Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Chloridobis(dimethylglyoximato-κ^2N,N')(ethyl pyridine-4-carboxylate-κN)cobalt(III) chloroform monosolvate

Ning Wang,* Xuzhuo Sun, Dongjin Wan, Jing Chen and Bo Li

Henan University of Technology, School of Chemistry and Chemical Engineering, Zhengzhou 450001, People's Republic of China
Correspondence e-mail: ssdyok@yahoo.com.cn

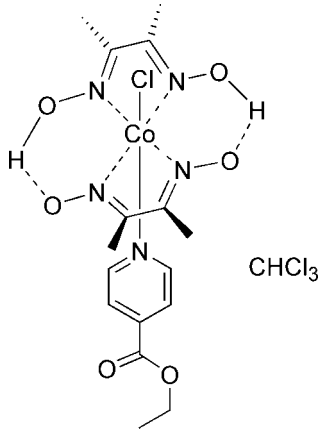
Received 5 January 2012; accepted 19 January 2012

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.006 Å; R factor = 0.051; wR factor = 0.158; data-to-parameter ratio = 14.7.

The title compound, $[Co(C_4H_7N_2O_2)_2Cl(C_8H_9NO_2)]\cdot CHCl_3$, was synthesized as a model complex of vitamin B_{12}. The Co^{III} cation displays an approximately octahedral coordination environment, being displaced by 0.0240 (15) Å from the mean plane of the four N atoms of the equatorial plane. The O—H distances in the dimethylglyoximate hydroxy groups are 0.89 (6) and 1.14 (6) Å; such long O—H bonds are very common in cobaloxime derivatives. Weak classical O—H···N and non-classical C—H···Cl hydrogen-bonding interactions further consolidate the crystal packing.

Related literature

For background on the chemistry of cobaloximes, see: Schrayzer (1968); Zangrando *et al.* (2003). For applications of cobaloximes in proton reduction, see: Razavet *et al.* (2005). For related structures, see: Bhuyan *et al.* (2007); Dutta *et al.* (2009); Mandal & Gupta (2005, 2007); William *et al.* (1973). For NMR research on O—H···O bridges, see: Bakac & Espenson (1984). For deprotonation of O—H···O bridges by $BF_3\cdot Et_2O$, see: Magnuson & Weber (1974).



Experimental

Crystal data

$[Co(C_4H_7N_2O_2)_2Cl(C_8H_9NO_2)]\cdot CHCl_3$
M_r = 595.14
Orthorhombic, *Pbca*
a = 10.053 (3) Å
b = 22.357 (7) Å
c = 23.099 (8) Å
V = 5192 (3) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 1.11 mm^{-1}
T = 293 K
0.29 × 0.14 × 0.06 mm

Data collection

Bruker APEXII area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2007)
T_{min} = 0.829, T_{max} = 0.935
24393 measured reflections
4558 independent reflections
3483 reflections with $I > 2\sigma(I)$
R_{int} = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.158
S = 1.08
4558 reflections
310 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 1.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.78 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O4	1.14 (6)	1.37 (6)	2.495 (4)	168 (5)
O2—H2···N4	1.14 (6)	2.10 (6)	3.004 (4)	133 (4)
O1—H1···O3	0.89 (6)	1.60 (6)	2.486 (4)	177 (6)
O1—H1···N3	0.89 (6)	2.25 (6)	3.000 (4)	142 (5)
C17—H17A···Cl1	0.98	2.49	3.437 (6)	163
C6—H6C···Cl1^{i}	0.96	2.79	3.675 (5)	153

Symmetry code: (i) $-x, -y, -z+1$.

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT-Plus* (Bruker, 2007); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

We are grateful to the Chinese National Natural Science Foundation (grant No. 21101057), the Science Foundation of the Education Department of Henan Province (No. 2011B150006) and the Doctoral Fund of Henan University of Technology (No. 2009BS053).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZL2444).

References

Bakac, A. & Espenson, J. H. (1984). *J. Am. Chem. Soc.* **106**, 5197–5202.
Bhuyan, M., Laskar, M., Mandal, D. & Gupta, B. D. (2007). *Organometallics*, **26**, 3559–3567.
Bruker (2007). *SADABS*, *SAINT-Plus* and *APEX2*. Bruker AXS Inc., Madison, Wisconsin, USA.
Dutta, G., Kumar, K. & Gupta, B. D. (2009). *Organometallics*, **28**, 3485–3491.
Magnuson, V. E. & Weber, J. H. (1974). *J. Organomet. Chem.* **74**, 135–141.
Mandal, D. & Gupta, B. D. (2005). *Organometallics*, **24**, 1501–1510.
Mandal, D. & Gupta, B. D. (2007). *Organometallics*, **26**, 658–670.
Razavet, M., Artero, V. & Fontecave, M. (2005). *Inorg. Chem.* **44**, 4786–4795.
Schrayzer, G. N. (1968). *Acc. Chem. Res.* **1**, 97–103.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

William, C. T., Robert, C. S., Leon, A. E. & Luigi, G. M. (1973). *Inorg. Chem.* **10**, 1564–1570.

