Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Tetraaquabis(6-chloropyridine-3-carboxylato-κO)cobalt(II) tetrahydrate

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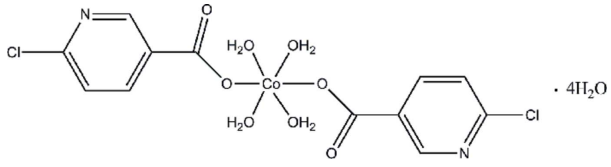
Received 7 October 2012; accepted 17 October 2012

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.029; wR factor = 0.085; data-to-parameter ratio = 17.9.

In the title compound, $[Co(C_6H_3ClNO_2)_2(H_2O)_4]\cdot 4H_2O$, the Co^{II} cation is located on an inversion center and is coordinated by four water molecules and two 6-chloropyridine-3-carboxylate anions in a slightly distorted octahedral geometry. In the crystal, complex molecules and lattice water molecules are linked by O—H···O and O—H···N hydrogen bonds into a three-dimensional network.

Related literature

For background and related structures, see: Long *et al.* (2007); Li *et al.* (2006).



Experimental

Crystal data

$[Co(C_6H_3ClNO_2)_2(H_2O)_4]\cdot 4H_2O$
M_r = 516.15
Triclinic, $P\overline{1}$
a = 7.0314 (14) Å
b = 7.3569 (15) Å
c = 11.564 (2) Å
α = 86.41 (3)°
β = 77.75 (3)°
γ = 64.80 (3)°
V = 528.7 (2) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 1.13 mm^{-1}
T = 293 K
0.42 × 0.37 × 0.18 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.754, T_{max} = 0.862
5250 measured reflections
2394 independent reflections
2094 reflections with $I > 2\sigma(I)$
R_{int} = 0.065

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.085
S = 1.16
2394 reflections
134 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
OW1—H1*WA*···O1	0.86	1.81	2.644 (2)	162
OW1—H1*WB*···OW4	0.82	2.01	2.818 (2)	173
OW2—H2*WA*···OW4^{i}	0.82	2.07	2.857 (2)	161
OW2—H2*WB*···OW3^{i}	0.89	1.92	2.791 (2)	167
OW3—H3*WA*···N^{ii}	0.85	2.00	2.842 (2)	172
OW3—H3*WB*···O1	0.81	1.95	2.763 (2)	176
OW4—H4*WA*···OW1^{iii}	0.85	2.23	2.948 (2)	141
OW4—H4*WB*···OW3^{i}	0.86	1.94	2.763 (2)	158

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $-x+1, -y+1, -z$; (iii) $-x+2, -y, -z+1$.

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

This work was supported by the National Natural Science Foundation (No.21207117), the Zhejiang Provincial Municipal Science and Technology Project (2008 C12055) and the Natural Science Foundation of Zhejiang Province (No. LY12B02013).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5631).

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supporting information

Acta Cryst. (2012). E**68**, m1394 [doi:10.1107/S160053681204319X]

Tetraaquabis(6-chloropyridine-3-carboxylato-*κO*)cobalt(II) tetrahydrate

Qiao-Hua Xia, Yue Zhang, Li Liu, Lin-Fang Shi and Bing Li

S1. Comment

In the crystal of the title compound, the Co(II) ion adopts a slightly distorted octahedral geometry and is located on a crystallographic inversion center. Four oxygen atoms from four coordination water molecules define the equatorial plane, while two oxygen atoms of two 6-chloro-3-carboxylate ligands occupy the axial sites (Figure 1). The Co—O bond lengths are in the range of 2.0723 (14)- 2.1162 (15) Å. The O—Co—O bond angles are 87.98 (6)–92.02 (6)° for the formally *cis* pairs of ligating atoms. The 6-chloropyridine-3-carboxylate carboxylate ligands are bound to the Co(II) ion in a monodentate mode through a carboxylate O atom. The three-dimensional supramolecular structure is formed by hydrogen bonds between six strong inter-molecular O—H···O and O—H···N hydrogen-bonding interactions and by additional intra-molecular O—H···O hydrogen bonds (Figure 2).

