive hydroxo bridging, (μ_2 -hydroxo)(8-amino-5-aza-4-methyl-3-octene-2-onato)copper(II), [(CuL)(OH)]_n, (**3**). Owing to its highly insoluble nature, no single crystals suitable for X-ray diffraction studies could be produced.

In a previous paper [1], we have reported the synthesis and characterization of Cu(II) complexes derived from the “half-units” by condensation of the free amino group with pyridine-2-carboxaldehyde giving an unsymmetrical tetradentate ligand. We involved the precursor “half-units” as ligands in this study in order to bind imidazole to the metal centre at its vacant coordination site, because binding of imidazole-type molecules to metal complexes are of interest with respect to biologically important metal complexes [2]. Complex (**1**) has been isolated by Costes *et al.* [6], as a monomer to subsequently synthesize hetero- or homo-binuclear imidazolato-bridged species. No detailed analysis or crystal structure has been reported. It was also our aim to investigate any structural changes around the central metal ion for this complex with respect to the bridged complexes reported. We have determined the X-ray crystal structure of this compound with this aim in mind. The other two complexes, (**2**) and (**3**), however, are hitherto unknown compounds.

EXPERIMENTAL

Synthesis of Ligands and Complexes

The “half-units” HAMA and HMAO were prepared as viscous oils by reported methods [1].

[(AMAH)CuImH] (**1**)

To a solution of HAMA (5 mmol) in 20 mL of methanol, Cu(ClO₄)₂ · 6H₂O (5 mmol, 1.75 g) in 10 mL of methanol was added. Then a methanol solution (5 mL) of imidazole (5 mmol, 0.034 g) was added followed by triethylamine (5 mmol, 0.7 mL). With stirring,

the blue solution gave a plum-coloured precipitate that was filtered and the filtrate was kept at room temperature. Overnight, brown-red needle-shaped single crystals suitable for X-ray diffraction were obtained (yield 80%). Anal. Calcd. for $C_{10}H_{17}CuClN_4O_5$ (%): C, 32.23; H, 4.57; N, 15.04; Cu, 17.05. Found: C, 32.61; H, 4.45; N, 17.13; Cu, 17.83; λ_{\max}/nm ($\epsilon_{\max}/M^{-1}cm^{-1}$) (acetonitrile), 539 (255), 367 (1639); $\mu_{\beta} = 1.77$.

[(AMAH)NiImH] (2)

Complex (2) was prepared in a similar way to (1) except that $Ni(ClO_4) \cdot 6H_2O$ was added instead of $Cu(ClO_4) \cdot 6H_2O$ and a dark orange coloured precipitate was immediately obtained. Needle-shaped single crystals suitable for X-ray diffraction were obtained (yield 80%). Anal. Calcd. for $C_{10}H_{17}NiClN_4O_5$ (%): C, 32.66; H, 4.62; N, 15.24; Ni, 15.98. Found: C, 32.83; H, 4.79; N, 15.03; Ni, 16.03; λ_{\max}/nm ($\epsilon_{\max}/M cm^{-1}$) (acetonitrile), 539 (255), 367 (1639); diamag.

[(AMAO)CuOH]_n (3)

To a solution of HMAAO (5 mmol) in 20 mL of methanol, $Cu(ClO_4)_2 \cdot 6H_2O$ (5 mmol, 1.75 g) in 10 mL of methanol was added. Then a methanol solution (5 mL) of imidazole (5 mmol, 0.034 g) was added followed by triethylamine (5 mmol, 0.7 mL). With stirring, the green solution gave a violet precipitate that was filtered and washed with methanol. Owing to its highly insoluble nature, no single crystals suitable for X-ray diffraction studies were produced (yield 92%). Anal. Calcd. for $(C_8H_{16}CuN_2O_4)_n$ (%): C, 40.8; H, 6.8; N, 11.9; Cu, 26.9. Found: C, 41.1; H, 6.5; N, 12.1; Cu, 26.01; λ_{\max}/nm (nujol), 853, 531; $\mu_{\beta} = 1.61$.

