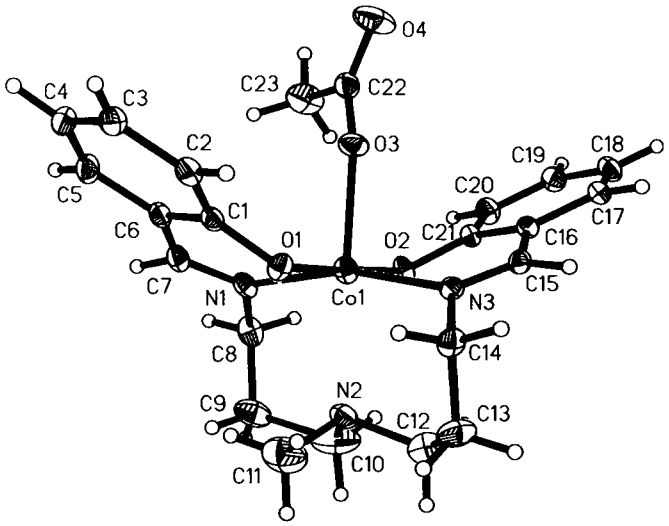
Crystal structure of acetato-μ-*N*,*N*′-bis(salicylidene)-3-methyl-3-aza-1,5-pentanediaminocobalt(III), $Co(C_2H_3O_2)(C_{21}H_{25}N_3O_2)$

S.-P. Xu*,[I], C.-H. Liu[II] and S.-F. Wang[III]

[I] Xuzhou Normal University, Chemistry Department, Xuzhou 221116, P. R. China
[II] Nanjing University, School of Life Sciences, Nanjing 210093, P. R. China
[III] Hainan University, Ocean College, Haikou 570228, P. R. China

Received July 26, 2006, accepted and available on-line September 12, 2006; CCDC no. 1267/1856



Abstract

$C_{23}H_{28}CoN_3O_4$, monoclinic, $P12_1/n1$ (no. 14), a = 9.400(4) Å, b = 19.203(5) Å, c = 12.112(5) Å, β = 92.33°, V = 2184.5 Å^3, Z = 4, $R_{gt}(F)$ = 0.066, $wR_{ref}(F^2)$ = 0.202, T = 293 K.

Source of material

Cobalt(II) acetate, equimolar 4-methyl-1,4,7-triazaheptane and two equivalent molar salicylaldehyde were dissolved in methanol and mixed together. The clear pink solution gradually got dark red. It was stood still in air for one week. Large cubic crystals were precipitated. They were filtered and washed with methanol and dried in air (yield 68 %).

Discussion

Cobalt is an important life-required element. Many cobalt(II,III) complexes were prepared and structurally determined by single X-ray analysis [1-7]. Especially, complexes with various Schiff bases are a hot branch of cobalt chemistry [1,2,4,6,7].

The title compound is a discrete mononuclear cobalt(III) complex. The central cobalt(III) atom is five-coordinated by three oxygen atoms and two nitrogen atoms from one tetradentate di-Schiff base ligand and one unidentate acetate anion. The metal is in a distorted square-pyramidal environment with the oxygen atom from the acetate situated in the apical position and the four donors from the Schiff base constituting the basal plane. The mean deviation of the basal plane is 0.151(2) Å, and the central cobalt(III) is 0.131(2) Å above the plane towards the apical oxygen atom. The dihedral angles between the Co basal plane and the two benzene rings in the Schiff base are 36.3(2)° and 28.2(2)°, and the two aromatic rings are at the angle of 64.3°.

Table 1. Data collection and handling.

Crystal:	dark blue prism, size 0.30 × 0.30 × 0.32 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	8.20 cm^{-1}
Diffractometer, scan mode:	Siemens SMART CCD, φ/ω
$2\theta_{max}$:	50.48°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4208, 3957
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3035
$N(param)_{refined}$:	280
Program:	SHELXTL [8]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(23A)	4*e*	−0.0356	0.7132	0.0868	0.08
H(23B)	4*e*	0.1110	0.7508	0.0805	0.08
H(23C)	4*e*	0.0430	0.7402	0.1962	0.08
H(2A)	4*e*	−0.0916	0.9762	0.4104	0.08
H(3A)	4*e*	−0.2236	0.9086	0.5303	0.08
H(4A)	4*e*	−0.1501	0.7971	0.5810	0.08
H(5A)	4*e*	0.0607	0.7531	0.5155	0.08
H(7A)	4*e*	0.2759	0.7672	0.4162	0.08
H(8A)	4*e*	0.4408	0.7908	0.1867	0.08
H(8B)	4*e*	0.4457	0.7538	0.3020	0.08
H(9A)	4*e*	0.6449	0.8197	0.3020	0.08
H(9B)	4*e*	0.5444	0.8586	0.3800	0.08
H(10A)	4*e*	0.6531	0.9394	0.2413	0.08
H(10B)	4*e*	0.5585	0.9028	0.1495	0.08
H(11A)	4*e*	0.5470	1.0090	0.3805	0.08
H(11B)	4*e*	0.3839	1.0237	0.3700	0.08
H(11C)	4*e*	0.4393	0.9516	0.4147	0.08
H(12A)	4*e*	0.4708	1.0087	0.0989	0.08
H(12B)	4*e*	0.5675	1.0420	0.1919	0.08
H(13A)	4*e*	0.3896	1.1169	0.1269	0.08
H(13B)	4*e*	0.3865	1.1052	0.2544	0.08
H(14A)	4*e*	0.1759	1.0589	0.2443	0.08
H(14B)	4*e*	0.1619	1.1010	0.1337	0.08
H(15A)	4*e*	0.1492	1.0474	−0.0300	0.08
H(17A)	4*e*	0.0885	0.9964	−0.2062	0.08
H(18A)	4*e*	0.1147	0.9082	−0.3383	0.08
H(19A)	4*e*	0.2244	0.8034	−0.2838	0.08
H(20A)	4*e*	0.3079	0.7869	−0.1001	0.08