S2. Experimental

All commercially obtained reagent grade chemicals were used without further purication. A mixture of cobalt acetate tetrahydrate (0.4062 g) and 6-chloronicotinic acid (0.1310 g) were added into 20 ml water with 8 drops of 0.1 mol/*L* sodium hydroxide solution, and then stirred for 30 min. Finally, 5 ml 95% ethanol carefully layered above-mentioned solution in glass tube. After 1 day large pink platelet of the title compound suitable for X-ray diffraction were obtained.

S3. Refinement

H atoms bonded to C atoms were introduced in calculated positions and refined using a riding model [C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C) for aromatic H atoms. H atoms belonging to water molecules were found in difference Fourier map and refined isotropically.

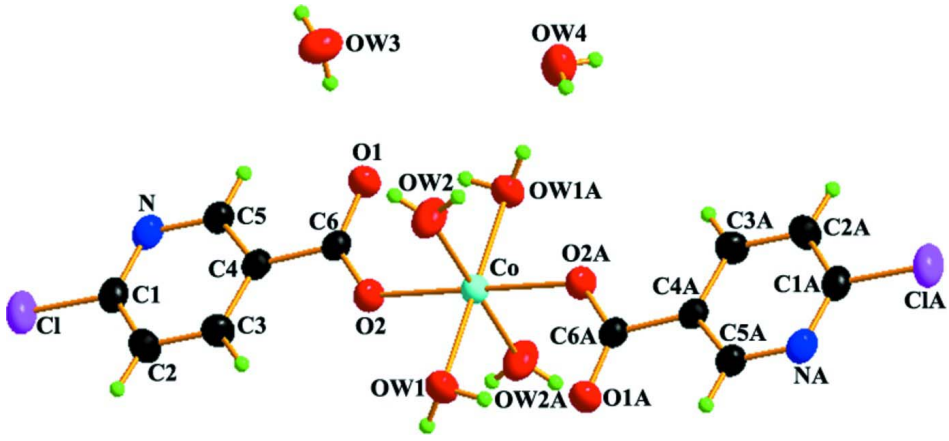

Figure 1

The molecular structure of the title complex, with 50% probability displacement ellipsoids for non-H atoms. [Symmetry codes: (A) 1 - *x*, -*y*, 1 - *z*.]

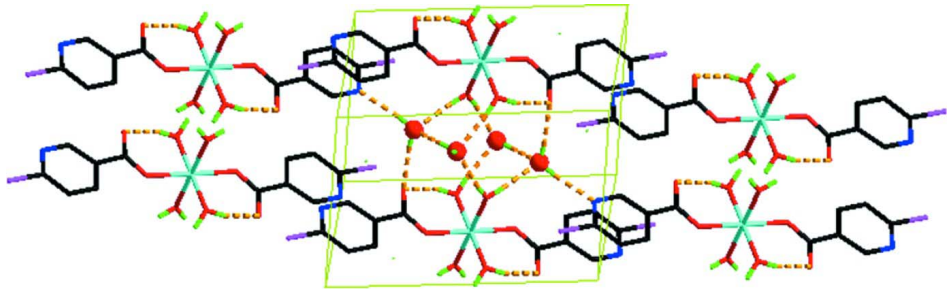

Figure 2

Crystal packing diagram for the title compound. All atoms are shown as isotropic spheres of arbitrary size. H atoms bonded to C atoms are omitted for clarity. The H-bonding interactions are shown as yellow dashed lines.

Tetraaquabis(6-chloropyridine-3-carboxylato-*κO*)cobalt(II) tetrahydrate

Crystal data

$[Co(C_6H_3ClNO_2)_2(H_2O)_4]\cdot 4H_2O$
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Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.0314\ (14)$ Å
$b = 7.3569\ (15)$ Å
$c = 11.564\ (2)$ Å
$\alpha = 86.41\ (3)°$
$\beta = 77.75\ (3)°$
$\gamma = 64.80\ (3)°$
$V = 528.7\ (2)$ Å^3
$Z = 1$
$F(000) = 265$
$D_x = 1.621$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2394 reflections
$\theta = 3.1–27.5°$
$\mu = 1.13$ mm^{-1}
$T = 293$ K
Platelet, pink
0.42 × 0.37 × 0.18 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.754$, $T_{max} = 0.862$
5250 measured reflections
2394 independent reflections

