Crystal structure of aqua[1,3-propanediaminediacetato]-cobalt(II) dihydrate, $\text{Co}(\text{H}_2\text{O})(\text{C}_7\text{H}_{12}\text{N}_2\text{O}_4) \cdot 2\text{H}_2\text{O}$

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Abstract

$\text{C}_7\text{H}_{18}\text{CoN}_2\text{O}_7$, monoclinic, $P2_1/n$ (no. 14), $a = 9.134(5)$ Å, $b = 7.704(5)$ Å, $c = 16.674(5)$ Å, $\beta = 102.517(5)^\circ$, $V = 1145.4$ Å³, $Z = 4$, $R_g(F) = 0.041$, $wR_{\text{ref}}(F^2) = 0.094$, $T = 293$ K.

Source of material

A solution of 1,3-propanediamine (0.222 g, 3 mmol), ethyl chloroacetate (0.735 g, 6 mmol), and K_2CO_3 (3.31 g, 24 mmol) in DMF (150 mL) was refluxed for 20 h and then filtered. The solvent was evaporated, and the residue was obtained. Then the residue was hydrolyzed with sodium hydroxide, and the H_2L was obtained. A mixture of H_2L (0.0301 g, 0.1 mmol) and $\text{Co}(\text{ClO}_4)_2 \cdot 6\text{H}_2\text{O}$ (0.0732 g, 2 mmol) in methanol (10 ml) and water (10 ml) was stirred for 10 min, the resulting solution was filtered. Red crystals were obtained by slow evaporation of the filtrate at room temperature (yield, 30 %).

Experimental details

All H atoms on C atoms were generated geometrically and refined as riding atoms with $d(\text{C}—\text{H}) = 0.93$ Å and $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$. The nitrogen H atom and water H atoms were located from difference Fourier map and refined with $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{O}, \text{N})$.

Discussion

Transition metal complexes of edta-like (edta = ethylenediamine tetraacetic) ligands show varieties in coordination number and stereochemistry. The role of chelate ring size appeared to be im-

portant in determining coordination number, configuration and stability of a complex [1].

The building unit of the title crystal structure consists of two cobalt ions. Each Co^{2+} is surrounded by two nitrogen atoms and two oxygen atoms of the H_2L ligand, and two water oxygen atoms in form of a distorted octahedron. One oxygen atom from water solvent molecule bridges two $\text{Co}(\text{II})$ centres to form a dimer with the $\text{Co} \cdots \text{Co}$ distance of 2.8617(1) Å. The bond lengths of $\text{Co}—\text{O}$ fall in the range of 1.880(2)–1.920(2) Å, which are similar to those reported in the literature [2].

Table 1. Data collection and handling.

Crystal:	red block, size $0.21 \times 0.26 \times 0.31$ mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ :	15.25 cm^{-1}
Diffraction, scan mode:	Oxford Diffraction Gemini R Ultra, ω
$2\theta_{\text{max}}$:	58.38°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	7605, 2715
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1886
$N(\text{param})_{\text{refined}}$:	178
Programs:	SHELXS-97 [3], SHELXL-97 [4]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U_{iso}
H(1A)	4e	1.1314	1.1445	0.3239	0.037
H(1B)	4e	1.0990	1.0611	0.2359	0.037
H(2A)	4e	1.3282	0.9612	0.3050	0.044
H(2B)	4e	1.2171	0.8038	0.2925	0.044
H(3A)	4e	1.3675	0.8057	0.4261	0.039
H(3B)	4e	1.3029	0.9879	0.4419	0.039
H(4A)	4e	1.1867	0.5335	0.4596	0.039
H(4B)	4e	1.1683	0.5901	0.3675	0.039
H(6A)	4e	0.7917	1.0222	0.2184	0.034
H(6B)	4e	0.8741	1.1862	0.2625	0.034
H(1WA)	4e	0.608(5)	0.707(5)	0.487(2)	0.056
H(3WA)	4e	1.123(2)	1.123(2)	0.436(2)	0.056
H(1WB)	4e	0.670(5)	0.647(8)	0.420(3)	0.117
H(2WA)	4e	0.977(5)	0.908(6)	0.084(5)	0.149
H(2WB)	4e	1.022(6)	0.733(3)	0.098(3)	0.149
H(3WB)	4e	0.998(3)	1.210(3)	0.443(2)	0.027
H(1N)	4e	0.955(4)	0.870(5)	0.288(2)	0.032
H(2N)	4e	1.177(4)	0.792(4)	0.494(2)	0.034