Physical Measurements

Elemental analyses (carbon, hydrogen and nitrogen) were performed using a Perkin-Elmer 240C instrument and copper contents in all complexes were estimated spectrophotometrically [7]. IR spectra in KBr (4500–500 cm^{-1}) were recorded using a Perkin-Elmer RXI FT-IR spectrophotometer. Electronic spectra in acetonitrile (800–200 nm) and as nujol mulls (1200–200 nm) were recorded on a Hitachi U-3501 spectrophotometer. Effective magnetic moments were calculated from magnetic susceptibility measurements with an EG & G vibrating sample magnetometer (model 155) at room temperature and diamagnetic corrections were made using Pascal's constants.

Crystal Structure Determination of [(AMAH)CuImH] (1) and [(AMAH)NiImH] (2)

Some 2310 and 2112 independent reflections for (1) and (2), respectively, were measured with $Mo K\alpha$ radiation using the MARresearch Image Plate System. Crystals were positioned 70 mm from the image plate. Some 100 frames were measured at 2° intervals with a counting time of 2 min. Data analysis was carried out with the XDS program [8]. The structures are isomorphous and were solved using direct methods with SHELX86 program [9]. Non-hydrogen atoms were refined with anisotropic thermal parameters. The hydrogen atoms bonded to carbon were included at geometrically appropriate

TABLE I Summary of crystal data, data collection and structural refinement parameters for (1) and (2)

	[CuL(ImH)](ClO ₄) (1)	[NiL(ImH)](ClO ₄) (2)
Formula	C ₁₀ H ₁₇ CuClN ₄ O ₅	C ₁₀ H ₁₇ NiClN ₄ O ₅
<i>M</i>	372.28	367.42
Crystal system	Triclinic	Triclinic
Space group	<i>P</i> 1̄	<i>P</i> 1̄
<i>a</i> /Å	7.522(9)	7.570(2)
<i>b</i> /Å	10.376(14)	10.262(14)
<i>c</i> /Å	10.578(14)	10.446(14)
α /°	107.991(10)	107.268(10)
β /°	101.606(10)	101.949(10)
γ /°	99.274(10)	100.681(10)
<i>V</i> /Å ³	746.8(17)	731.2(18)
<i>Z</i>	2	2
<i>D_c</i> /g cm ⁻³	1.656	1.669
<i>F</i> (000)	382	380
μ /mm ⁻¹ [Mo <i>K</i> α]	1.7	1.5
No. of unique data	2310	2112
No. of data with <i>I</i> > 2 σ (<i>I</i>)	1881	1026
<i>R</i> 1, <i>wR</i> 2	0.0880, 0.2301	0.0724, 0.1449

TABLE II Selected bond lengths (Å) and angles (°) for Complexes (1) and (2)

	(1)	(2)
M(1)–N(21)	2.005(6)	1.980(8)
M(1)–O(11)	1.922(5)	1.815(7)
M(1)–N(15)	1.953(6)	1.841(8)
M(1)–N(18)	2.019(6)	2.013(8)
O(11)–M(1)–N(18)	173.3(2)	174.7(3)
O(11)–M(1)–N(21)	87.9(2)	87.6(3)
N(15)–M(1)–N(18)	84.4(2)	88.9(3)
N(15)–M(1)–N(21)	174.4(2)	174.1(3)
N(18)–M(1)–N(21)	94.7(2)	88.0(3)
O(11)–M(1)–N(15)	93.6(2)	95.8(3)

M = Cu for complex (1) and Ni for complex (2).

positions and given thermal parameters equivalent to 1.2 times those of the atom to which they were attached. Absorption corrections were carried out using the DIFABS program [10]. The structures were refined on F^2 using SHELXL [11] to $R1 = 0.0880$, $wR2 = 0.2309$; $R1 = 0.0724$, $wR2 = 0.1449$, for 1881 and 1026 reflections with $I > 2\sigma(I)$, respectively. Relevant crystal data are summarized in Table I. Selected bond lengths and angles are listed in Table II.