2094 reflections with $I > 2\sigma(I)$
$R_{int} = 0.065$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.1°$
$h = -9 \rightarrow 8$
$k = -9 \rightarrow 9$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.085$
$S = 1.16$
2394 reflections
134 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0248P)^2 + 0.0893P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.45$ e Å^{-3}
$\Delta\rho_{min} = -0.48$ e Å^{-3}
Extinction correction: *SHELXTL* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.356 (12)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Co	0.5000	0.0000	0.5000	0.02745 (15)
Cl	0.00669 (8)	0.29228 (8)	−0.14887 (5)	0.04646 (18)
O1	0.6093 (2)	0.1896 (2)	0.23682 (12)	0.0371 (3)
O2	0.3932 (2)	0.0600 (2)	0.34210 (12)	0.0362 (3)
OW1	0.77755 (19)	0.0456 (2)	0.42492 (13)	0.0368 (3)
H1WA	0.7475	0.0959	0.3580	0.055*
H1WB	0.8053	0.1204	0.4606	0.055*
OW2	0.3261 (2)	0.3039 (2)	0.55709 (14)	0.0460 (4)
H2WA	0.2602	0.4021	0.5201	0.069*
H2WB	0.3531	0.3527	0.6166	0.069*
OW3	0.5269 (2)	0.5885 (2)	0.26396 (14)	0.0476 (4)
H3WA	0.5745	0.6384	0.2029	0.071*
H3WB	0.5535	0.4718	0.2526	0.071*
OW4	0.8332 (2)	0.3212 (2)	0.55760 (15)	0.0482 (4)
H4WB	0.7442	0.3244	0.6224	0.072*
H4WA	0.9634	0.2619	0.5653	0.072*
N	0.3076 (2)	0.2781 (2)	−0.04625 (14)	0.0331 (4)
C1	0.1321 (3)	0.2467 (3)	−0.02902 (17)	0.0320 (4)
C2	0.0458 (3)	0.1820 (3)	0.07433 (18)	0.0361 (4)
H2A	−0.0791	0.1638	0.0813	0.043*

C3	0.1507 (3)	0.1451 (3)	0.16737 (18)	0.0347 (4)
H3A	0.0989	0.0992	0.2384	0.042*
C4	0.3362 (3)	0.1776 (2)	0.15307 (16)	0.0275 (4)
C5	0.4056 (3)	0.2444 (3)	0.04594 (17)	0.0312 (4)
H5A	0.5279	0.2678	0.0366	0.037*
C6	0.4549 (3)	0.1400 (2)	0.25230 (16)	0.0277 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co	0.0297 (2)	0.0310 (2)	0.0229 (2)	−0.01422 (15)	−0.00582 (13)	0.00395 (15)
Cl	0.0562 (3)	0.0444 (3)	0.0377 (3)	−0.0138 (2)	−0.0237 (2)	0.0020 (3)
O1	0.0387 (6)	0.0463 (8)	0.0343 (8)	−0.0251 (6)	−0.0107 (6)	0.0093 (7)
O2	0.0438 (7)	0.0453 (7)	0.0270 (7)	−0.0250 (6)	−0.0123 (5)	0.0102 (6)
OW1	0.0376 (6)	0.0474 (8)	0.0324 (7)	−0.0245 (6)	−0.0085 (5)	0.0051 (6)
OW2	0.0636 (9)	0.0306 (7)	0.0398 (9)	−0.0127 (7)	−0.0177 (7)	−0.0007 (7)
OW3	0.0655 (9)	0.0427 (8)	0.0366 (8)	−0.0288 (7)	−0.0013 (7)	−0.0007 (7)
OW4	0.0412 (7)	0.0517 (9)	0.0476 (9)	−0.0152 (7)	−0.0116 (7)	0.0065 (8)
N	0.0375 (8)	0.0326 (8)	0.0254 (8)	−0.0124 (7)	−0.0047 (6)	0.0039 (7)
C1	0.0360 (8)	0.0247 (8)	0.0290 (10)	−0.0051 (7)	−0.0091 (7)	−0.0034 (8)
C2	0.0313 (8)	0.0437 (10)	0.0355 (11)	−0.0173 (8)	−0.0081 (8)	0.0013 (9)
C3	0.0342 (8)	0.0430 (10)	0.0288 (10)	−0.0201 (8)	−0.0027 (7)	0.0028 (9)
C4	0.0300 (8)	0.0241 (8)	0.0246 (9)	−0.0087 (7)	−0.0034 (7)	−0.0003 (7)
C5	0.0311 (8)	0.0331 (9)	0.0284 (9)	−0.0137 (7)	−0.0046 (7)	0.0036 (8)
C6	0.0312 (8)	0.0237 (8)	0.0252 (9)	−0.0090 (7)	−0.0048 (7)	0.0002 (7)