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Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
C(1)	4e	1.1105(4)	1.0361(4)	0.2940(2)	0.033(2)	0.033(2)	0.028(2)	−0.005(1)	0.009(1)	0.003(1)
C(2)	4e	1.2406(4)	0.9133(5)	0.3211(2)	0.030(2)	0.045(2)	0.038(2)	0.001(2)	0.015(2)	0.001(2)
C(3)	4e	1.2789(4)	0.8786(4)	0.4131(2)	0.024(2)	0.038(2)	0.033(2)	0.006(1)	0.003(1)	−0.004(2)
C(4)	4e	1.1311(4)	0.6088(4)	0.4171(2)	0.043(2)	0.018(2)	0.037(2)	0.009(1)	0.007(2)	−0.002(1)
C(5)	4e	0.9667(4)	0.5631(4)	0.4010(2)	0.045(2)	0.020(2)	0.015(1)	0.006(1)	−0.002(1)	0.002(1)
C(6)	4e	0.8397(4)	1.0701(4)	0.2714(2)	0.040(2)	0.026(2)	0.016(1)	0.006(1)	−0.002(1)	0.007(1)
C(7)	4e	0.7265(3)	1.0798(4)	0.3260(2)	0.030(2)	0.016(1)	0.023(2)	0.001(1)	−0.001(1)	−0.002(1)
O(1)	4e	0.7690(2)	1.0185(2)	0.3986(1)	0.025(1)	0.020(1)	0.019(1)	0.0045(8)	−0.0002(8)	0.0030(8)
O(2)	4e	0.9250(3)	0.4106(3)	0.3944(2)	0.056(2)	0.012(1)	0.040(1)	0.002(1)	−0.009(1)	0.002(1)
Co(1)	4e	0.96362(4)	0.91211(4)	0.42264(2)	0.0230(2)	0.0119(2)	0.0152(2)	0.0014(2)	−0.0001(1)	−0.0002(2)
O(3)	4e	0.8760(2)	0.6919(2)	0.3935(1)	0.030(1)	0.012(1)	0.021(1)	0.0015(8)	−0.0022(9)	−0.0011(8)
O(4)	4e	0.6019(3)	1.1473(3)	0.2997(1)	0.030(1)	0.046(1)	0.032(1)	0.015(1)	−0.001(1)	0.011(1)
O(1W)	4e	0.5977(4)	0.6468(5)	0.4437(2)	0.056(2)	0.096(3)	0.092(3)	0.012(2)	0.037(2)	0.020(2)
O(2W)	4e	1.0521(5)	0.8383(6)	0.1020(3)	0.122(4)	0.086(3)	0.102(3)	−0.015(3)	0.048(3)	0.016(3)
O(3W)	4e	1.0583(2)	1.1183(2)	0.4669(1)	0.025(1)	0.015(1)	0.014(1)	0.0009(8)	0.0042(8)	−0.0008(8)
N(1)	4e	0.9692(3)	0.9614(3)	0.3086(1)	0.025(1)	0.021(1)	0.016(1)	0.002(1)	0.002(1)	0.000(1)
N(2)	4e	1.1557(3)	0.7930(3)	0.4433(2)	0.026(1)	0.022(1)	0.019(1)	0.007(1)	0.001(1)	−0.001(1)

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