Zangrando, E., Trani, M., Stabon, E., Carfagna, C., Milani, B. & Mestroni, G. (2003). *Eur. J. Inorg. Chem.* pp. 2683–2692.

supplementary materials

Acta Cryst. (2012). E**68**, m204-m205 [**doi:10.1107/S1600536812002449**]

Chloridobis(dimethylglyoximato-κ^2N,N')(ethyl pyridine-4-carboxylate-κN)cobalt(III) chloroform monosolvate

N. Wang, X. Sun, D. Wan, J. Chen and B. Li

Comment

The cobaloximes have extensively been used to mimic the vitamin B_{12} coenzyme (Schrayzer, 1968). Recently they also are used to catalyze proton reduction as a functional model of hydrogenase (Razavet *et al.*, 2005). The title compound, $[Co(dmgH)_2(4\text{-}(COOEt)C_5H_4N)Cl]$, was prepared as previously described (William *et al.*, 1973). The cobalt atom is coordinated in a distorted-octahedral geometry by four nitrogen atoms of the pseudomacrocyclic $[(dmgH)_2]^{2-}$ ligand in the equatorial plane and by one chlorine atom and a nitrogen atom of ethyl isonicotinate, respectively, in mutually *trans* positions (N5—Co—Cl1 = 179.33° (10)). The cobalt atom which is linked to four nitrogen atoms belonging to the equatorial plane, displays an approximately octahedral coordination. The mean Co—N bond length is 1.898 Å (3). The mean O—O distance between neighboring dimethylglyoximato oxygen atoms is 2.491 Å (5). These ligands form strong O—H···O bridges with each other which is very common in cobaloxime derivatives. The presence of the O—H···O bridging moieties in cobaloxime derivatives ensures coplanarity of the two ligand molecules and promotes the stability of the cobaloxime molecule (Zangrando *et al.*, 2003). The existence of O—H···O bridging is supported by the NMR data and further substantiated by their chemical behavior with $BF_3.Et_2O$ in readily forming an O—BF_2—O system by deprotonation of the O—H···O bridge (Bakac & Espenson, 1984; Magnuson & Weber, 1974). The distance between O2 and H2 is 1.14 (6) Å indicating a strongly hydrogen bonded nearly symmetric O—H···O system (Bhuyan *et al.*, 2007; Dutta *et al.*, 2009; Mandal & Gupta, 2005, 2007). The Co atom is 0.0240 Å (15) out of the mean plane of the four nitrogen atoms. The plane of the four nitrogen atoms is practically planar.

Experimental

$Co(dmgH)(dmgH_2)Cl$, (3.6 g, 0.01 mol) and ethyl isonicotinate (3.0 g, 0.02 mol) were added to chloroform (90 ml) (William *et al.*,1973). The suspension was stirred for 20 minutes. Then water (30 ml) was added to the flask and the mixture was vigorously stirred for 2 h. The aqueous layer was discarded and the chloroform layer filtered and extracted with water until the washings were nearly colorless. The solution was reduced in volume and the product precipitated by addition of ethanol (95%); yield 69%. Brown single crystals of $[Co(dmgH)_2(4\text{-}(COOEt)C_5H_4N)Cl]$ were grown from a $CHCl_3$/ethyl acetate solution (v:v = 1:1). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.45 (d, 6.4 Hz, 2 H, *o*-H_{py}), 7.75 (d, 6 Hz, 2 H, *m*-H_{py}), 4.38 (q, 7.2 Hz, 2 H, CH_2), 2.30 (s, 12 H, N=CCH_3), 1.35 (t, 7.0 Hz, 3 H, CH_3).

Refinement

H1 and H2 were located in difference Fourier maps and their positions and displacement parameters were fully rerfined. All other hydrogen atoms were placed in calculated positions and refined as riding with C—H = 0.93 Å (CH) and 0.97 Å (CH_3), and with U_{iso}(H) = 1.5Ueq(C) for the methyl group and U_{iso}(H) = 1.2U_{eq}(C) for all others.

Figures

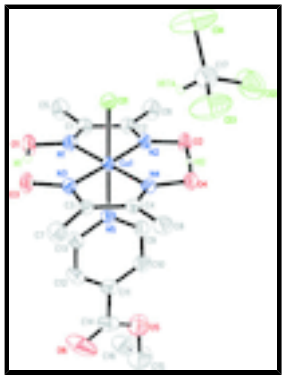


Fig. 1. The molecular structure of the title compound. Displacement ellipsoids are drawn at the 30% probability level.

