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NOTE

Synthesis and Crystal Structure of a New Compound: $[Ni(C_{16}H_{32}N_4)]_2{\cdot}Fe(SCN)_4{\cdot}(ClO_4)_2{\cdot}0.5H_2O$

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The nickel(II) compound of a tetraazamacrocycle ligand (L) (5,5,7,12, 12,14 -hexamethyl-1,4,8,11-tetraazamacrocyclic crown ethers) has been synthesized and characterized by IR spectra and elemental analysis. The crystal structure of the NiL was determined by single-crystal X-ray. The possessing parameters: a = 21.075(4)Å, b = 7.7038(13)Å, c = 16.366 (3)Å, α = 90º, β =104.870(2)º, γ = 90º, V = 2568.3(8)Å^3, Z = 2, Mr = 1174.41, Dc = 1.519 Mg/m^3, μ = 1.330 mm^{-1}, F(000) = 1226, T = 293(2) K, R = 0.0356, wR = 0.0969 for 5834 reflections with I > 2σ(I)

Key Words: Nickle(II) compound, Tetraazamacrocycle, Crystal structure.

During recent years, there has been increasing interest of tetraazamacrocycle, the construction of functional system compound lies on the character of mutual effect in reagents. Further study of tetraazamacrocycle material may contribute to the development of modern chemistry[1-3]. Hence, the synthesis of a substance with novel structures is of great significance. Recently, in our laboratory, a series of transition metal compounds have been synthesized and studied[4-7]. In this paper, the synthesis and crystal structure of a nickle(II) compound: $[Ni(C_{16}H_{32}N_4)]_2{\cdot}Fe(SCN)_4{\cdot}(ClO_4)_2{\cdot}0.5H_2O$ is reported.

$Ni(ClO_4)_2{\cdot}6H_2O$ was prepared in our laboratory, the other reagents were of A.R. grade and used without further purification. IR spectra were recorded on a Nexus-870 spectrophotometer. Elemental analysis for C, H and N were performed on an Elementar Vario EL-III analyzer. The crystal structure was determined by Siemens SMART CCD area-detector diffractometer.

Synthesis: 5,5,7,12,12,14-hexamethyl-1,4,8,11-tetraazamacrocyclic crown ethers (L) was prepared by similar procedure reported in the literature[5,8]. A mixture of $Ni(ClO_4)_2$ (0.366 g, 1.4 mmol), L (0.785 g, 2.8 mmol), $K_2[Fe(SCN)_4]$ (0.5124 g,

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1.4 mmol) and acrylonitrile (20 mL) was refluxed for 6 h to obtain a clear yellow solution and after standing at room temperature for 2 weeks well-shaped pale orange single crystals were obtained by slow evaporation. Main IR (KBr, cm^{-1}): 3130(s), 2110(m), 1651(m), 1090(s), 622(m). Elemental analysis: Calcd. (%) for $C_{36}H_{65}N_{12}O_{8.50}S_4Cl_2FeNi_2$: C, 36.78; H, 5.53; N, 14.31. Found(%): C, 36.75; H, 5.29; N, 14.26.

Crystal structure determination: A pale orange crystal (0.60 mm × 0.50 mm × 0.13 mm) was selected for crystallographic data collection at 293(2)K and structure determined with graphite-monochromatic MoK_α radiation ($\lambda = 0.71073$Å). A total of 18738 reflections were collected in the range of $2.51° \leq \theta \leq 27.48°$, of which 5834 reflections were unique with $R_{int} = 0.0202$. The final full-matrix least-squares refinement including 299 variable parameters for 5834 reflections with $I > 2\sigma(I)$ and converged with unweighted and weighted agreement factors of R = 0.0356, wR = 0.0969. where $w = 1/[\sigma^2(F_0^2) + (0.0577P)^2 + 1.1482P]$ and $P = (F_0^2 + 2F_C^2)/3$. The maximum and minimum peaks on the final difference Fourier map are corresponding to 0.566 and -0.357 $e.A^{-3}$, (CCDC No. 666019), respectively.

The atomic coordinates and thermal parameters are given in Table-1 and the selected bond lengths and bond angles are in Table-2. The molecular structure of $[Ni(C_{16}H_{32}N_4)]_2{\cdot}Fe(SCN)_4{\cdot}(ClO_4)_2{\cdot}0.5H_2O$ is shown in Fig. 1, in which the center nickel(II) ion is coordinated by four N atoms of the macrocycle in a little distortion plane square geometry. The molecular packing arrangement in the unit cell is shown in Fig. 2. There is a positive negative charge interaction between $[NiL]^{2+}$ and ClO_4^- or $[Fe(SCN)_4]^{2-}$ and the moleculars pack in a 2-D layer structure.

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

Auto	X	Y	Z	U (eq)
Ni	2576(1)	1609(1)	5017(1)	33(1)
N(1)	3106(1)	126(2)	5838(1)	40(1)
N(2)	1914(1)	1403(2)	5633(1)	34(1)
N(3)	2015(1)	2897(2)	4133(1)	38(1)
N(4)	3259(1)	1955(2)	4447(1)	42(1)

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

Bond	Length	Angle	(º)
Ni-N(1)	1.8946(16)	N(1)-Ni-N(3)	174.42(7)
Ni-N(2)	1.9256(16)	C(2)-N(1)-Ni	129.72(17)
Ni-N(3)	1.8963(16)	N(4)-Ni-N(2)	176.10(7)
Ni-N(4)	1.9243(18)	C(2)-N(1)-C(3)	121.06(19)
N(1)-C(2)	1.288(3)	C(3)-N(1)-Ni	108.98(13)
N(2)-C(5)	1.506(2)	C(4)-N(2)-C(5)	114.89(15)
N(3)-C(9)	1.287(3)	C(4)-N(2)-Ni	108.70(12)
N(4)-C(13)	1.505(3)	C(5)-N(2)-Ni	113.35(12)

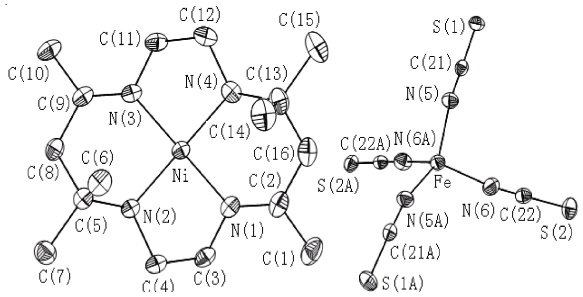

Fig. 1. Molecular structure of the title compound

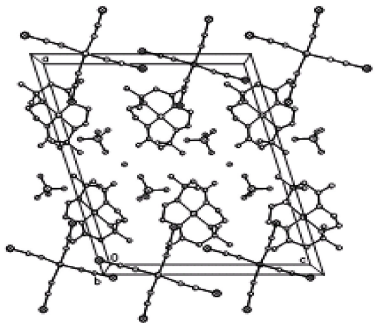

Fig. 2. Molecular packing arrangement in the unit cell

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