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Chlorobis(dimethylglyoximato)(isonicotinic acid)cobalt(III) monohydrate

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In the title compound, $[Co(C_4H_7N_2O_2)_2Cl(C_6H_5NO_2)]\cdot H_2O$, the two crystallographically distinct Co atoms are both coordinated by a chloride anion, an N-bonded isonicotinic acid ligand and two *N*,*N*-bidentate dimethylglyoximate ligands in a distorted octahedral geometry. A network of O—H···O and O—H···N hydrogen bonds helps to establish the crystal packing.

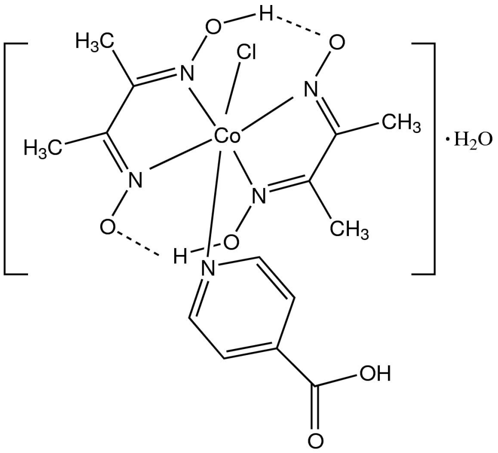
Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.008 Å
H-atom completeness 96%
R factor = 0.044
wR factor = 0.085
Data-to-parameter ratio = 12.1

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

Comment

In recent years, the crystal engineering of supramolecular architectures based on metal complexes has been rapidly expanding due to their ability to afford functional materials with potentially useful properties, as well as fascinating molecular structures (Lehn, 1995, 1999; Khlobystov *et al.*, 2001). Dimethylglyoximate (dmg) is a familiar ligand with excellent coordination capability to generate mono-, bi- or trinuclear complexes, which are commonly used as precursors for the formation of supramolecular architectures (Chaudhuri *et al.*, 1991; Kubiak *et al.*, 1995; Cervera *et al.*, 1997). Isonicotinic acid is also a good mono- or bidentate ligand for the construction of supramolecular complexes with versatile binding modes (Cova *et al.*, 2001; Sekiya & Nishikiori, 2001). However, crystal structures of complexes containing both these ligands have been less well documented thus far (Hashizume & Ohashi, 1998). In order to explore the coordination behaviour of these two ligands, we synthesized the title compound, a new cobalt(III) complex, (I), and determined its structure.



(I)

As shown in Fig. 1, the asymmetric unit of (I) consists of two Co-centred complexes and two uncoordinated water molecules. Both of the Co atoms are hexacoordinated with a slightly distorted octahedral geometry (Table 1). Each Co

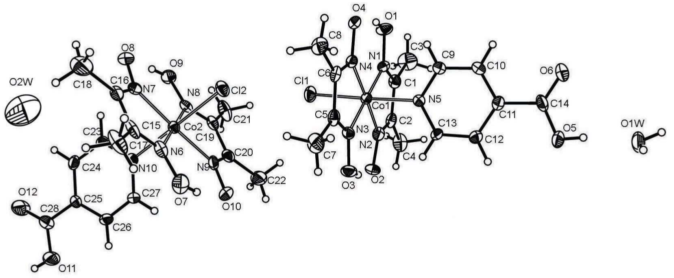


Figure 1
View of the asymmetric unit of (I), with displacement ellipsoids drawn at the 40% probability level (arbitrary spheres for the H atoms).

atom is coordinated by four N atoms from two bidentate dimethylglyoximate ligands, where the two dmg ligands are *trans* to each other. The Co—N(dmg) bonds range in length from 1.885 (5) to 1.931 (5) Å. A chloride anion and an N atom from the isonicotinic acid ligand occupy the other two coordination positions of each Co atom. Intramolecular hydrogen bonds between the two dmg ligands of each Co-centred complex (Fig. 1) enhance the stability of the structure. Intermolecular hydrogen bonds between water molecules and isonicotinic acid O atoms support the molecular packing (Table 2). Fig. 2 shows the packing arrangement of the complex.

Experimental

To a 95% ethanol solution (40 ml) containing [$CoCl_2(dmg)_2$]H [0.36 g, 1 mmol; synthesized according to the method of Heeg & Elder (1980)], isonicotinic acid (0.123 g, 1 mmol) was added. After being stirred for 15 min, the resulting dark-red solution was filtered and allowed to evaporate at room temperature. After 7 d, dark-red crystals of (I) suitable for X-ray analysis were obtained.

