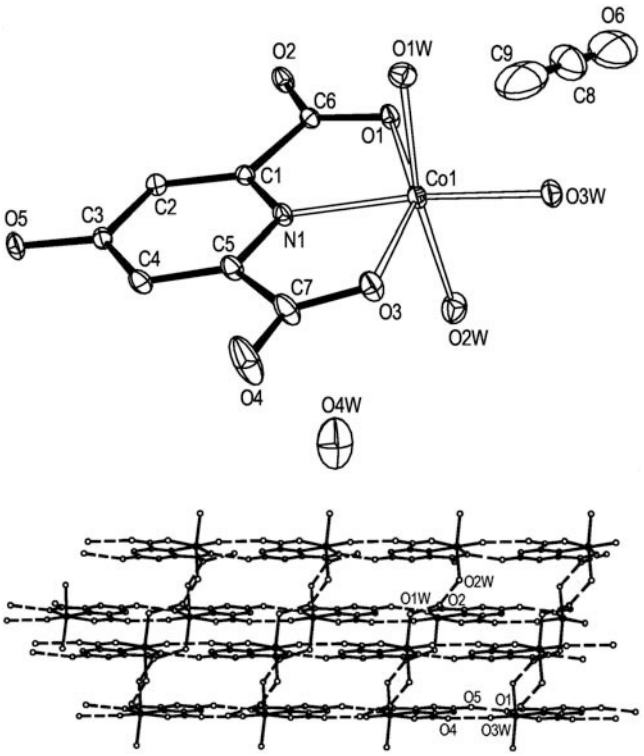
# Crystal structure of triaqua-(4-hydroxypyridine-2,6-dicarboxylato-*N*,*O*,*O'*)cobalt(II) — ethanol — water (1:0.25:1), $Co(H_2O)_3(C_7H_3NO_5) \cdot 0.25C_2H_5OH \cdot H_2O$

Jian-Ping Zou*, Qiang Peng, Zhi-Min Huang and Qiu-Ju Xing

Nanchang Hangkong University, School of Environmental and Chemical Engineering, Nanchang 330063, Jiangxi, P. R. China

Received February 28, 2010, accepted and available on-line April 6, 2010; CCDC no. 1267/2945







**Abstract**

$C_{7.50}H_{12.50}CoNO_{9.25}$, monoclinic, *C*12/*c*1 (no. 15), $a = 14.596(1)$ Å, $b = 7.0542(5)$ Å, $c = 22.570(2)$ Å, $\beta = 91.729(1)°$, $V = 2322.8$ Å$^3$, $Z = 8$, $R_{gf}(F) = 0.038$, $wR_{ref}(F^2) = 0.151$, $T = 293$ K.

**Source of material**

A mixture of cobalt(II) chloride dihydrate (0.25 mmol, 0.042 g), and chelidamic acid (0.25 mmol, 0.05 g,) was dissolved in a 10 ml water. After adding 5 ml ethanol (1 M) to the solution and stirring for about 4 h, the mixture was filtered. The filtrate was allowed to stand at room temperature. Red prismatic-shaped crystals that are stable in air were obtained after 10 d. Elemental analysis — found: C, 27.93 %; H, 3.93 %; N, 4.40 %; calculated for $C_{7.50}H_{12.50}CoNO_{9.25}$: C, 27.81 %; H, 3.86 %; N, 4.33 %.

**Experimental details**

Hydrogen atoms of water molecules were found in difference Fourier map and refined with $U_{iso}(H) = 1.5\ U_{eq}(O)$. The hydrogen atoms of C2, C4, and O5 were placed in calculated positions and allowed to ride on their parent atoms with $d$(O—H) = 0.82 Å, $d$(C—H) = 0.93 Å, and $U_{iso}(H) = 1.2\ U_{eq}(C)$, $U_{iso}(H) = 1.5\ U_{eq}(O)$, respectively. The hydrogen atoms at the ethanol molecule (C8, C9 and O6) were not located. The site occupancy of the ethanol molecule was determined according to its electron density. The same coordinate and anisotropic displacement parameters are used for O6 and C9 during the refinement.