Geometric parameters (Å, º)

Co—O2^{i}	2.0717 (14)	OW3—H3WB	0.8101
Co—O2	2.0717 (14)	OW4—H4WB	0.8629
Co—OW2	2.1078 (15)	OW4—H4WA	0.8520
Co—OW2^{i}	2.1078 (15)	N—C1	1.322 (2)
Co—OW1	2.1157 (14)	N—C5	1.344 (2)
Co—OW1^{i}	2.1157 (14)	C1—C2	1.375 (3)
Cl—C1	1.7379 (19)	C2—C3	1.380 (3)
O1—C6	1.261 (2)	C2—H2A	0.9300
O2—C6	1.250 (2)	C3—C4	1.398 (3)
OW1—H1WA	0.8642	C3—H3A	0.9300
OW1—H1WB	0.8153	C4—C5	1.373 (3)
OW2—H2WA	0.8246	C4—C6	1.504 (2)
OW2—H2WB	0.8853	C5—H5A	0.9300
OW3—H3WA	0.8466		
O2^{i}—Co—O2	180.0	H3WA—OW3—H3WB	111.9
O2^{i}—Co—OW2	88.51 (6)	H4WB—OW4—H4WA	112.2
O2—Co—OW2	91.49 (6)	C1—N—C5	116.27 (18)
O2^{i}—Co—OW2^{i}	91.49 (6)	N—C1—C2	125.08 (17)
O2—Co—OW2^{i}	88.51 (6)	N—C1—Cl	115.68 (16)

OW2—Co—OW2^{i}	180.0	C2—C1—Cl	119.24 (14)
O2^{i}—Co—OW1	88.00 (6)	C1—C2—C3	117.79 (17)
O2—Co—OW1	92.00 (6)	C1—C2—H2A	121.1
OW2—Co—OW1	91.76 (7)	C3—C2—H2A	121.1
OW2^{i}—Co—OW1	88.24 (7)	C2—C3—C4	118.94 (19)
O2^{i}—Co—OW1^{i}	92.00 (6)	C2—C3—H3A	120.5
O2—Co—OW1^{i}	88.00 (6)	C4—C3—H3A	120.5
OW2—Co—OW1^{i}	88.24 (7)	C5—C4—C3	117.88 (17)
OW2^{i}—Co—OW1^{i}	91.76 (7)	C5—C4—C6	121.45 (15)
OW1—Co—OW1^{i}	180.00 (3)	C3—C4—C6	120.67 (18)
C6—O2—Co	128.85 (12)	N—C5—C4	124.03 (16)
Co—OW1—H1WA	100.8	N—C5—H5A	118.0
Co—OW1—H1WB	117.8	C4—C5—H5A	118.0
H1WA—OW1—H1WB	110.1	O2—C6—O1	125.95 (17)
Co—OW2—H2WA	129.2	O2—C6—C4	116.96 (15)
Co—OW2—H2WB	121.6	O1—C6—C4	117.09 (17)
H2WA—OW2—H2WB	105.8		

Symmetry code: (i) −*x*+1, −*y*, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O*W*1—H1*WA*···O1	0.86	1.81	2.644 (2)	162
O*W*1—H1*WB*···O*W*4	0.82	2.01	2.818 (2)	173
O*W*2—H2*WA*···O*W*4^{ii}	0.82	2.07	2.857 (2)	161
O*W*2—H2*WB*···O*W*3^{ii}	0.89	1.92	2.791 (2)	167
O*W*3—H3*WA*···N^{iii}	0.85	2.00	2.842 (2)	172
O*W*3—H3*WB*···O1	0.81	1.95	2.763 (2)	176
O*W*4—H4*WA*···O*W*1^{iv}	0.85	2.23	2.948 (2)	141
O*W*4—H4*WB*···O*W*3^{ii}	0.86	1.94	2.763 (2)	158

Symmetry codes: (ii) −*x*+1, −*y*+1, −*z*+1; (iii) −*x*+1, −*y*+1, −*z*; (iv) −*x*+2, −*y*, −*z*+1.