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Crystal structure of tetraqua-bis(4-(hydroxymethyl)benzoato-κO)cobalt(II), $C_{16}H_{22}O_{10}Co$

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Abstract

$C_{16}H_{22}O_{10}Co$, monoclinic, $P2_1/c$ (no. 14), $a = 9.811(2)$ Å, $b = 15.530(3)$ Å, $c = 13.639(3)$ Å, $\beta = 122.122(3)^\circ$, $V = 1760.0(6)$ Å³, $Z = 4$, $R_{gt}(F) = 0.0243$, $wR_{ref}(F^2) = 0.0821$, $T = 297(2)$ K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of materials

The title compound was synthesized by mixing 0.761 g (0.5 mmol) 4-hydroxymethylbenzoic acid, 0.146 g (0.5 mmol) $Co(NO_3)_2 \cdot 6 H_2O$, 12 mL ethyl alcohol absolute and 6 mL H_2O in a 50 mL beaker. After all the raw materials were dissolved, triethylamine was added dropwise to adjust the pH to 6. Then waiting for the solvent evaporate, red rod crystals were separated out with 79% yield after about 20 days. **Elemental analysis** calculated for $C_{16}H_{22}O_{10}Co$: C 44.35, H 5.08, O 36.93%;

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Table 1: Data collection and handling.

Crystal:	Red block
Size:	$0.12 \times 0.10 \times 0.10$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	1.03 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	26.5° , >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	13698, 3630, 0.024
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3147
$N(\text{param})_{\text{refined}}$:	275
Programs:	Bruker [1], SHELX [2, 3]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Co1	0.5000	1.0000	0.0000	0.01816(11)
O1	0.64030(14)	0.81942(7)	0.15867(9)	0.0337(3)
O2	0.49999(12)	0.93834(7)	0.13528(8)	0.0263(2)
O3	0.87911(15)	0.80479(7)	0.74794(9)	0.0369(3)
H3A	0.826(2)	0.7601(9)	0.7288(17)	0.055*
O4	0.56340(12)	0.88314(6)	−0.04615(8)	0.0241(2)
H4A	0.585(2)	0.8522(10)	0.0100(10)	0.036*
H4B	0.5001(19)	0.8584(11)	−0.1071(10)	0.036*
O5	0.25526(12)	0.97162(8)	−0.10987(9)	0.0257(2)
H5A	0.221(2)	0.9309(9)	−0.0903(17)	0.039*
H5B	0.196(2)	1.0121(9)	−0.1184(19)	0.039*
C9	0.62874(16)	0.87075(9)	0.31850(11)	0.0217(3)
C10	0.70424(18)	0.79877(9)	0.38683(12)	0.0263(3)
H10	0.7204	0.7503	0.3542	0.032*
C11	0.75581(17)	0.79852(10)	0.50356(11)	0.0273(3)
H11	0.8067	0.7501	0.5485	0.033*
C12	0.73178(18)	0.87002(9)	0.55331(12)	0.0261(3)
C13	0.65422(18)	0.94148(9)	0.48438(12)	0.0291(3)
H13	0.6365	0.9896	0.5168	0.035*
C14	0.60319(17)	0.94206(9)	0.36874(12)	0.0268(3)
H14	0.5513	0.9904	0.3239	0.032*
C15	0.58610(16)	0.87520(9)	0.19539(11)	0.0215(3)
C16	0.7845(2)	0.87454(11)	0.67937(13)	0.0396(4)
H16A	0.8452	0.9273	0.7120	0.048*
H16B	0.6893	0.8780	0.6839	0.048*
Co2	0.0000	1.0000	0.0000	0.01894(11)
O6	0.20139(14)	0.84480(7)	0.18122(9)	0.0340(3)
O7	0.00257(12)	0.93662(7)	0.13624(8)	0.0254(2)

Table 2 (continued)

