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catena-Poly[[bis(pyridine-κN)cobalt(II)]-μ-oxalato] 0.25-hydrate]

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Key indicators

Single-crystal X-ray study
T = 110 K
Mean σ(C–C) = 0.003 Å
H-atom completeness 96%
Disorder in solvent or counterion
R factor = 0.039
wR factor = 0.115
Data-to-parameter ratio = 15.8

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

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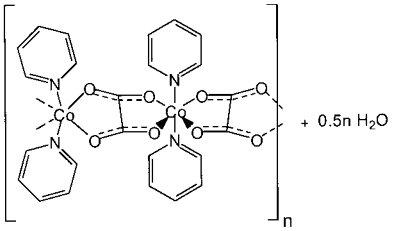
The structure of the title compound, $\{[Co(C_2O_4)(C_5H_5N)_2]\cdot 0.25H_2O\}_n$, has been determined at *ca* 110 K with relatively high precision, revealing the polymeric aggregation mode of this species.

Comment

Oxalate-bridged polynuclear metal complexes have been the focus of intensive research due to their interesting magnetic properties. The latter are highly dependent on the nature of the metal ion, the peripheral ligand and/or the counter-ion (Castillo *et al.*, 2003, and references therein). The ability of the oxalate moiety to connect to metal ions as a bis-bidentate bridging ligand enables the formation of diverse 'supramolecular' polymeric assemblies with different dimensionalities (Kitagawa *et al.*, 1995; Decurtins *et al.*, 1994). Indeed, the coordination pattern has pronounced influence on the magnetic behavior of such materials. Although a large number of two- and three-dimensional networks consisting of metal-bridged oxalate complexes are known, only a small number of homometallic one-dimensional systems have been structurally characterized (Castillo *et al.*, 2001, and references therein). We report here a precise crystallographic characterization of the title compound, (I), in which pyridine ligands occupy the fifth and sixth coordination sites around the octahedral cobalt(II) ion (Fig. 1). The zigzag-type polymerization pattern of (I), illustrated in Fig. 2, resembles that observed in closely related compounds [Castillo *et al.* (2001); wherein the 3-aminopyridine analog of (I) has been determined with low precision]. Each chain propagates and lies on consecutive inversion centers, along the *a* axis The crystal packing of the polymeric entities, parallel to one another, is shown in Fig. 3. Noteworthy are the elongated C—C bonds in the oxalate groups (Table 1), a result of their geometric requirement to occupy two coordination sites of the central cobalt ion.



(I)

Experimental

Single crystals of the title compound, (I), were obtained by reacting solvothermally cobalt acetate (6 mg), oxalic acid (6 mg), isopropyl

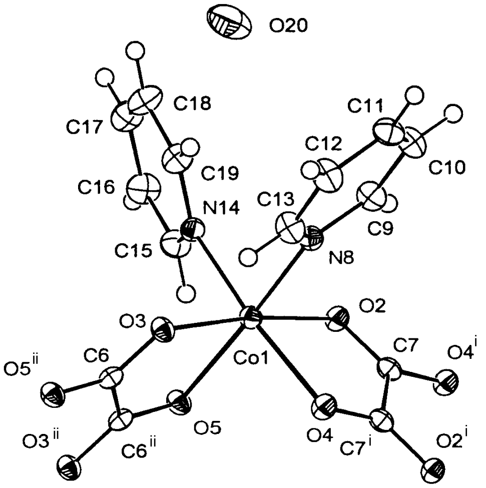


Figure 1
The molecular structure of the asymmetric unit of (I), showing the atom-labeling scheme. To indicate bonding continuity, several atoms of adjacent units related by inversion at the centers of the oxalate C—C bonds are also displayed. Displacement ellipsoids are drawn at the 50% probability level. Water H atoms were not located. [Symmetry codes: (i) $-x, -y, -z$; (ii) $1 - x, -y, -z$.]

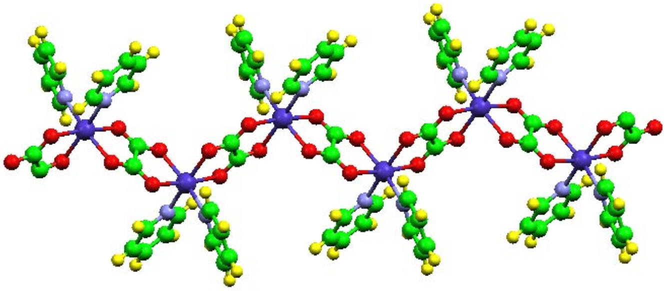


Figure 2
View of the one-dimensional polymeric arrays in (I) (ball-and-stick representation). Color code: C green, H yellow, Co blue, N light blue and O red.

alcohol (1 ml), potassium hydroxide (0.1 N, 0.5 ml) and pyridine (0.5 ml) in a sealed reactor at 400 K for one week.

