Crystal structure of bis[*N*-(2-(2-hydroxyethylamino)ethyl)salicylideneimine]-cobalt(III) nitrate, $C_{22}H_{30}CoN_5O_7$

H.-L. Zhu*[,I], X.-Y. Liu[I], Y.-F. Wang[I] and D.-Q. Wang[II]

[I] Wuhan Institute of Science and Technology, Department of Environmental and Chemical Engineering, Wuhan, 430073 P. R. China
[II] Liaocheng University, Department of Chemistry, Liaocheng, 252059 P. R.China

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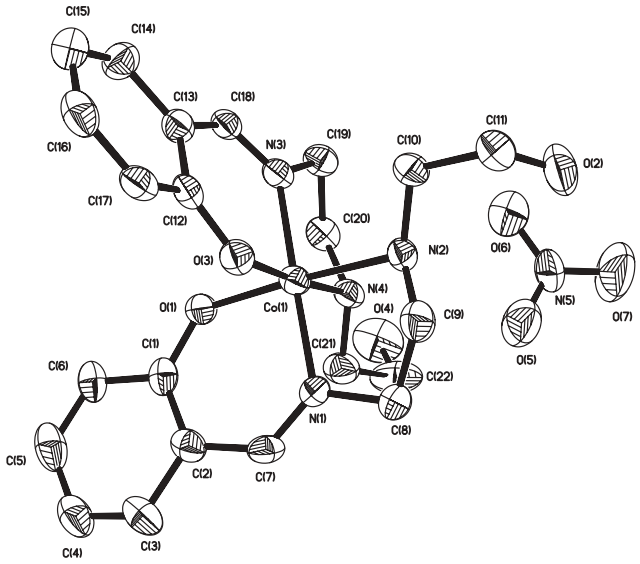

Abstract

$C_{22}H_{30}CoN_5O_7$, orthorhombic, *Fdd*2 (No. 43), $a = 18.576(8)$ Å, $b = 56.96(2)$ Å, $c = 9.874(4)$ Å, $V = 10448$ Å^3, $Z = 16$, $R_{gt}(F) = 0.057$, $wR_{ref}(F^2) = 0.123$, $T = 298$ K.

Source of material

All reagents and solvents were used as obtained without further purification. Equimolar salaldehyde (1 mmol, 122 mg) and 2-hydroxylaminoehtylamine (1 mmol, 104 mg) were dissolved in anhydrous alcohol solution (5 ml), with stirring. To the solution above was added $CoCl_2 \cdot 6H_2O$ (1 mmol, 238 mg) in anhydrous alcohol solution (5 ml). The resulting mixture was stood still in air to evaporate slowly. After about half of the solvent escaped, dark red prism crystals of the title compound were deposited and collected by filtration, washed with alcohol and dried in a vacuum desiccator over silica gel (yield 42%). Elemental analysis: found – C, 49.50%; H, 5.71%; N, 13.00%; calc. for $C_{21.88}H_{30}CoN_5O_7$ – C, 49.21%; H, 5.66%; N, 13.11%.

Discussion

The discovery of cobalt-containing coordination complexes that are able to bind molecular oxygen reversibly was made by Werner over a century ago [1]. Since that time a wide range of other complexes have been found to have similar behavior. In particular, the study of Schiff's base compounds has been particularly popular due to the structural similarity of such compounds to biological systems. A Schiff's base can be thought of as the imine product of the condensation reaction of a primary amine with an aldehyde or ketone. We have been interested in transition metal complexes with various Schiff's bases. In this paper we report the crystal structure of a new cobalt(III) complex with a new Schiff base. The title compound consists of a $[CoL_2]^+$ cation and a nitrate anion. The cobalt(III) atom in the cation is in a distorted octahedral geometry, being coordinated with four nitrogen and two phenoxo oxygen atoms from two chelate Schiff base ligands. The average Co(III)—N(imine) bond length of 1.955(5) Å is slightly shorter than that of the mean Co(III)—N(amine) distance (2.046(4) Å), however, all the Co—N bond contacts are in normal range comparing to those in the similar compounds. Four atoms, O(1), O(3), N(2) and N(4) constitute the equatorial plane of the Co octahedron with the mean deviation 0.071 Å. The three diagonal angles of the Co polyhedron are 175.5(2)°, 175.9(2)° and 178.9(2) Å, respectively, indicating a slightly distorted octahedron around the cobalt. There is also a hydrogen bond between the alcohol oxygen (O(2) atom) from the Schiff's base and O(5) atom from nitrate anion.

Table 1. Data collection and handling.