**Discussion**

The symmetry independent unit of the crystal structure of the title compound consists of one Co(II) ion, one chelidamate ligand, three coordinated water molecules, one lattice water molecule, and a quater ethanol molecule. The Co(II) ion is coordinated by one N atom and two O atoms from the tridentate chelidamate ligands and three coordinated water molecules forming a distorted octahedron (figure, top). The equatorial plane consists of N and O atoms from the chelidamate ligands and one coordinated water molecule (O3W), the axial positions are occupied by two water molecules (O1W and O2W). The Co—O bond distances range from 2.025(2) to 2.209(3) Å, which fall in the normal range reported for the cobalt complexes [1-3]. The bond distance of Co—N (2.030(3) Å) is close to the values reported in the cobalt complexes [2,3]. Similar to the cobalt complex with chelidamate ligands [2], the discrete cobalt complexes in the title crystal structure are linked by the hydrogen bonds between the atoms O1 and O5, and O3W and O4 to form an infinite chain along [100]. And the neighbor chains are connected into a layer in (001) through the hydrogen bonds between O1W and O2, O2W and O2 (figure, bottom). These layers are in turn inter-linked through a very complex hydrogen bonding network involving water molecules and oxygen atoms from the chelidamate ligands to form a 3D supramolecular framework.

**Table 1.** Data collection and handling.

| | |
|---|---|
| Crystal: | red prism, size 0.13 × 0.20 × 0.30 mm |
| Wavelength: | Mo $K_\alpha$ radiation (0.71073 Å) |
| $\mu$: | 15.24 cm$^{-1}$ |
| Diffractometer, scan mode: | Bruker SMART CCD, $\varphi/\omega$ |
| $2\theta_{max}$: | 50° |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$: | 8310, 2058 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\ \sigma(I_{obs})$, 1926 |
| $N(param)_{refined}$: | 201 |
| Programs: | SHELXS-97 [4], SHELXL-97 [5] |

* Correspondence author (e-mail: zoujp@nchu.edu.cn)

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

| Atom | Site | $x$ | $y$ | $z$ | $U_{iso}$ |
|---|---|---|---|---|---|
| H(1WA) | 8*f* | 0.460(3) | 0.582(7) | 0.606(2) | 0.054 |
| H(1WB) | 8*f* | 0.377(2) | 0.533(7) | 0.636(2) | 0.054 |
| H(2WA) | 8*f* | 0.552(4) | –0.045(8) | 0.610(2) | 0.066 |
| H(2WB) | 8*f* | 0.609(3) | 0.030(8) | 0.648(3) | 0.066 |
| H(3WA) | 8*f* | 0.631(3) | 0.413(6) | 0.694(2) | 0.058 |
| H(3WB) | 8*f* | 0.589(4) | 0.278(6) | 0.727(2) | 0.058 |
| H(4WA) | 8*f* | 0.213(5) | 0.12(1) | 0.850(3) | 0.117 |
| H(4WB) | 8*f* | 0.236(6) | –0.02(1) | 0.808(3) | 0.117 |
| H(5A) | 8*f* | 0.1286 | –0.1178 | 0.5084 | 0.048 |
| H(2A) | 8*f* | 0.3192 | 0.0586 | 0.4529 | 0.028 |
| H(4A) | 8*f* | 0.1858 | –0.0581 | 0.6032 | 0.035 |

