Crystal structure of bis[aqua-μ-(2*R*,3*R*)-tartratocobalt(II)] trihydrate, $[\{Co(C_4H_4O_6)(H_2O)\}_2] \cdot 3H_2O$

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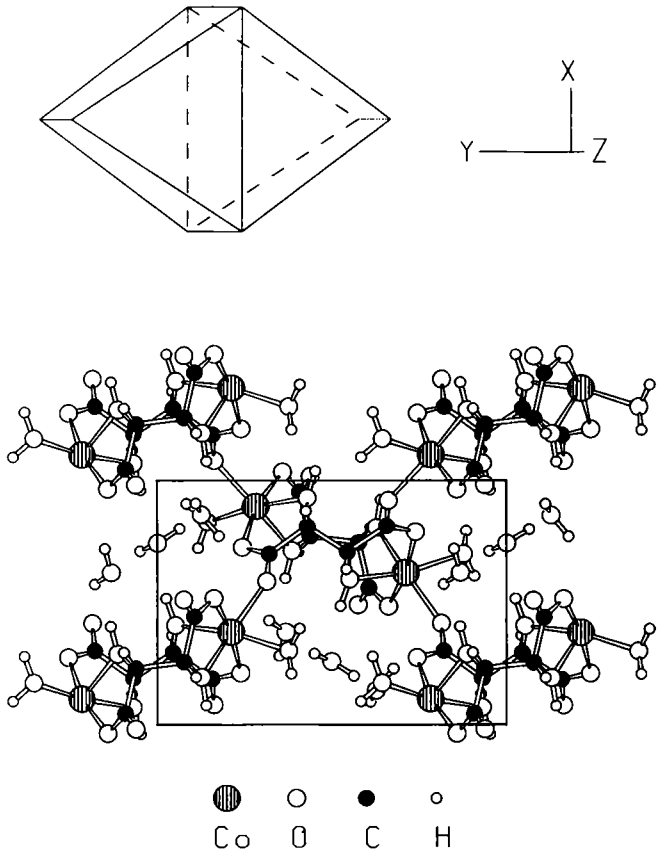

Source of material: Prismatic crystals of the title compound were obtained from a super saturated solution standing on the shelf for two weeks. The solution was prepared by dissolving 5 mmol $Co(NO_3)_2 \cdot 6H_2O$ (1.455g) and 5 mmol $KNaC_4H_4O_6 \cdot 4H_2O$ (1.411g) in 100 ml water.

The Flack parameter is x = –0.01(1); further programs used : PLATON, POP, SCHAKAL (see ref. 3, 4, 5). The structure of the title compound is isotypic to that of the analogous Zn-compound (see ref. 1). Two crystals were measured in different orientations, about 17000 reflections from each. After scaling the data sets were combined. In the upper part of the figure the typical crystal form is shown. Well developed faces are 110, $\bar{1}10$, $\bar{1}\bar{1}0$, $1\bar{1}0$, 012, $0\bar{1}2$, $0\bar{1}\bar{2}$, $01\bar{2}$. The crystals are elongated in the *c*-direction. In the lower part of the figure a projection of a layer along [001] is shown. In this layer the molecules are connected by Co–O contacts. The numerous hydrogen bonds present in the structure are not shown. Co–O distances in the distorted octahedron are in the range 2.04 Å to 2.16 Å.

$C_8H_{18}Co_2O_{17}$, orthorhombic, $P2_12_12_1$ (No. 19), a =7.908(5) Å, b =11.189(6) Å, c =18.206(7) Å, V =1610.9 Å^3, Z =4, $R(F)$ =0.030, $R_w(F^2)$ =0.041.

Table 1. Parameters used for the X-ray data collection

Crystal:	violet, shape 0.08 x 0.09 x 0.34 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	21.6 cm^{-1}
Diffractometer:	Stoe IPDS
Scan mode:	150 exposures, $\Delta\varphi = 1.5°$
$T_{measurement}$:	298 K
$2\theta_{max}$:	56°
$N(hkl)_{unique}$:	3940
Criterion for I_o:	$I_o > 2\,\sigma(I_o)$
$N(param)_{refined}$:	288
Program:	SHELXL-93

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	4*a*	0.6340(4)	0.4568(3)	0.3201(2)	0.018(4)
H(2)	4*a*	0.8765(4)	0.5730(3)	0.3199(1)	0.018(4)
H(3)	4*a*	0.8056(4)	0.3891(3)	–0.0263(2)	0.018(4)
H(4)	4*a*	0.7053(5)	0.5949(3)	–0.0257(2)	0.018(4)
H(5)	4*a*	0.492(3)	0.472(5)	0.222(2)	0.093(5)
H(6)	4*a*	1.034(3)	0.552(5)	0.217(2)	0.093(5)
H(7)	4*a*	0.920(3)	0.363(4)	0.081(2)	0.093(5)
H(8)	4*a*	0.606(3)	0.634(5)	0.072(2)	0.093(5)
H(9)	4*a*	0.608(4)	0.090(4)	0.086(2)	0.093(5)
H(10)	4*a*	0.790(4)	0.124(4)	0.079(2)	0.093(5)
H(11)	4*a*	0.902(5)	0.917(3)	0.071(2)	0.093(5)
H(12)	4*a*	0.722(3)	0.879(4)	0.063(2)	0.093(5)
H(13)	4*a*	1.295(7)	0.536(3)	0.296(2)	0.093(5)
H(14)	4*a*	1.217(7)	0.427(2)	0.263(2)	0.093(5)
H(15)	4*a*	0.458(4)	0.674(4)	0.083(2)	0.093(5)
H(16)	4*a*	0.280(3)	0.634(4)	0.079(2)	0.093(5)
H(17)	4*a*	1.242(4)	0.335(4)	0.092(2)	0.093(5)
H(18)	4*a*	1.103(6)	0.388(4)	0.049(1)	0.093(5)