Crystal:	dark-red rectangular, size 0.13 × 0.24 × 0.30 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	7.05 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	52.84°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	13275, 4809
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2604
$N(param)_{refined}$:	316
Programs:	SHELXTL [2], SHELXTL-plus [3]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	16*b*	0.2418	0.0416	0.7933	0.059
H(3)	16*b*	0.3094	0.0438	0.6170	0.063
H(2)	16*b*	0.2499	0.0201	1.1082	0.145
H(4)	16*b*	0.4065	0.0422	0.2812	0.191
H(5)	16*b*	0.0316	0.0664	0.2432	0.087
H(6)	16*b*	0.0487	0.0935	0.0763	0.097
H(7)	16*b*	0.1373	0.1220	0.1017	0.100
H(8)	16*b*	0.2116	0.1211	0.2820	0.081

* Correspondence author (e-mail: hlzhu@wist.edu.cn)

Table 2. Continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(9)	16*b*	0.0676	0.0467	0.4426	0.064
H(8A)	16*b*	0.1543	0.0209	0.6335	0.080
H(8B)	16*b*	0.0743	0.0298	0.6453	0.080
H(9A)	16*b*	0.1016	0.0532	0.8258	0.086
H(9B)	16*b*	0.1371	0.0287	0.8581	0.086
H(10A)	16*b*	0.1921	0.0779	0.9442	0.083
H(10B)	16*b*	0.2728	0.0702	0.9332	0.083
H(11A)	16*b*	0.2279	0.0571	1.1369	0.092
H(11B)	16*b*	0.1641	0.0442	1.0637	0.092
H(10)	16*b*	0.2656	0.1570	0.8297	0.077
H(11)	16*b*	0.1657	0.1767	0.9003	0.088

Table 2. Continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(12)	16*b*	0.0522	0.1562	0.8949	0.082
H(13)	16*b*	0.0462	0.1192	0.8117	0.069
H(14)	16*b*	0.3283	0.1237	0.7586	0.059
H(19A)	16*b*	0.3762	0.0738	0.7647	0.070
H(19B)	16*b*	0.4008	0.0966	0.6875	0.070
H(20A)	16*b*	0.3619	0.0817	0.4820	0.079
H(20B)	16*b*	0.4074	0.0615	0.5490	0.079
H(21A)	16*b*	0.2900	0.0563	0.3516	0.084
H(21B)	16*b*	0.2406	0.0373	0.4193	0.084
H(22A)	16*b*	0.3511	0.0153	0.4674	0.123
H(22B)	16*b*	0.3256	0.0152	0.3163	0.123

