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***NOTE***

# Synthesis and Structure of Cu(II) Azamacrocyclic Complex: {$Me_6$[14]$N_4$dieneCu-SCN}$ClO_4$

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A novel copper(II) complex {$Me_6$[14]$N_4$dieneCu-SCN}$ClO_4$, where $Me_6$[14]$N_4$diene is 5,5,7,12,12,14-hexamethyl-1,4,8,11-tetraaza-macrocyclic-4,11-diene, was synthesized and characterized by IR spectra, elemental analysis and single-crystal X-ray. The crystal is monoclinic, space group P2(1)/n with unit cell parameters: a = 12.8691(4) Å, b = 11.3306(3) Å, c = 15.7727(5) Å, $\alpha = 90^o$, $\beta = 102.8200(10)^o$, $\gamma = 90^o$, V = 2242.56(12) $Å^3$, Z = 4, Mr = 501.53, Dc = 1.485 $Mg/cm^3$, $\mu = 1.219$ $mm^{-1}$, F(000) = 1052, T = 293(2) K, R = 0.0394, wR = 0.1068 for 16706 reflections with $I > 2\sigma(I)$. The center copper(II) is a five-coordinated in a slightly distorted square pyramidal environment.

**Key Words: Crystal structure, Copper(II) complex, Azamacrocycle, Electrostatic interaction.**

As we know, the structure of the polyaza-crown-ether compounds is similar to the basic structural units of the macrocycle compounds that exist widely in the life. Synthesis and studies on the structure and properties of this kind of compound are helpful to reveal the rules of chemical reactions in the organism[1-5]. Here we present a novel copper(II) azamacrocycle complex {$Me_6$[14]$N_4$dieneCu-SCN}$ClO_4$ and describe its synthesis and crystal structure.

$Me_6$[14]$N_4$dieneCu$(ClO_4)_2$ was prepared according to the report of literature[6]. IR spectrum was recorded on an Nexus-870 spectromer. Elemental analyses on an Elementar Vario EL-III elemental analyzer.

**Synthesis:** To a 20 mL methanol solution of $Me_6$[14]$N_4$dieneCu$(ClO_4)_2$ (2 mmol) was added to an aqueous solution (10 mL) of KSCN (6 mmol) and mixed 24 h, to obtain a clear solution and after standing at room temperature for 1 week. The product was blue square-shaped crystals. IR spectrum (KBr, $cm^{-1}$): 3446, 3241; 2044; 1663; 1105, 623. Elemental analysis (%) calcd. for $C_{17}H_{32}N_5O_4$ CuSCl: C, 40.71; H, 6.43; N, 13.96. Found: (%) C, 40.77; H, 6.35; N, 13.90.

**Crystal structure determination:** A blue crystal (0.60 mm × 0.50 mm × 0.50 mm) was selected for collection crystallographic data at 293(2) K and structure

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determinated with graphite-monochromatic $MoK_\alpha$ radiation ($\lambda$ = 0.71073 Å). A total of 16706 reflections were collected in the range of $2.23^\circ \leq \theta \leq 27.48^\circ$, of which 5094 reflections were unique with Rint = 0.0162 and R = 0.0394, wR = 0.1068; where $w = 1/[\sigma^2(F_0^2) + (0.0672P)^2 + 1.4260P]$ and $P = (F_0^2 + 2F_C^2)/3$. The maximum and minimum peaks on the final difference Fourier map are corresponding to 0.820 and -0.715e/Å$^3$ (CCDC No. 666018), respectively.

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, the Cu(II) ion is coordinated with four N atoms of the macrocycle and one N atom of the $SCN^-$. So it is obviously that copper(II) ion is in a slightly distorted square pyramidal geometry. Fig. 2 shows the packing diagram in the unit cell, that the moleculars are linked to the neighbours by electrostatic interactions.