RESULTS AND DISCUSSION

ORTEP diagrams of Complex (1) together with the (common) atom numbering scheme is shown in Fig. 1. In both (1) and (2) the deprotonated ligand L is tridentate, forming one six-membered and one five-membered chelate ring. Both the structures consist of discrete mononuclear cations and perchlorate anions.

The metal atoms have square-planar coordination with a slight tetrahedral distortion in both cases, the larger deviation being associated with copper, as found also in related

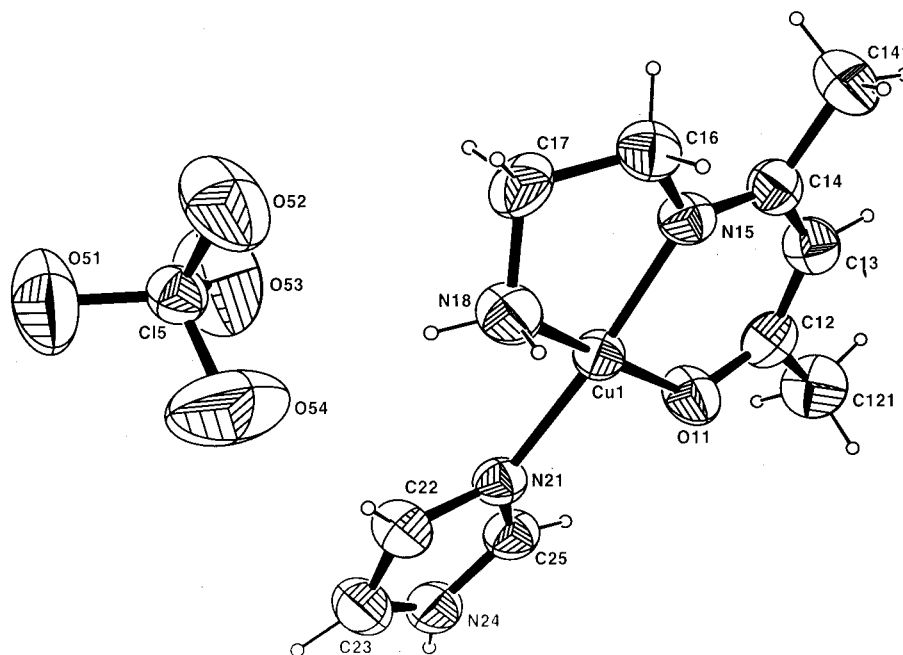


FIGURE 1 Ortep view of Complex (1) with the atom labelling scheme. Thermal ellipsoids are shown at 50% probability.

systems [6]. Deviations of the coordination atoms O(11), N(15), N(18), N(21) from the least-squares mean plane through them are $-0.102(6)$, $0.098(5)$, $-0.130(6)$, $0.075(5)$ Å, in Complex (1) and $-0.052(5)$, $0.066(6)$, $-0.072(7)$, $0.062(6)$ Å in Complex (2) respectively. Deviations of copper(II) and nickel(II) from the same planes are $-0.008(1)$ Å and $-0.018(1)$ Å, respectively. Bond distances in both (1) and (2) are within the range of values normally found in these types of complexes [6,12]. It is interesting to note that the two bonds M–O(11) and M–N(15) are shortened by *ca.* 0.1 Å in changing from Cu to Ni while the other two bonds remain relatively the same. Comparison with related systems shows that almost in all complexes the bonds are shorter when M = Ni than when M = Cu, but the difference is much less than 0.1 Å.