Crystal data

$[Co(C_2O_4)(C_5H_5N)_2]\cdot 0.25H_2O$
$M_r = 309.65$
Monoclinic, $P2_1/n$
$a = 9.4320\ (3)$ Å
$b = 9.1810\ (4)$ Å
$c = 14.9370\ (5)$ Å
$\beta = 94.369\ (2)^\circ$
$V = 1289.71\ (8)$ Å^3
$Z = 4$
$D_x = 1.595$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2213 reflections
$\theta = 2.5$–27.5°
$\mu = 1.34$ mm^{-1}
$T = 110\ (2)$ K
Rod, pink
0.35 × 0.15 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (Blessing, 1995)
$T_{min} = 0.652$, $T_{max} = 0.878$
9965 measured reflections
2884 independent reflections
2213 reflections with $I > 2\sigma(I)$
$R_{int} = 0.046$
$\theta_{max} = 27.5^\circ$
$h = -12 \rightarrow 12$
$k = -110 \rightarrow 11$
$l = -19 \rightarrow 19$

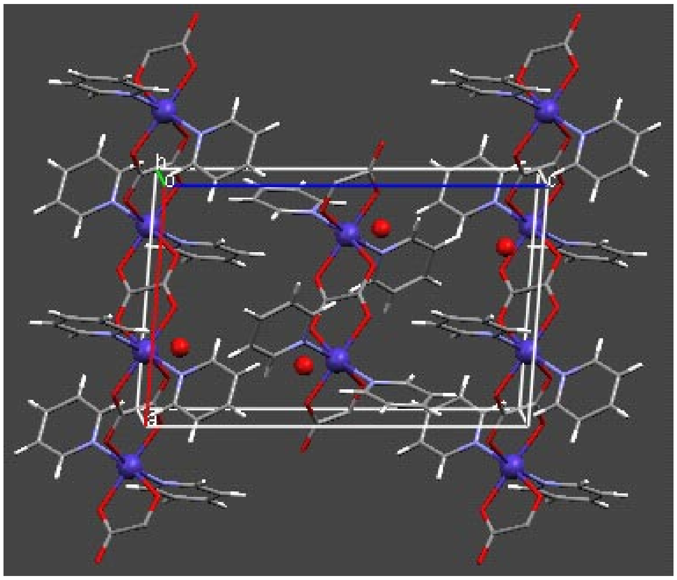


Figure 3
View of the crystal structure, approximately down *b* (*c* is horizontal; capped-stick representation). The cobalt ions and water O atoms are represented by small spheres. The polymeric species propagate in the crystal structure parallel to *a*. Color code: C gray, H white, Co blue, N light blue and O red.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.115$
$S = 1.02$
2884 reflections
182 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0729P)^2 + 0.0368P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.65$ e Å^{-3}
$\Delta\rho_{min} = -0.77$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Co1—O3	2.0721 (15)	O3—C6	1.255 (3)
Co1—O2	2.0903 (14)	O4—C7^i	1.251 (2)
Co1—O4	2.1209 (15)	O5—C6^{ii}	1.247 (2)
Co1—O5	2.1350 (14)	C6—O5^{ii}	1.248 (2)
Co1—N14	2.1375 (18)	C6—C6^{ii}	1.561 (4)
Co1—N8	2.1400 (18)	C7—O4^i	1.251 (2)
O2—C7	1.252 (2)	C7—C7^i	1.566 (4)
O3—Co1—O2	173.27 (6)	O4—Co1—N14	166.50 (6)
O3—Co1—O4	96.84 (6)	O5—Co1—N14	90.25 (7)
O2—Co1—O4	79.32 (5)	O3—Co1—N8	90.33 (6)
O3—Co1—O5	79.47 (5)	O2—Co1—N8	95.13 (6)
O2—Co1—O5	94.81 (5)	O4—Co1—N8	89.56 (7)
O4—Co1—O5	88.19 (6)	O5—Co1—N8	169.22 (6)
O3—Co1—N14	96.06 (6)	N14—Co1—N8	94.35 (8)
O2—Co1—N14	87.46 (6)		

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

The H atoms of the pyridine rings were positioned geometrically (C—H = 0.95 Å) and refined using a riding model, with fixed displacement parameters [$U_{iso}(H) = 1.2U_{eq}(C)$]. The H atoms of the partial water molecule could not be located.

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *DENZO*; program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*III (Burnett & Johnson, 1996) and

MERCURY (Bruno *et al.*, 2002); software used to prepare material for publication: *SHELXL*97.

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