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NOTE

Synthesis and Crystal Structure of Binuclear Ni(II) Complex Derived from 2-Nitrobenzoic Acid and Tetramethylethylenediamine

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A nickel(II) complex of the formula $[Ni_2(nba)_4(tmen)_2(H_2O)]\cdot C_2H_5OH$ (**1**), where nba = 2-nitrobenzoate, tmen = tetramethylethylenediamine, has been synthesized and determined by single crystal X-ray diffraction method. The crystal belongs to monoclinic system, space group P121/c1 with cell dimensions of a = 11.3931 (4), b = 33.0702 (4), c = 13.5623 (4) Å, α = 90, β = 111.310 (4) and γ = 90°. The two nickel atoms are connected by one bridging water molecule and two bidentate bridging carboxylate ligands. The octahedral environment around each nickel atom is completed by two nitrogen atoms from tetramethylethylenediamine and one oxygen atom from monodentate carboxylate.

Key Words: 2-Nitrobenzoic acid, Nickel, Crystal, Tetramethylethylenediamine.

The versatility of carboxylate ligand is reflected by variety of binding modes. It can coordinate as monodentate, bidentate, monodentate bridging, bidentate bridging, *etc*. The metal carboxylates play an important role in synthetic chemistry, biological and catalytic activities and physiological effect[1,2]. Among these carboxylate complexes, nickel representatives have attracted great attention since nickel has been recognized as a considerable important biological agent forming the active site of variety of metalloproteins[3,4]. In addition, their adducts with nitrogen have potential applications in area of organic conductor and magnetic materials[5]. In this paper, we reported the synthesis and crystal structure of $[Ni_2(nba)_4(tmen)_2(H_2O)]\cdot C_2H_5OH$, where nba = 2-nitrobenzoate, tmen = tetramethylethylenediamine.

Synthesis: To ethanolic solution (30 mL) of $NiCl_2\cdot 6H_2O$ (0.48 g, 2 mmol), was added ethanolic solution (30 mL) of 2-nitrobenzoate (0.67 g, 4 mmol) followed by tetramethylethylenediamine (0.23 g, 2 mmol). Catalytic amount of Et_3N was also added. The mixture was refluxed for 1 h. Single crystal suitable for X-ray crystallographic analysis was obtained by slow evaporation at room temperature after 3 days.

Structure determination: A single crystal of the present Ni(II) complex with dimensions of 0.2933 mm × 0.2052 mm × 0.0623 mm was mounted with paratone on a glass needle and the data were collected on an Oxford Diffraction SuperNovaarea-detector diffractometer with mirror optics monochromated MoK_α radiation (λ = 0.71073 Å) using ω scan mode at 173 K. The structure was solved by direct methods using SIR97[6]. All H-atoms were placed in geometrically calculated positions. Refinement of the structure was carried out on F^2 using full-matrix least-squares procedures. All calculations were performed using the SHELXL-97[6,7] program. Crystal data and refinement for complex **1** are listed in Table-1.

A molecular structure with atoms labeling scheme of complex **1** are shown in Fig. 1. Selected bond lengths and bond angles are listed in Table-2.

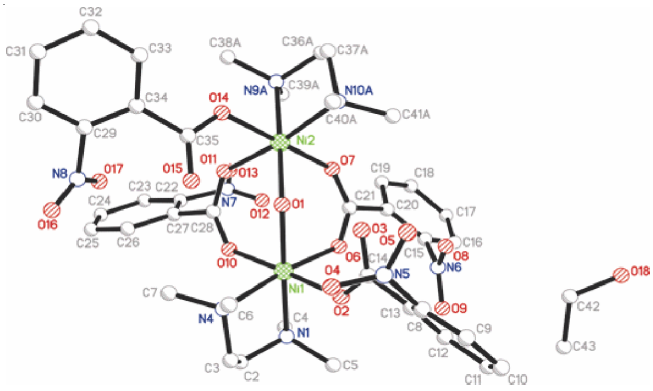

Fig. 1. Molecular structure of complex **1**

In the structure of $[Ni_2(nba)_4(tmen)_2(H_2O)]\cdot C_2H_5OH$ each nickel atom is six coordinated by two nitrogen atoms from tmen, three oxygen atoms from three nba ligands and one oxygen atom from water molecule showing distorted octahedral geometry. Two coordination modes of carboxylate ligands are

TABLE-1
CRYSTAL DATA AND STRUCTURE REFINMENT FOR COMPLEX 1

Empirical formula	$C_{42}H_{56}N_8O_{18}Ni_2$	Formula weight	1078.37
Space group	P121/c1	Crystal system	Monoclinic
a (Å)	11.3931(4)	α (º)	90
b (Å)	33.0702(4)	β (º)	111.310(4)
c (Å)	13.5623(4)	γ (º)	90
Volume $(Å)^3$	4760.5(2)	Z	4
D_c (Mg/m^3)	1.505	Absorption coefficient (mm^{-1})	0.874
$F_{(000)}$	22.56	θ Range (º)	1.73-28.24
h, k, l ranges	-1514, -4342, -17 18	Reflection collected/unique	52127/10855 [$R_{(int)}$ = 0.0912]
Data/restraints/parameters on F^2	10855/133/699	Goodness-of-fit	1.059
R indices [I > 2σ(I)]	R_1 = 0.0719, wR_2 = 0.2009	R indices (all data)	R_1 = 0.1213, wR_2 = 0.2192
Largest diff. peak and hole $(Å^{-3})$	0.989 and -0.833	–	–

TABLE-2
SELECTED BOND DITANCES (Å)
AND ANGLES (º) FOR COMPLEX **1**

Ni(1)-O(10)	2.042(3)	Ni(1)-O(2)	2.059(4)
Ni(1)-O(6)	2.034(3)	Ni(1)-N(1)	2.160(4)
Ni(2)-N(9A)	2.155(14)	Ni(2)-O(1)	2.119(3)
Ni(2)-N(10A)	2.165(14)	Ni(2)-O(11)	2.040(3)
O(10)-Ni(1)-N(4)	90.03(15)	O(6)-Ni(1)-O(10)	91.82(14)
O(1)-Ni(1)-N(4)	96.17(14)	O(10)-Ni(1)-O(2)	177.73(13)
O(6)-Ni(1)-N(4)	172.68(14)	N(1)-Ni(1)-N(4)	84.09(15)
O(2)-Ni(1)-N(4)	88.45(15)	Ni(1)-O(1)-Ni(2)	117.96(15)

present in the complex, monodentate and bidentate bridging modes. The bidentate carboxylates have *syn-syn* configurations. The binuclear Ni(II) atoms (Ni1, Ni2) are bridged by the O1 from aqua ligand and four oxygen atoms (O6, O7, O10, O11) from two different nba. The Ni-O-Ni linkage is nonlinear, exhibitting an angle of 117.96(15)º for Ni(1)-O(1)-Ni(2).

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