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NOTE

Synthesis and Crystal Structure of $Ni[phen(DTC)_2H_2O]\cdot 0.5H_2O$

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A novel complex $Ni[phen(DTC)_2H_2O]\cdot 0.5H_2O$, where phen = 1,10-phenanthroline and DTC is biphthalate, was synthesized and characterized by IR spectra, elemental analysis and single-crystal X-ray. The crystal is orthorhombic, space group Pbcm with unit cell parameters: a = 11.6669(6) Å, b = 11.1701(6) Å, c = 14.0370(6) Å, $\alpha = 90^o$, $\beta = 90^o$, $\gamma = 90^o$, V = 1829.31(16) $Å^3$, Z = 4, Mr = 430.05, Dc = 1.561Mg/cm^3, $\mu = 1.099$ mm^{-1}, F(000) = 884, T = 293(2) K, R = 0.0351, wR = 0.0930 with $I > 2\sigma(I)$. The crystal structure analysis shows that the nickel(II) is a five-coordinated in a slightly distorted tetragonal pyramid.

Key Words: Nickel(II) compound, Interactions, Crystal structure.

The organic aromatic carboxylic acid with metal ions build the one-dimensional chain, layered or mesh structure of the polymer supramolecular, which is currently of great interest chemistry and crystal research in the field of engineering[1-8]. In this paper, the synthesis and crystal structure of nickel(II) complex with the composition, $Ni[phen(DTC)_2H_2O]\cdot 0.5H_2O$, where phen = 1,10-phenanthroline and DTC = biphthate.

IR spectra were recorded on a Nexus-870 spectrophotometer. Elemental analysis were performed on a Elementar Vario ELZ(III) analyzer.

Synthesis: A mixture of $Ni(phen)_2$ (10 mmol), biphthalate (10 mmol) and H_2O (30 mL) was sealed in a 50 mL cone bottle under room temperature for 7 d. The product was brown pillar-shaped crystal. Elemental analysis (%) calcd. For $C_{20}H_{15}N_2O_{5.5}Ni$: C, 38.71 , H, 4.84, N, 9.03, Ni, 19.03; Found C, 38.70, H, 4.86, Ni, 19.00, N, 9.06. IR (KBr, ν_{max}, cm^{-1}): (C=O) 1620, (C=C) 1520, 1420, (phen) 848, 725.

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Crystal structure determination: A single crystal 0.65 mm × 0.35 mm × 0.20 mm was selected for crystallographic data collection at 293(2)K, and structure determinated with graphite-monochromatic MoK$_\alpha$ radiation (λ = 0.71073 Å). A total of 13299 reflections were collected in the range of $2.52° \leq \theta \leq 27.48°$, of which 2160 reflections were unique with R_{int} = 0.0211 and R = 0.0363 and wR = 0.0939; where $w = 1/[\sigma^2(F_0^2) + (0.0465P)^2 + 1.1654P]$ and $P = (F_0^2 + 2F_C^2)/3$. The maximum and minimum peaks on the final difference Fourier map are corresponding to 0.789 and -0.251 e/Å^3, respectively. The CCDC numbers was 607363.

The atomic coordinates and thermal parameters are listed in Table-1, and the selected bond lengths and bond angles in Table-2. Fig. 1 shows the molecular structure of the title compound. Fig. 2 shows the packing diagram of the title compound. From the Fig. 1, it is easy to see that the Ni(II) ion is five-coordinated and the whole molecular is in a slightly tetragonal pyramid.

TABLE-1
ATOMIC COORDINATES (× 10^4) AND THERMAL PARAMETERS (× 10^3 Å^2)

Atom	X	Y	Z	U(eq)
Ni	4768	3865(1)	2500(1)	24(1)
N	6059(2)	4034(1)	3411(1)	31(1)
C(1)	3478(2)	2703(2)	3948(1)	28(2)
C(2)	2387(2)	2624(2)	4511(1)	27(1)
C(5)	6027(2)	4012(2)	4358(3)	37(1)
C(6)	6990(2)	4272(2)	4940(2)	44(1)
O(1)	3575(1)	3647(1)	3446(1)	37(1)
O(2)	4176(8)	1868(1)	3985(1)	38(1)

TABLE-2
SELECTED BOND DISTANCES (Å) AND ANGLES (º)

Bond	Length	Angle	(º)	Angle	(º)
Ni-O(1)	1.9390(14)	O(1A)-Ni-O(1)	86.46(9)	N(11)-Ni-N(12)	79.7(2)
Ni-N(3)	2.0123(17)	O(1A)-Ni-O(1)	176.98(7)	C(20)-N(12)-Ni	129.0(4)
N-C(5)	1.3260(3)	O(1A)-Ni-O(1A)	95.70(7)	N(14)-Ni-N(11)	169.5(19)
N-C(9)	1.3610(3)	O(1)-Ni-N(1)	90.40(12)	C(21)-N(12)-Ni	112.2(4)
C(1)-O(2)	2.0830(5)	O(5)-N(1)-O(4)	28.40(6)	N(4)-C(4)-Ni	174.7(7)

In Fig. 2, it is notable that the structure of infinite one-dimensional chains expanding along the a axis, and the adjacent one-dimensional chains are extended into a two-dimensional supramolecular array with pentagonal large cavities by hydrogen bonds and other weak interaction.

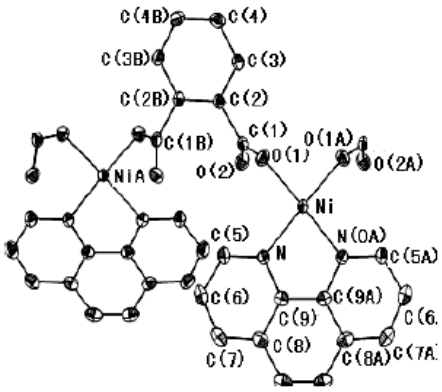

Fig. 1. Molecular strucxture of the $Ni[phen(DTC)_2H_2O]\cdot 0.5H_2O$

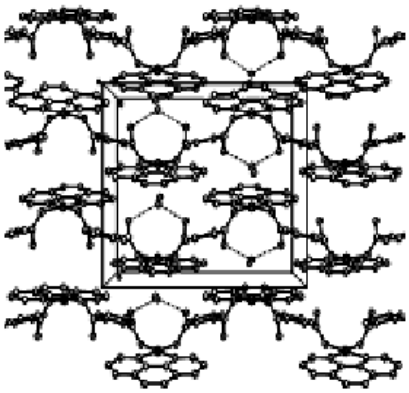

Fig. 2. Packing diagram of the $Ni[phen(DTC)_2H_2O]\cdot 0.5H_2O$

Conclusion

Crystal structure of a novel Nick(II) complex has been synthesized and characterized by IR, elemental analysis and X-ray diffraction analysis.

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