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
O8	0.35407(14)	0.80992(7)	0.74756(9)	0.0344(3)
H8A	0.3380	0.7512	0.7408	0.052*
O9	0.07394(13)	0.88217(7)	−0.04228(9)	0.0261(2)
H9A	0.116(2)	0.8560(11)	0.0198(10)	0.040*
H9B	0.017(2)	0.8502(10)	−0.0971(11)	0.039*
O10	−0.24270(12)	0.96813(7)	−0.10997(9)	0.0253(2)
H10A	−0.282(2)	0.9312(10)	−0.0888(17)	0.038*
H10B	−0.300(2)	1.0108(9)	−0.1268(19)	0.038*
C1	0.13634(17)	0.87290(9)	0.32336(11)	0.0225(3)
C2	0.27268(18)	0.83125(9)	0.41126(12)	0.0292(3)
H2	0.3465	0.8078	0.3957	0.035*
C3	0.2997(2)	0.82429(10)	0.52167(13)	0.0313(3)
H3	0.3917	0.7966	0.5795	0.038*
C4	0.19044(18)	0.85846(9)	0.54631(12)	0.0262(3)
C5	0.05240(18)	0.89816(10)	0.45795(12)	0.0288(3)
H5	−0.0233	0.9197	0.4728	0.035*
C6	0.02630(17)	0.90594(10)	0.34837(12)	0.0276(3)
H6	−0.0659	0.9336	0.2906	0.033*
C7	0.11252(16)	0.88439(9)	0.20573(11)	0.0218(3)
C8	0.2183(2)	0.85791(11)	0.66624(12)	0.0344(4)
H8C	0.2312	0.9168	0.6934	0.041*
H8B	0.1237	0.8344	0.6620	0.041*

found C 44.42, H 5.10, O 36.91%. **IR spectrum** (cm^{−1}, KBr pellet): 3284(s), 1611(m), 1529(s), 1446(w), 1383(s), 1203(w), 1047(s), 987(w), 765(s), 501(m).

Experimental details

H-atoms attached to carbon were refined using a riding model implemented in the SHELXL program [2]. H-atoms attached to oxygen were freely refined subject to distance restraints and *U*_{iso} constraints, with the exception of hydroxy H8A, whose coordinates were constrained to ride on the parent atom.

The structure was refined in space group P2₁/c rather than in P2₁/n, even though the latter leads to a beta angle closer to 90°, to enable a comparison with the isomorphous structure of the Ni(II) analogue [4]. Strong pseudo-translational symmetry is observed in the structure for 92% of non-H atoms.

Comment

Cobalt complexes play an important role in the development of functional materials because of their catalytic properties and their excellent optical and magnetic properties. Studies have shown that cobalt complexes have active performance in the fields of catalytic chemistry, biochemistry, and analytical chemistry [4–6]. As a rigid ligand, aromatic carboxylic acids facilitate the construction of specific morphological and functional coordination compounds [7]. The study of aromatic carboxylic acid cobalt complexes is of great significance in the field of material chemistry.

In the crystal structure of the title compound there are two halves of the titled molecules existing in the asymmetric unit with the metal cations sitting on inversion centres. Each cation adopts a distorted octahedral coordination configuration, and is coordinated to four water molecules and to two carboxylate oxygen atoms, with O4(water) and its symmetry equivalent [O10(water) and its symmetry equivalent for the second molecule in the asymmetric unit] occupying the axial positions. In the complex, the Co1–O2 bond length is 2.0789(10) Å, the Co1–O5 bond length is 2.1926(11) Å, the Co1–O4 bond length is 2.1196(10) Å, and the Co2–O10 bond length is 2.0896(10) Å, the bond length of Co2–O7 is 2.0911(10) Å, and the length of the Co2–O9 bond is 2.1596(11) Å, which is longer than that reported in [8–10] and in the isomorphous Ni(II) analogue [11]. The O2–Co1–O2 bond angle is 180.0° as defined by symmetry, the O2–Co1–O5 bond angle is 89.83(4)°, the O2–Co1–O4 bond angle is 90.44(4)°, and the O5–Co1–O4 bond angle is 90.76(4)°, O10–Co2–O7 bond angle is 89.83(4)°, O10–Co2–O9 bond angle is 91.38(4)°, O7–Co2–O9 bond angle is 89.50(4)°, the remaining bond lengths and angles are all within the normal range. The hydroxy groups and the four coordinated water molecules act as the hydrogen-bonding donors and acceptors giving rise to a complex three-dimensional hydrogen-bonded network is formed.

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