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

| Atom | Site | Occ. | $x$ | $y$ | $z$ | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Co(1) | 8*f* | | 0.48547(3) | 0.25649(6) | 0.63760(2) | 0.0232(5) | 0.0309(5) | 0.0220(5) | –0.0075(2) | –0.0008(3) | –0.0005(2) |
| O(1W) | 8*f* | | 0.4350(2) | 0.5383(4) | 0.6349(1) | 0.039(2) | 0.037(1) | 0.033(2) | –0.002(1) | 0.005(1) | 0.004(1) |
| O(2W) | 8*f* | | 0.5584(2) | –0.0048(4) | 0.6434(1) | 0.042(2) | 0.044(2) | 0.046(2) | 0.003(1) | –0.009(1) | –0.010(1) |
| O(3W) | 8*f* | | 0.5823(2) | 0.3531(4) | 0.6962(1) | 0.036(2) | 0.048(2) | 0.031(1) | –0.017(1) | –0.008(1) | 0.005(1) |
| O(4W) | 8*f* | | 0.2516(3) | 0.0558(8) | 0.8316(2) | 0.060(3) | 0.132(4) | 0.044(2) | –0.010(2) | 0.009(2) | –0.017(2) |
| O(1) | 8*f* | | 0.5277(2) | 0.2904(4) | 0.5450(1) | 0.023(1) | 0.038(1) | 0.026(1) | –0.011(1) | 0.002(1) | –0.002(1) |
| O(2) | 8*f* | | 0.4817(2) | 0.2327(3) | 0.4512(1) | 0.029(2) | 0.040(2) | 0.027(2) | –0.0125(9) | 0.007(1) | –0.0033(9) |
| O(3) | 8*f* | | 0.3880(2) | 0.1678(4) | 0.7020(1) | 0.039(2) | 0.051(2) | 0.023(1) | –0.017(1) | 0.002(1) | –0.003(1) |
| O(4) | 8*f* | | 0.2536(3) | 0.0268(8) | 0.7110(2) | 0.084(2) | 0.139(3) | 0.039(2) | –0.077(2) | 0.014(2) | –0.008(2) |
| O(5) | 8*f* | | 0.1701(2) | –0.0812(3) | 0.4876(1) | 0.022(1) | 0.044(1) | 0.031(1) | –0.014(1) | –0.002(1) | 0.001(1) |
| O(6) | 8*f* | 0.25 | 0.5810(9) | 0.739(2) | 0.7275(8) | 0.18(2) | 0.055(7) | 0.13(1) | –0.010(6) | –0.04(1) | –0.007(5) |
| N(1) | 8*f* | | 0.3816(2) | 0.1407(4) | 0.5878(1) | 0.022(1) | 0.030(1) | 0.022(1) | –0.008(1) | 0.002(1) | 0.001(1) |
| C(1) | 8*f* | | 0.3847(2) | 0.1367(4) | 0.5289(1) | 0.019(2) | 0.021(1) | 0.024(2) | –0.000(1) | 0.002(1) | 0.000(1) |
| C(2) | 8*f* | | 0.3154(2) | 0.0613(4) | 0.4939(1) | 0.022(2) | 0.026(2) | 0.021(2) | –0.001(1) | 0.002(1) | –0.002(1) |
| C(3) | 8*f* | | 0.2386(2) | –0.0118(4) | 0.5221(2) | 0.021(2) | 0.022(1) | 0.026(2) | –0.001(1) | –0.001(1) | –0.003(1) |
| C(4) | 8*f* | | 0.2357(2) | –0.0087(5) | 0.5837(2) | 0.027(2) | 0.032(2) | 0.030(2) | –0.008(1) | 0.007(1) | 0.000(1) |
| C(5) | 8*f* | | 0.3089(2) | 0.0697(5) | 0.6148(2) | 0.028(2) | 0.034(2) | 0.027(2) | –0.010(1) | 0.005(1) | –0.001(1) |
| C(6) | 8*f* | | 0.4709(3) | 0.2275(4) | 0.5053(2) | 0.021(2) | 0.022(2) | 0.023(2) | –0.003(1) | 0.004(2) | –0.001(1) |
| C(7) | 8*f* | | 0.3164(3) | 0.0872(6) | 0.6817(2) | 0.047(2) | 0.055(2) | 0.026(2) | –0.030(2) | 0.007(2) | –0.003(2) |
| C(8) | 4*e* | 0.50 | ½ | 0.747(2) | ¾ | 0.06(1) | 0.043(8) | 0.07(1) | 0 | 0.00(1) | 0 |
| C(9) | 8*f* | 0.25 | 0.5810(9) | 0.739(2) | 0.7275(8) | 0.18(2) | 0.055(7) | 0.13(1) | –0.010(6) | –0.04(1) | –0.007(5) |

*Acknowledgments.* We gratefully acknowledge the financial support by the NSF of China (grant no. 20801026), and the NSF of Jiangxi Province (grant no. 2008GQC0036).

## References

1. Cui, J.-Z.; Zhang, H.; Lin, T.; Kang, H.-J.; Gao, H.-L.: Tetraaqua-(4-hydroxypyridine-2,6-dicarboxylato)cobalt(II). Acta Crystallogr. **E62** (2006) m2499-m2501.
2. Zhou, G.-W.; Guo, G.-C.; Liu, B.; Wang, M.-S.; Cai, L.-Z.; Huang, J.-S.: 3-D Hydrogen-Bonded Frameworks of Two Metal Complexes With Chelidamic Acid: Syntheses, Structures and Magnetism. Bull. Korean Chem. Soc. **25** (2004) 676-680.
3. Xiao, D.; Wang, E.; An, H.; Hu, C.: The helical structure induced by metal–organic complexes: synthesis and characterization of a novel layered vanadate complex containing double helical chains. J. Mol. Struct. **707** (2004) 77-81.
4. Sheldrick, G. M.: SHELXS-97. Program for the Solution of Crystal Structures. University of Göttingen, Germany 1997.
5. Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.