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)	16*b*	0.21662(4)	0.07437(1)	0.63565(8)	0.0549(4)	0.0522(3)	0.0429(3)	–0.0053(4)	–0.0073(4)	0.0015(3)
N(1)	16*b*	0.1409(3)	0.05389(7)	0.5644(5)	0.063(3)	0.039(2)	0.050(3)	–0.005(2)	–0.007(2)	–0.001(2)
N(2)	16*b*	0.2087(2)	0.05322(8)	0.8036(4)	0.059(3)	0.048(2)	0.042(2)	0.002(2)	–0.003(2)	0.005(2)
N(3)	16*b*	0.2933(2)	0.09424(8)	0.7078(4)	0.047(3)	0.058(3)	0.042(2)	–0.004(2)	–0.006(2)	0.005(2)
N(4)	16*b*	0.3002(2)	0.05561(8)	0.5576(4)	0.059(3)	0.057(2)	0.040(2)	0.003(2)	–0.008(2)	–0.002(2)
N(5)	16*b*	0.3412(5)	0.0072(1)	0.7706(7)	0.107(5)	0.066(4)	0.094(5)	0.024(4)	–0.026(4)	0.019(3)
O(1)	16*b*	0.2175(2)	0.09297(6)	0.4775(4)	0.072(3)	0.052(2)	0.048(2)	–0.013(2)	–0.009(2)	0.007(2)
O(2)	16*b*	0.2565(3)	0.02869(9)	1.0427(5)	0.132(4)	0.087(3)	0.070(3)	0.021(3)	–0.003(3)	0.028(3)
O(3)	16*b*	0.1434(2)	0.09350(6)	0.7162(4)	0.047(2)	0.058(2)	0.057(2)	–0.003(2)	–0.001(2)	0.006(2)
O(4)	16*b*	0.4115(4)	0.0337(1)	0.3476(6)	0.108(4)	0.166(5)	0.109(5)	0.014(5)	0.018(3)	–0.024(4)
O(5)	16*b*	0.2858(4)	0.00794(9)	0.7135(6)	0.137(5)	0.070(3)	0.115(4)	0.014(4)	0.003(4)	–0.002(3)
O(6)	16*b*	0.3740(4)	0.0261(1)	0.7896(9)	0.164(6)	0.097(4)	0.141(5)	–0.023(4)	–0.039(5)	0.024(4)
O(7)	16*b*	0.3678(5)	–0.0113(1)	0.808(1)	0.191(8)	0.087(4)	0.30(1)	0.011(5)	–0.096(8)	0.058(6)
C(1)	16*b*	0.1702(3)	0.09241(9)	0.3773(6)	0.058(3)	0.050(3)	0.047(3)	0.017(3)	0.015(3)	0.001(3)
C(2)	16*b*	0.1147(3)	0.0762(1)	0.3634(6)	0.044(3)	0.066(3)	0.059(4)	0.015(3)	–0.004(3)	–0.011(3)
C(3)	16*b*	0.0690(4)	0.0771(1)	0.2508(6)	0.063(4)	0.099(5)	0.055(4)	0.022(4)	–0.008(3)	–0.007(4)
C(4)	16*b*	0.0781(4)	0.0935(1)	0.1526(7)	0.094(5)	0.094(5)	0.054(4)	0.027(5)	–0.011(4)	0.003(4)
C(5)	16*b*	0.1319(5)	0.1103(1)	0.1668(6)	0.111(6)	0.090(5)	0.048(4)	0.038(5)	0.012(4)	0.018(3)
C(6)	16*b*	0.1759(4)	0.1097(1)	0.2745(7)	0.070(4)	0.059(3)	0.072(4)	0.021(3)	0.003(3)	0.011(3)
C(7)	16*b*	0.1046(3)	0.0573(1)	0.4601(5)	0.061(4)	0.057(3)	0.044(3)	–0.013(3)	–0.003(3)	–0.011(3)
C(8)	16*b*	0.1244(4)	0.03434(9)	0.6553(6)	0.079(4)	0.052(3)	0.069(4)	–0.015(3)	–0.014(3)	0.013(3)
C(9)	16*b*	0.1384(4)	0.0422(1)	0.7975(7)	0.081(5)	0.066(4)	0.067(4)	–0.011(3)	–0.005(4)	0.020(3)
C(10)	16*b*	0.2237(4)	0.0644(1)	0.9342(5)	0.109(5)	0.062(3)	0.036(3)	–0.010(4)	–0.011(3)	0.001(2)
C(11)	16*b*	0.2143(4)	0.0487(1)	1.0554(6)	0.094(5)	0.077(4)	0.058(4)	–0.002(4)	–0.008(4)	0.006(3)
C(12)	16*b*	0.1519(3)	0.11452(9)	0.7660(5)	0.046(3)	0.056(3)	0.045(3)	0.003(3)	–0.008(2)	0.012(2)
C(13)	16*b*	0.2197(3)	0.12641(9)	0.7778(6)	0.051(3)	0.054(3)	0.054(3)	–0.008(3)	–0.006(3)	0.010(2)
C(14)	16*b*	0.2214(4)	0.1494(1)	0.8264(6)	0.070(4)	0.053(3)	0.070(4)	–0.007(3)	0.000(3)	–0.001(3)
C(15)	16*b*	0.1625(4)	0.1613(1)	0.8691(7)	0.085(5)	0.058(3)	0.078(4)	0.004(4)	–0.010(4)	0.002(3)
C(16)	16*b*	0.0938(4)	0.1489(1)	0.8640(6)	0.075(4)	0.082(4)	0.048(4)	0.024(4)	0.004(3)	0.011(3)
C(17)	16*b*	0.0906(3)	0.1267(1)	0.8142(5)	0.059(4)	0.069(4)	0.044(3)	–0.003(3)	–0.011(3)	0.004(3)
C(18)	16*b*	0.2864(3)	0.11499(9)	0.7476(5)	0.051(3)	0.053(3)	0.043(3)	–0.009(3)	–0.008(2)	0.009(2)
C(19)	16*b*	0.3649(3)	0.0842(1)	0.6903(6)	0.048(4)	0.069(4)	0.058(3)	–0.004(3)	–0.001(3)	0.001(3)
C(20)	16*b*	0.3640(4)	0.0708(1)	0.5576(7)	0.062(4)	0.075(4)	0.061(4)	0.001(3)	–0.004(3)	0.005(3)
C(21)	16*b*	0.2882(4)	0.0443(1)	0.4211(6)	0.067(4)	0.069(3)	0.073(4)	0.011(3)	–0.013(3)	–0.017(3)
C(22)	16*b*	0.3445(5)	0.0251(2)	0.3879(9)	0.098(6)	0.127(6)	0.082(5)	–0.001(5)	0.007(5)	–0.063(5)

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