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NOTE

Synthesis and Crystal Structure of $[(phen)_3Co]_2 \cdot Ni(SCN)_6$

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A novel compound $[(phen)_3Co]_2.Ni(SCN)_6$ where phen = 1,10-phenanthroline, 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 = 10.2295(19)Å, b = 19.413(4)Å, c = 18.951(4)Å, α = 90º, β = 95.157(3)º, γ = 90º, V = 3748.3 (12)Å^3, Z = 2, Mr = 1722.43, Dc = 1.526 Mg/cm^3, μ = 0.970 mm^{-1}, F(000) = 1756, T = 293(2) K, R = 0.0459, wR = 0.1239 for 28699 reflections with $I > 2\sigma(I)$.

Key Words: 1,10-Phenanthroline, Cobalt(II) compound, Crystal structure.

The replacement of 2,2′-bipyridine (bpy) subunits with 1,10-phenanthroline (phen) has led to greater conformational rigidity in these chelating systems and, to some extent, greater control of helicate formation[1-4]. Recently, in our laboratory, a series of transition metal compounds have been synthesized and studied[5-7]. In this paper, the synthesis and crystal structure of cobalt(II) complex $[(phen)_3Co]_2 \cdot Ni(SCN)_6$ is reported.

All of the reagents were of AR grade and used without further purification. IR spectra were recorded on a Nexus-870 spectrophotometer. Elemental analysis were performed on a Elementar Vario EL-III analyzer.

Synthesis: An aqueous solution (50 mL) of $NiSO_4$ (155 mg, 1 mmol) and KSCN (583 mg, 6 mmol) were mixed within 24 h, then diffused slowly into a mixed solution (80 mL) of $Co(ClO_4)_2 \cdot 6H_2O$ (732 mg, 2 mmol, 20 mL water) and 1,10-phenanthroline (1188 mg, 6 mmol, 60 mL alcohol) in a U-shaped tube, across an agar-gel medium. The temperature was maintained at 4 ± 0.5 ºC in a constant-temperature box. Well-shaped brown-red single crystals grew within 3 weeks and were isolated in about a 30 % yield. IR (KBr, cm^{-1}): 3434(w), 2089, 2042(s), 1518, 1429(m), 847, 712(m). Elemental analysis: Calcd. (%) for $C_{78}H_{48}N_{18}S_6Co_2Ni$: C, 58.32; H, 3.01; N, 15.69; Found(%): C, 58.29; H, 2.96; N, 15.74.

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Crystal structure determination: A single crystal (0.50 mm × 0.30 mm × 0.20 mm) was selected for crystallographic data collection at 293(2) K and structure determination on a Siemens SMART CCD area-detector diffractometer with graphite-monochromatic MoK_α radiation ($\lambda = 0.71073$ Å). A total of 28699 reflections were collected in the range of $2.10° \leq \theta \leq 27.48°$, of which 8600 reflections were unique with $R_{int} = 0.0231$. The final full-matrix least-squares refinement including 511 variable parameters for 8600 reflections with $I > 2\sigma(I)$ and converged with unweighted and weighted agreement factors of $R = 0.0459$ and $wR = 0.1239$, where $w = 1/[\sigma^2(F_0^2) + (0.0652P)^2 + 2.3041P]$ and $P = (F_0^2 + 2FC^2)/3$. The maximum and minimum peaks on the final difference Fourier map are corresponding to 0.971 and -0.614e/Å^3 (CCDC No. 719394), 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 $[(phen)_3Co]_2{\cdot}Ni(SCN)_6$. Fig. 2 shows the packing diagram of $[(phen)_3Co]_2{\cdot}Ni(SCN)_6$. From Fig. 1, it is easy to see that the cobalt(II) ion is six-coordinated with six N atoms. By the interaction of 1,10-phenanthroline and SCN^-, the $[(phen)_3Co]_2{\cdot}Ni(SCN)_6$ molecules pack in a three-dimensional structure.

TABLE-1
ATOMIC COORDINATES (× 10^4) AND THERMAL PARAMETERS (× 10^3 Å^2) of $[(phen)_3Co]_2{\cdot}Ni(SCN)_6$

Atom	x	Y	Z	U(eq)
Ni	0	0	5000	34(1)
Co	3035(1)	2476(1)	3142(1)	29(1)
N(1)	1564(3)	10(1)	4345(1)	44(1)
N(11)	4381(2)	3143(1)	3484(1)	34(1)
C(1)	2171(3)	-72(1)	3864(2)	39(1)
C(11)	5679(3)	3066(2)	3606(2)	40(1)
C(22)	3844(3)	3753(1)	3672(1)	37(1)
S(1)	3017(1)	-188(1)	3182(1)	58(1)

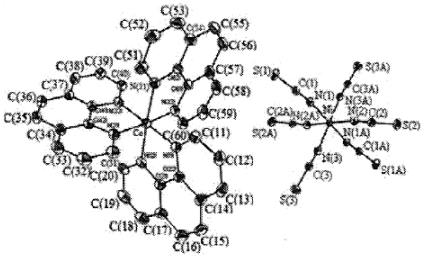

Fig.1. Atom labelling scheme for $[Co(Phen)_3]^{2+}$

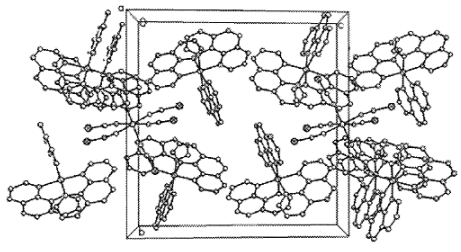

Fig. 2. Packing diagram of the title compound

TABLE-2
SELECTED BOND DISTANCES (Å) AND ANGLES (°)
OF $[(phen)_3Co]_2 \cdot Ni(SCN)_6$

Bond	Length	Angle	(°)	Angle	(°)
Co-N(11)	1.958(2)	N(12)-Co-N(11)	83.55(9)	N(1)-Ni-N(1A)	180.0
Ni-N(1)	2.112(3)	N(21)-Co-N(12)	88.27(9)	N(2)-Ni-N(1)	92.93(10)
N(1)-C(1)	1.159(4)	N(31)-Co-N(12)	175.34(9)	N(3)-Ni-N(1)	89.30(10)
S(1)-C(1)	1.634(3)	N(32)-Co-N(12)	93.60(9)	N(1)-C(1)-S(1)	179.6(3)
N(11)-C(11)	1.335(3)	N(21)-Co-N(11)	95.33(9)	C(1)-N(1)-Ni	161.6(2)

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