* Correspondence author (e-mail: suoping_xu863@yahoo.com.cn)

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Co(1)	4e	0.21961(7)	0.90799(4)	0.19185(6)	0.0380(4)	0.0282(4)	0.0296(4)	0.0023(3)	−0.0018(3)	0.0013(3)
O(3)	4e	0.0142(4)	0.8719(2)	0.1430(3)	0.034(2)	0.027(2)	0.047(2)	−0.005(2)	−0.010(2)	−0.003(2)
O(4)	4e	−0.1698(5)	0.8200(2)	0.0595(5)	0.052(3)	0.042(2)	0.096(4)	−0.003(2)	−0.035(3)	−0.009(2)
C(22)	4e	−0.0515(5)	0.8184(3)	0.1049(4)	0.039(3)	0.031(3)	0.033(3)	−0.003(2)	−0.003(2)	0.002(2)
C(23)	4e	0.0237(7)	0.7494(3)	0.1183(7)	0.060(4)	0.027(3)	0.086(5)	0.001(3)	−0.018(4)	−0.005(3)
O(1)	4e	0.1375(3)	0.9448(2)	0.3204(3)	0.036(2)	0.022(2)	0.029(2)	−0.002(1)	0.006(1)	−0.002(1)
O(2)	4e	0.2980(4)	0.8697(2)	0.0638(3)	0.040(2)	0.031(2)	0.023(2)	0.013(2)	−0.005(1)	0.001(1)
N(1)	4e	0.2976(4)	0.8278(2)	0.2900(3)	0.034(2)	0.025(2)	0.029(2)	0.006(2)	−0.008(2)	−0.001(2)
N(2)	4e	0.4505(4)	0.9618(2)	0.2490(3)	0.029(2)	0.047(3)	0.029(2)	−0.002(2)	−0.001(2)	−0.005(2)
N(3)	4e	0.1944(4)	0.9996(2)	0.1081(3)	0.027(2)	0.021(2)	0.029(2)	−0.001(2)	−0.001(2)	−0.000(2)
C(1)	4e	0.0624(5)	0.9044(2)	0.3850(4)	0.032(2)	0.027(2)	0.020(2)	−0.005(2)	−0.004(2)	−0.002(2)
C(2)	4e	−0.0605(5)	0.9300(3)	0.4294(4)	0.036(3)	0.033(3)	0.028(2)	0.001(2)	−0.004(2)	−0.001(2)
C(3)	4e	−0.1383(6)	0.8901(3)	0.5015(4)	0.036(3)	0.050(3)	0.029(3)	−0.004(2)	0.002(2)	−0.001(2)
C(4)	4e	−0.0949(6)	0.8246(3)	0.5322(4)	0.043(3)	0.045(3)	0.031(3)	−0.014(2)	0.002(2)	0.005(2)
C(5)	4e	0.0275(6)	0.7981(3)	0.4914(4)	0.051(3)	0.031(3)	0.031(3)	−0.008(2)	−0.002(2)	0.007(2)
C(6)	4e	0.1065(5)	0.8363(3)	0.4158(4)	0.036(3)	0.031(3)	0.022(2)	−0.003(2)	−0.005(2)	0.002(2)
C(7)	4e	0.2351(5)	0.8059(3)	0.3758(4)	0.041(3)	0.027(2)	0.026(2)	0.002(2)	−0.005(2)	0.003(2)
C(8)	4e	0.4362(6)	0.7981(3)	0.2657(4)	0.043(3)	0.034(3)	0.037(3)	0.016(2)	0.000(2)	−0.001(2)
C(9)	4e	0.5574(6)	0.8449(3)	0.3041(6)	0.036(3)	0.053(4)	0.072(4)	0.013(3)	−0.016(3)	−0.008(3)
C(10)	4e	0.5645(7)	0.9161(4)	0.2268(8)	0.041(4)	0.072(5)	0.114(7)	−0.003(3)	0.001(4)	−0.031(5)
C(11)	4e	0.4557(9)	0.9889(5)	0.3635(6)	0.075(5)	0.083(6)	0.069(5)	−0.009(4)	0.001(4)	−0.030(4)
C(12)	4e	0.4740(8)	1.0244(4)	0.1749(7)	0.058(4)	0.063(4)	0.071(5)	−0.015(3)	−0.002(3)	0.003(4)
C(13)	4e	0.3699(6)	1.0832(3)	0.1831(6)	0.042(3)	0.034(3)	0.074(4)	−0.010(2)	0.000(3)	−0.007(3)
C(14)	4e	0.2132(5)	1.0643(3)	0.1717(4)	0.044(3)	0.021(2)	0.036(3)	−0.003(2)	0.002(2)	−0.003(2)
C(15)	4e	0.1718(5)	1.0028(2)	0.0022(4)	0.026(2)	0.021(2)	0.033(2)	0.002(2)	0.001(2)	0.005(2)
C(16)	4e	0.1787(5)	0.9441(2)	−0.0724(4)	0.022(2)	0.027(2)	0.029(2)	0.001(2)	−0.001(2)	0.003(2)
C(17)	4e	0.1320(5)	0.9532(3)	−0.1840(4)	0.028(2)	0.034(3)	0.030(2)	−0.001(2)	−0.005(2)	0.005(2)
C(18)	4e	0.1482(6)	0.9015(3)	−0.2623(4)	0.040(3)	0.040(3)	0.028(3)	−0.003(2)	−0.010(2)	0.000(2)
C(19)	4e	0.2131(6)	0.8397(3)	−0.2299(4)	0.045(3)	0.036(3)	0.027(3)	−0.006(2)	−0.002(2)	−0.006(2)
C(20)	4e	0.2612(6)	0.8296(3)	−0.1210(4)	0.042(3)	0.029(3)	0.033(3)	0.005(2)	−0.002(2)	−0.001(2)
C(21)	4e	0.2442(5)	0.8810(2)	−0.0392(4)	0.027(2)	0.027(2)	0.023(2)	0.001(2)	−0.001(2)	0.002(2)