The acetylacetonate ring around the metal ion is essentially planar with no atom deviating from the least-squares plane by more than 0.042 Å and 0.044 Å in Complexes (1) and (2), respectively. In both complexes, the five-membered chelate ring, M(1)–N(15)–C(17)–N(18) has an envelope conformation with puckering parameters [13] $q_2 = 0.399(7)$ and $\phi_2 = 80.6(8)^\circ$ in Complex (1) and $q_2 = 0.299(7)$ and $\phi_2 = 75.2(1)^\circ$ in Complex (2).

Both crystal structures exhibit identical H-bonding patterns (Table III). Amino nitrogen N(18) is involved in an N–H \cdots O hydrogen bond with one of the perchlorate oxygen atoms O(53). Imidazole nitrogen N(24) is involved in a pair of bifurcated N–H \cdots O hydrogen bonds with the same oxygen, O(51), of two symmetry-related perchlorate groups. An interesting feature of the structures is that one of the imidazole carbon atoms, C(25), has a C–H \cdots O contact (Table III) with the coordinating oxygen atom O(11). This probably inhibits free rotation of the imidazole moiety about the Cu(1)–N(21) coordination bond.

TABLE III Hydrogen bond distances (Å) and angles (°) for Complexes (1) and (2)

$D-H\cdots A$	$D-H$	$D\cdots A$	$H\cdots A$	$\angle D-H\cdots A$
Complex (1)				
N(18)–H(18A)···O(53)	0.901	3.284(11)	2.4678	150.92
N(24)–H(24)···O(51)	0.859	3.125(10)	2.4378	137.34
N(24)–H(24)···O(53) ^a	0.859	3.107(9)	2.3708	143.84
C(25)–H(25)···O(11)	0.929	2.824(8)	2.373	109.58
Complex (2)				
N(18)–H(18A)···O(53)	0.901	3.085(13)	2.267	150.86
N(24)–H(24)···O(51)	0.860	3.105(13)	2.378	142.45
N(24)–H(24)···O(53) ^a	0.860	3.205(15)	2.529	136.10
C(25)–H(25)···O(11)	0.930	2.706(14)	2.263	108.48

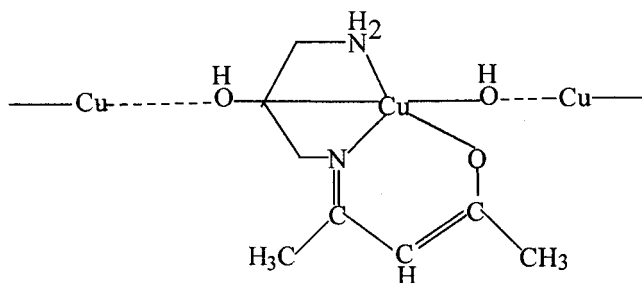
^aSymmetry equivalent: $x, 1+y, z$.

A structural comparison of these two complexes with that of their imidazolate-bridged bimetallic homologue [6] reveals that all four bonds around copper(II) and two bonds (imidazolate and primary amine N) around nickel(II) are shortened by 0.04–0.06 Å in the latter, while the two bonds around nickel(II) which are the part of the acetylacetonate ring are lengthened by 0.04 and 0.051 Å, respectively. This is probably associated with the greater deviations (up to 0.1 Å) of the atoms of the ring from the least-squares mean plane in the bimetallic complex. It is also interesting to note that tetrahedral distortions around copper(II) remain almost the same in the monomeric and dimeric complexes but are higher around nickel(II) in the former. The distortion may conveniently be measured by the trans angles [14] which are ideally 180° for a square-planar complex and 109.5° in a tetrahedral complex. The angles are found to be *ca.* 174° around copper(II) in both complexes whereas the two trans angles around nickel(II) are 176.5° and 178° in the dimeric and 174.7° and 174.1° in the monomeric complexes.