Crystal data

[$Co(C_4H_7N_2O_2)_2Cl(C_6H_5NO_2)$]·$H_2O$
$M_r = 465.74$
Monoclinic, $P2_1$
$a = 8.2904$ (6) Å
$b = 14.2195$ (8) Å
$c = 16.9550$ (7) Å
$\beta = 90.615$ (4)°
$V = 1998.6$ (2) Å^3
$Z = 4$
$D_x = 1.515$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2570 reflections
$\theta = 2.4$–22.4°
$\mu = 1.04$ mm^{-1}
$T = 295$ (2) K
Block, dark red
0.43 × 0.21 × 0.15 mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{\min} = 0.665$, $T_{\max} = 0.860$
11 170 measured reflections
6172 independent reflections
4394 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.052$
$\theta_{\max} = 25.0°$
$h = -9 \rightarrow 6$
$k = -14 \rightarrow 16$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.085$
$S = 0.90$
6172 reflections
511 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0281P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.017$
$\Delta\rho_{\max} = 0.51$ e Å^{-3}
$\Delta\rho_{\min} = -0.31$ e Å^{-3}
Absolute structure: Flack (1983), 2502 Friedel pairs
Flack parameter = −0.005 (16)

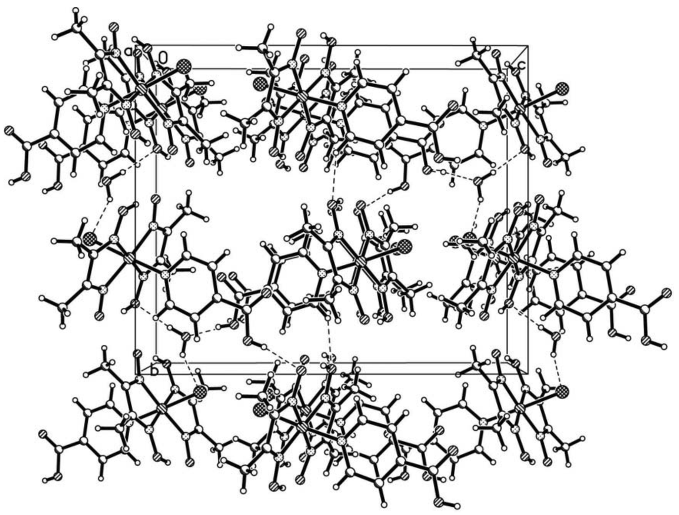


Figure 2
A view, down the *a* axis, of the packing in (I), with hydrogen bonds indicated by dashed lines.

Table 1
Selected geometric parameters (Å, °).

Co1—N4	1.887 (5)	Co2—N6	1.891 (5)
Co1—N3	1.895 (4)	Co2—N8	1.896 (5)
Co1—N1	1.901 (5)	Co2—N9	1.898 (4)
Co1—N2	1.914 (5)	Co2—N7	1.900 (5)
Co1—N5	1.962 (4)	Co2—N10	1.977 (4)
Co1—Cl1	2.2190 (15)	Co2—Cl2	2.2444 (15)
N4—Co1—N3	80.96 (18)	N6—Co2—N9	98.6 (2)
N4—Co1—N1	99.6 (2)	N8—Co2—N9	80.6 (2)
N3—Co1—N2	99.0 (2)	N6—Co2—N7	81.8 (2)
N1—Co1—N2	80.45 (19)	N8—Co2—N7	98.9 (2)
N3—Co1—N5	91.11 (18)	N6—Co2—N10	90.42 (18)
N1—Co1—N5	89.08 (18)	N8—Co2—N10	90.64 (17)
N5—Co1—Cl1	177.53 (13)	N10—Co2—Cl2	178.98 (14)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O4	0.82	1.72	2.515 (5)	161
O3—H3···O2	0.82	1.72	2.514 (5)	163
O5—H5···O1W	0.82	1.74	2.550 (5)	171
O7—H7···O10	0.82	1.72	2.511 (6)	162
O9—H9···O8	0.82	1.70	2.495 (6)	162
O11—H11···O4$^{\rm i}$	0.82	1.89	2.669 (5)	159
O1W—H1W···O7$^{\rm ii}$	0.85 (3)	1.95 (3)	2.797 (6)	172 (5)
O1W—H2W···Cl2$^{\rm iii}$	0.84 (2)	2.64 (5)	3.188 (6)	125 (4)

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

The H atoms attached to O1W were located in a difference Fourier map and refined with the distance restraint O—H = 0.85 (1) Å. The H atoms attached to O2W were not found in the difference map. The other H atoms were positioned geometrically (C—H = 0.93–0.96 Å and O—H = 0.82 Å) and refined as riding on their carrier atoms. All H atoms were assigned a fixed isotropic displacement parameter of $U_{\rm iso}$(H) = 0.08 Å^2.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2002); program(s) used to refine

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structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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