References

1. Zhu, H.-L.; Liu, W.-J.; Wang, Y.-F.; Wang, D.-Q.: Crystal structure of bis[*N*-(2-aminopropyl)-salicylaldiminato]]cobalt(III) perchlorate, $C_{20}H_{26}ClCoN_4O_6$. Z. Kristallogr. NCS **218** (2003) 255-256.
2. Zhu, H.-L.; Liu, X.-Y.; Wang, Y.-F.; Wang, D.-Q.: Crystal structure of bis[*N*-(2-(2-hydroxyethylamino)ethyl)salicylideneimine]cobalt(III) nitrate, $C_{22}H_{30}CoN_5O_7$. Z. Kristallogr. NCS **218** (2003) 257-258.
3. Zhu, H.-L.; Zeng, Q.-F.; Xia, D.-S.; Liu, X.; Zhu, X.-L.; Wang, D.-Q.: Crystal structure of diaqua-di(2-methoxyacetato)cobalt(II), $C_6H_{14}CoO_8$. Z. Kristallogr. NCS **218** (2003) 313-314.
4. Zhu, H.-L.; Lin, Y.-S.; Meng, F.-J.; Zou, Y.; Wang, D.-Q.: Bis(*N*-(2-(2-hydroxyethylamino)ethyl)salicylideneimine)cobalt(III)chloride. Acta Crystallogr. E**59** (2003) m878-m879.
5. Qu, Y.; Zhu, H.-L.; You, Z.-L.; Tan, M.-Y.: Synthesis and Characterization of the Cobalt(III) and Nickel(II) Complexes with Pentane-2,4-dione $[Co(acac)_2NO_3]\cdot 2H_2O$ and $[Ni(acac)_2]\cdot 0.5CH_3OH$ (acac = Pentane-2,4-dione). Molecules **9** (2004) 950-957.
6. You, Z.-L.; Zhu, H.-L.; Liu, W.-S.: A linear trinuclear CoII-CoII-CoII complex with μ-acetate bridges: di-μ-acetato-bis[μ-*N*,*N*'-bis(salicylidene)-1,4-butanediaminato]tricobalt(II). Acta Crystallogr. E**60** (2004) m1900-m1902.
7. Zhu, H.-L.; Liu, X.-Y.: Syntheses and Crystal Structures of a Pair of Trinuclear Cobalt(II) Complexes with the Cations $[Co_3L_4(APYH)_2]$ and $[Co_3L_4(APY)_2]$ (APY = 2-aminopyridine, *L* = *N*-salicylaldehyde-2-iminopyridine). Syn. React. Inorg. Met.-Org. Nano-Met. Chem. **35** (2005) 193-196.
8. Sheldrick, G. M.: SHELXTL. Structure Determination Software Suite. Version 5.1. Bruker AXS, Madison, Wisconsin, USA 1997.