The preparation of tridentate “half-units” has been carried out by the condensation of the reagents in dilute conditions in a 1 : 1 molar ratio. The isolation of (1) and (2) was successful only after addition of a base, triethylamine. Under similar conditions, hydroxo-bridged species separated out for AMAO. The first species to separate out was the trinuclear μ_3 -hydroxo complex when triethylamine, ligand and copper perchlorate were reacted in 1 : 1 : 1 molar proportions. The trinuclear complex was structurally characterized and found to be same as that reported by Kwiatkowski *et al.* [4]. However, when the concentration triethylamine was doubled, the polymeric hydroxo-bridged Complex (3) was isolated. Since the first isolated species is a hydroxo-bridged trinuclear example, in all probability the polymeric compound isolated in the second attempt is of the same nature. The IR spectrum and DTA-TG studies substantiated that it was a hydroxo-bridged polymer. No corresponding Ni(II) complexes could be isolated with the ligand HAMAQ.

Spectroscopic Magnetic and Thermal Studies

The IR spectra of Compounds (1) and (2) show distinct bands at 3339 and 3305 cm^{-1} , respectively attributed to ν_{NH_2} in the “half-unit” tridentate ligand. Bands at 3157 and 3155 cm^{-1} , respectively, may be due to $\nu_{\text{N-H}}$ of the imidazole moiety. The $\delta_{\text{N-H}}$ mode is at 1518 and 1514 cm^{-1} for (1) and (2), respectively. The bands corresponding to



SCHEME 2

$\nu_{\text{C}=\text{N}}$ at 1591 and 1590 cm^{-1} indicate their coordination to metal centres. Compound (1) shows a sharp peak at 1106 cm^{-1} due to ionic perchlorate and the corresponding peak for (2) is observed at 1100 cm^{-1} .

A broad band centered at 3433 cm^{-1} for Compound (3) gives an indication of $\nu_{\text{O}-\text{H}}$ merged with amine modes; $\nu_{\text{C}=\text{N}}$ is observed at 1594 cm^{-1} , suggesting the metal ion is bonded to this group. No peak corresponding to ionic perchlorate is observed. Thermogravimetric analysis of Compound (3) does not show any mass loss up to 220°C, after which it starts to decompose. This result suggests the absence of any water in the complex. Therefore, the appearance of $\nu_{\text{O}-\text{H}}$ should be due to the presence of OH^- . The absence of perchlorate and microanalyses also suggest the presence of OH^- , which usually acts as a μ_2 -bridge. Thus Compound (3) may be considered as being a hydroxo-bridged, one-dimensional, polymeric compound as shown in Scheme 2. Its insolubility in common solvents is consistent with a polymeric structure. Room-temperature magnetic moments of (1) and (3) correspond to one electron (1.7 BM) and Compound (2) is diamagnetic as expected.

Electronic spectroscopic data in acetonitrile solutions suggest a square-planar environment for Compound (1) [1,15] as substantiated by the crystal structure. A band at 18761 and a shoulder at 15094 cm^{-1} are observed. Other higher energy bands are charge-transfer in origin. Electronic spectra of Complex (2) show a single band with λ_{max} at 451 nm in accord with square-planar geometry around Ni(II). Spectra of Compound (3) (nujol mull) show a broad band with λ_{max} at 19493 cm^{-1} in conformity with the proposed structure.

The X-band epr spectrum of (3) in the solid state at 77°K clearly shows $g^\perp > g_1$. The pattern is indicative of an approximate $\{d_z^2\}^1$ ground state [16]. The tridentate ligand (AMAO) together with the μ_2 -OH bridge should form a pentacoordinate environment around copper (II). Of the two probable structures, square pyramidal and pentagonal bipyramidal, the latter possess a $\{d_z^2\}^1$ ground state and therefore it can be assumed that the most likely molecular geometry around copper in (3) is trigonal bipyramidal.

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Supplementary Data

Complete listings of coordinates, bond lengths, bond angles and anisotropic thermal parameters have been deposited at CCDC, with codes 181604 (Complex 1) and 181605 (Complex 2) and also are available from the authors upon request.

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