Rerefinement of *catena*-poly[[tetraaquacobalt(II)-μ-pyrazine] phthalate] in a lower-symmetry space group

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Key indicators

Single-crystal X-ray study
$T = 298$ K
Mean σ(C–C) = 0.004 Å
R factor = 0.023
wR factor = 0.074
Data-to-parameter ratio = 11.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The polymeric title compound, $\{[Co(C_4H_4N_2)(H_2O)_4]\cdot(C_8H_4O_4)\}_n$, when refined in *Imm*2 from the diffraction intensities originally used for refinement in *Immm* [Yang, Li, Cao & Yao (2003). *Acta Cryst.* E**59**, m961–m963], is an ordered structure whose Co^{II} atom, pyrazine ligand and phthalate counter-ion lie on special positions. The metal atom and *N*-heterocycle have *mm*2 site symmetry and the dianion *m* site symmetry.

Comment

For diffraction measurements that cannot be unambiguously assigned to a particular space group, the structure should preferably be refined in a centrosymmetric space group, even though the structure is disordered in this higher-symmetry setting (Marsh, 1986). As such, the structure of [Co(H$_2$O)$_4$(C$_4$H$_4$N$_2$)](C$_8$H$_4$O$_4$), (I), was originally refined with disorder in *Immm*; a number of restraints were applied to the model, and the high residual index ($R_1 = 0.096$) was attributed to disorder and not to low quality of the measurements (Yang *et al.*, 2003).

(I)
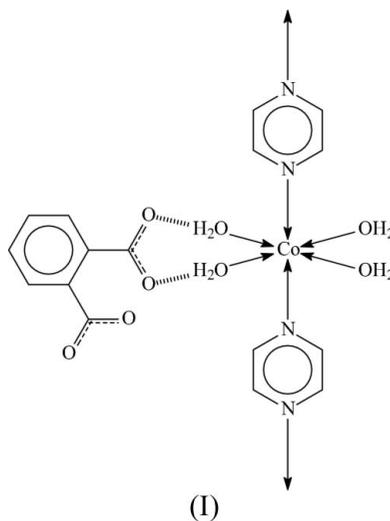
The zinc analogue crystallizes with similar cell dimensions; the authors of that study refined the structure in *Imm*2 because the compound exhibited second-harmonic generation (SHG) (Zhang *et al.*, 2005); the SHG test is one which is able to distinguish between centrosymmetric and non-centrosymmetric structures. Refinement of the cobalt compound in this space group led to a significantly improved R index ($R_1 = 0.023$). In this setting, the Co and pyrazine lie on special positions of *mm*2 site symmetry and the phthalate on a special position of *m* site symmetry (Fig. 1). The phthalate is ordered; the anions surround the linear polycationic chain and interact with the chains through hydrogen bonds (Fig. 2 and Table 2).
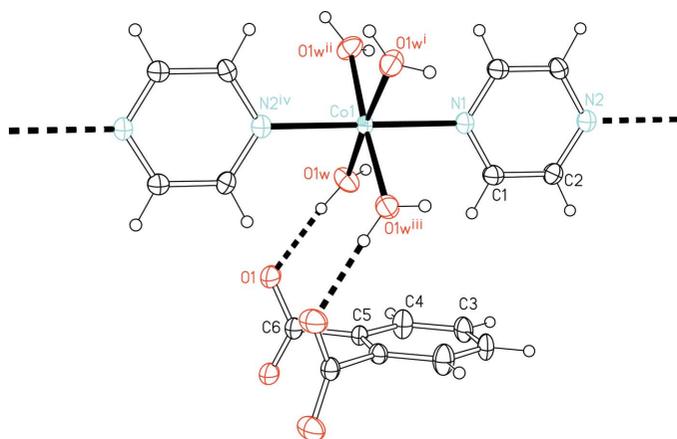
Figure 1
A plot of (I). Displacement ellipsoids are drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radii. [Symmetry codes: (i) $-x, 1-y, z$; (ii) $-x, y, z$; (iii) $x, 1-y, z$; (iv) $x, y, 1+z$.]

Experimental

Crystal data

$[Co(C_4H_4N_2)(H_2O)_4](C_8H_4O_4)$	Mo $K\alpha$ radiation
$M_r = 375.20$	Cell parameters from 2933
Orthorhombic, $Imm2$	reflections
$a = 9.4033 (6)$ Å	$\theta = 2.9–28.4°$
$b = 10.2886 (7)$ Å	$\mu = 1.28$ mm^{-1}
$c = 7.1791 (5)$ Å	$T = 298 (2)$ K
$V = 694.55 (8)$ Å^3	Prism, orange
$Z = 2$	$0.45 \times 0.28 \times 0.27$ mm
$D_x = 1.794$ Mg m^{-3}	

Data collection

Bruker APEX area-detector diffractometer	871 independent reflections
	871 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.018$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$\theta_{max} = 27.5°$
	$h = -12 \rightarrow 11$
$T_{min} = 0.545$, $T_{max} = 0.723$	$k = -13 \rightarrow 13$
2928 measured reflections	$l = -9 \rightarrow 9$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0542P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.023$	$+ 0.0753P]$
$wR(F^2) = 0.074$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.25$	$(\Delta/\sigma)_{max} = 0.001$
871 reflections	$\Delta\rho_{max} = 0.35$ e Å^{-3}
75 parameters	$\Delta\rho_{min} = -0.51$ e Å^{-3}
All H-atom parameters refined	Absolute structure: Flack (1983), 391 Friedel pairs
	Flack parameter: 0.02 (1)

Table 1
Selected geometric parameters (Å, °).

Co1—O1w	2.051 (1)	Co1—N2^{iv}	2.188 (4)
Co1—N1	2.194 (4)		
O1w—Co1—O1w^{i}	176.3 (1)	O1w—Co1—N1	88.1 (1)
O1w—Co1—O1w^{ii}	87.9 (1)	O1w—Co1—N2^{iv}	91.9 (1)
O1w—Co1—O1w^{iii}	92.0 (1)		

Symmetry codes: (i) $-x, -y+1, z$; (ii) $-x, y, z$; (iii) $x, -y+1, z$; (iv) $x, y, z+1$.

Figure 2
A plot depicting the hydrogen bonds (dashed lines).

Table 2
Hydrogen-bond geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O1w—H1w1$\cdots$O1	0.84 (1)	1.87 (1)	2.709 (2)	174 (4)
O1w—H1w2$\cdots$O1^{v}	0.83 (1)	1.91 (1)	2.736 (2)	178 (3)

Symmetry codes: (v) $-x+\frac{1}{2}, -y+\frac{1}{2}, z-\frac{1}{2}$.

The C-bound H atoms were generated geometrically (C—H = 0.93 Å) and were treated as riding, with U_{iso}(H) parameters set at $1.2U_{eq}$(C). The two O-bound H atoms were refined with a distance restraint of O—H = 0.85 (1) Å; the U_{iso}(H) parameters were freely refined.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; method used to solve structure: atomic coordinates taken from the Zn analogue; program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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