Table 3. Final 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)	4*a*	0.62206(5)	0.28770(4)	0.14925(2)	0.0172(3)	0.0130(2)	0.0145(2)	–0.0004(2)	–0.0007(2)	0.0003(2)
Co(2)	4*a*	0.89416(5)	0.71655(4)	0.13907(2)	0.0173(3)	0.0126(2)	0.0139(2)	–0.0005(2)	0.0008(2)	–0.0011(2)
C(1)	4*a*	0.8128(4)	0.3422(3)	0.2793(1)	0.020(2)	0.013(1)	0.014(1)	–0.002(1)	–0.001(1)	0.004(1)
C(2)	4*a*	0.7051(4)	0.4545(3)	0.2761(2)	0.015(2)	0.015(2)	0.009(1)	0.004(1)	–0.005(1)	0.000(1)
C(3)	4*a*	0.8113(4)	0.5688(3)	0.2742(1)	0.017(2)	0.018(2)	0.006(1)	–0.001(1)	0.003(1)	–0.001(1)
C(4)	4*a*	0.6975(4)	0.6786(3)	0.2703(2)	0.010(2)	0.013(2)	0.022(2)	–0.001(1)	–0.002(1)	–0.006(1)
C(5)	4*a*	0.5607(5)	0.3934(3)	0.0077(2)	0.022(2)	0.011(2)	0.014(2)	–0.002(1)	–0.004(1)	–0.001(1)
C(6)	4*a*	0.7441(4)	0.4232(3)	0.0154(2)	0.016(2)	0.013(2)	0.012(1)	–0.002(1)	0.002(1)	–0.001(1)
C(7)	4*a*	0.7693(5)	0.5607(3)	0.0153(2)	0.020(2)	0.018(2)	0.010(1)	–0.001(1)	0.001(1)	–0.001(1)
C(8)	4*a*	0.9528(4)	0.5908(3)	0.0056(2)	0.017(2)	0.010(1)	0.018(2)	–0.001(1)	0.004(1)	–0.001(1)
O(1)	4*a*	0.9184(3)	0.3368(2)	0.3306(1)	0.029(2)	0.024(1)	0.021(1)	0.010(1)	–0.012(1)	–0.0019(8)
O(2)	4*a*	0.7888(3)	0.2601(2)	0.2330(1)	0.037(2)	0.014(1)	0.024(1)	0.009(1)	–0.015(1)	–0.0048(9)
O(3)	4*a*	0.5955(3)	0.4480(2)	0.2126(1)	0.017(1)	0.018(1)	0.023(1)	0.004(1)	–0.009(1)	–0.0046(8)
O(4)	4*a*	0.9279(3)	0.5693(2)	0.2140(1)	0.014(1)	0.022(1)	0.018(1)	0.004(1)	0.007(1)	0.0011(8)
O(5)	4*a*	0.5865(3)	0.6859(2)	0.31847(9)	0.027(1)	0.023(1)	0.025(1)	0.010(1)	0.014(1)	0.0022(8)
O(6)	4*a*	0.7242(3)	0.7554(2)	0.2201(1)	0.032(1)	0.013(1)	0.019(1)	0.0063(9)	0.0092(9)	0.0024(8)
O(7)	4*a*	0.4897(3)	0.4238(2)	–0.0510(1)	0.024(2)	0.026(1)	0.025(1)	–0.007(1)	–0.010(1)	0.008(1)
O(8)	4*a*	0.4845(3)	0.3373(2)	0.0591(1)	0.014(1)	0.029(1)	0.021(1)	–0.005(1)	–0.0013(9)	0.0077(9)
O(9)	4*a*	0.8077(3)	0.3715(2)	0.0817(1)	0.016(1)	0.027(1)	0.018(1)	–0.002(1)	–0.006(1)	0.0107(9)
O(10)	4*a*	0.7095(3)	0.6125(2)	0.0824(1)	0.018(1)	0.022(1)	0.021(1)	–0.002(1)	0.004(1)	–0.0101(8)
O(11)	4*a*	1.0181(3)	0.5620(2)	–0.0543(1)	0.022(1)	0.029(1)	0.026(1)	–0.005(1)	0.008(1)	–0.008(1)
O(12)	4*a*	1.0347(3)	0.6408(2)	0.0569(1)	0.016(1)	0.024(1)	0.023(1)	0.000(1)	0.001(1)	–0.006(1)
O(1W)	4*a*	0.6981(3)	0.1226(2)	0.1098(1)	0.020(1)	0.024(1)	0.028(1)	–0.002(1)	0.009(1)	–0.010(1)
O(2W)	4*a*	0.8294(3)	0.8555(2)	0.0703(1)	0.026(2)	0.024(1)	0.040(1)	–0.001(1)	–0.011(1)	0.012(1)
O(3W)	4*a*	1.2583(4)	0.4997(2)	0.2526(1)	0.037(2)	0.022(1)	0.034(1)	–0.004(1)	0.002(1)	–0.003(1)
O(4W)	4*a*	0.3767(4)	0.6309(3)	0.1067(2)	0.025(2)	0.109(3)	0.053(1)	0.009(2)	–0.002(2)	–0.007(2)
O(5W)	4*a*	1.1411(4)	0.3728(3)	0.0964(2)	0.024(2)	0.076(2)	0.085(2)	0.009(2)	–0.013(2)	–0.004(2)

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