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

| Atom | x | y | z | U (eq) |
|---|---|---|---|---|
| Cu | 4773(1) | 3790(1) | 2258(1) | 26(1) |
| S | 1097(1) | 3621(1) | 726(1) | 74(1) |
| N(1) | 4204(1) | 3426(2) | 3310(1) | 31(1) |
| N(2) | 4543(1) | 2134(1) | 1747(1) | 29(1) |
| N(3) | 5691(1) | 4032(2) | 1396(1) | 31(1) |
| N(4) | 5353(1) | 5256(1) | 2933(1) | 28(1) |
| N(5) | 3179(2) | 4279(2) | 1465(1) | 42(1) |
| Cl | 7840(1) | 3907(1) | 4230(1) | 48(1) |
| O(1) | 7384(3) | 4919(3) | 4502(2) | 123(1) |

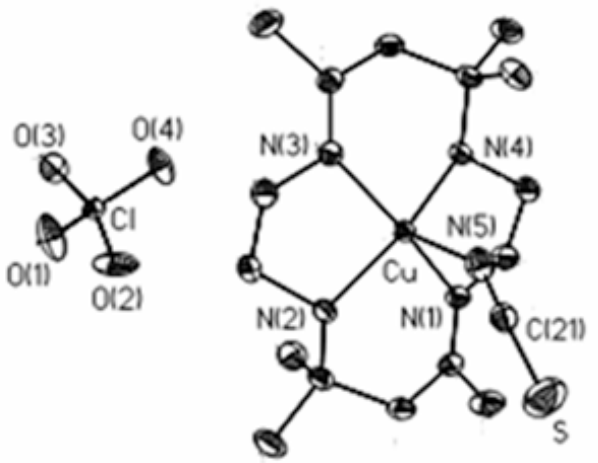


Fig. 1. Molecular structure of $\{Me_6[14]N_4$dieneCu-SCN$\}ClO_4$

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

| Bond | Length | Angle | (º) | Angle | (º) |
|---|---|---|---|---|---|
| Cu-N(1) | 2.0004(16) | N(1)-Cu-N(3) | 165.65(7) | O(2)-Cl-O(3) | 105.3(3) |
| Cu-N(3) | 2.0075(17) | N(1)-Cu-N(4) | 83.87(7) | C(11)-N(3)-Cu | 109.14(13) |
| Cu-N(5) | 2.2223(19) | N(1)-Cu-N(2) | 95.21(7) | C(12)-N(2)-Cu | 106.15(12) |
| S-C(21) | 1.634(2) | N(1)-Cu-N(5) | 93.23(7) | C(21)-N(5)-Cu | 147.08(18) |
| N(5)-C(21) | 1.143(3) | N(2)-Cu-N(5) | 88.42(7) | C(4)-N(4)-Cu | 104.95(12) |
| Cl-O(1) | 1.398(3) | N(5)-C(21)-S | 177.4(2) | N(4)-C(5)-C(8) | 107.17(16) |

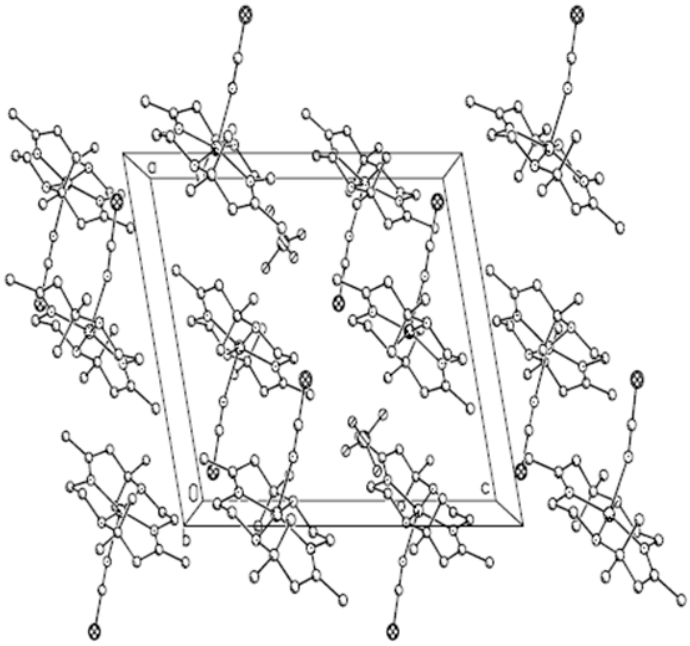

Fig. 2. Packing diagram of {$Me_6$[14]$N_4$dieneCu-SCN}$ClO_4$

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