Chloridobis(dimethylglyoximato-κ^2*N*,*N*')(ethyl pyridine-4-carboxylate-κ*N*)cobalt(III) chloroform monosolvate

Crystal data

$[Co(C_4H_7N_2O_2)_2Cl(C_8H_9NO_2)]\cdot CHCl_3$	$F(000) = 2432$
$M_r = 595.14$	$D_x = 1.523$ Mg m^{-3}
Orthorhombic, *Pbca*	Mo *K*α radiation, λ = 0.71073 Å
Hall symbol: -P 2ac 2ab	Cell parameters from 5509 reflections
$a = 10.053$ (3) Å	θ = 2.4–24.8°
$b = 22.357$ (7) Å	μ = 1.11 mm^{-1}
$c = 23.099$ (8) Å	$T = 293$ K
$V = 5192$ (3) Å^3	Needle, brown
$Z = 8$	0.29 × 0.14 × 0.06 mm

Data collection

Bruker APEXII area-detector diffractometer	4558 independent reflections
Radiation source: fine-focus sealed tube	3483 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.039$
phi and ω scans	$\theta_{max} = 25.0°$, $\theta_{min} = 2.0°$
Absorption correction: multi-scan (*SADABS*; Bruker, 2007)	$h = -11\rightarrow9$
$T_{min} = 0.829$, $T_{max} = 0.935$	$k = -26\rightarrow26$
24393 measured reflections	$l = -27\rightarrow24$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.051$	Hydrogen site location: inferred from neighbouring sites

$wR(F^2) = 0.158$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.08$	$w = 1/[\sigma^2(F_o^2) + (0.0892P)^2 + 4.4304P]$ where $P = (F_o^2 + 2F_c^2)/3$
4558 reflections	$(\Delta/\sigma)_{max} = 0.001$
310 parameters	$\Delta\rho_{max} = 1.25$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.78$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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}
Co1	0.23591 (5)	0.00646 (2)	0.63484 (2)	0.03477 (19)
Cl1	0.08443 (9)	0.07824 (5)	0.62035 (4)	0.0473 (3)
N2	0.2686 (3)	0.00560 (14)	0.55381 (14)	0.0387 (7)
N4	0.3608 (3)	0.06594 (13)	0.65679 (12)	0.0364 (7)
O2	0.3577 (3)	0.04186 (12)	0.52883 (11)	0.0489 (7)
N3	0.1977 (3)	0.00927 (14)	0.71495 (13)	0.0407 (7)
N5	0.3706 (3)	−0.05592 (13)	0.64728 (13)	0.0406 (7)
O4	0.4372 (3)	0.09421 (12)	0.61771 (11)	0.0473 (7)
O3	0.1032 (3)	−0.02516 (14)	0.73944 (11)	0.0558 (7)
C4	0.3647 (4)	0.07978 (16)	0.71110 (15)	0.0384 (8)
N1	0.1090 (3)	−0.05257 (15)	0.61321 (13)	0.0443 (8)
C2	0.2011 (4)	−0.03273 (18)	0.52384 (15)	0.0438 (9)
O1	0.0261 (3)	−0.07852 (15)	0.65120 (13)	0.0617 (9)
C3	0.2708 (3)	0.04442 (17)	0.74594 (16)	0.0406 (9)
C1	0.1055 (4)	−0.06658 (18)	0.55898 (16)	0.0478 (10)
C6	0.2193 (5)	−0.0395 (2)	0.45984 (18)	0.0661 (13)
H6A	0.3024	−0.0591	0.4522	0.099*
H6B	0.2193	−0.0008	0.4420	0.099*
H6C	0.1478	−0.0630	0.4443	0.099*
C7	0.2605 (4)	0.0476 (2)	0.81032 (17)	0.0580 (12)
H7A	0.1723	0.0599	0.8210	0.087*
H7B	0.3238	0.0760	0.8248	0.087*
H7C	0.2787	0.0089	0.8266	0.087*
C9	0.4808 (4)	−0.05867 (19)	0.61441 (18)	0.0514 (10)
H9A	0.4920	−0.0307	0.5850	0.062*

C8	0.4506 (4)	0.12779 (18)	0.73592 (18)	0.0522 (10)
H8A	0.5049	0.1446	0.7059	0.078*
H8B	0.5065	0.1111	0.7655	0.078*
H8C	0.3955	0.1585	0.7524	0.078*
C10	0.5774 (4)	−0.10112 (19)	0.6225 (2)	0.0571 (11)
H10A	0.6512	−0.1022	0.5982	0.069*
C13	0.3583 (4)	−0.09637 (18)	0.68994 (17)	0.0518 (10)
H13A	0.2825	−0.0953	0.7130	0.062*
C5	0.0100 (5)	−0.1112 (2)	0.5347 (2)	0.0683 (13)
H5A	−0.0506	−0.1237	0.5645	0.102*
H5B	0.0582	−0.1452	0.5205	0.102*
H5C	−0.0389	−0.0933	0.5035	0.102*
C11	0.5646 (5)	−0.14251 (19)	0.66696 (19)	0.0548 (11)
C12	0.4530 (5)	−0.1392 (2)	0.70090 (19)	0.0601 (12)
H12A	0.4414	−0.1659	0.7313	0.072*
O5	0.7541 (4)	−0.1941 (2)	0.6368 (2)	0.1054 (16)
O6	0.6596 (6)	−0.2213 (2)	0.7205 (2)	0.134 (2)
C14	0.6648 (6)	−0.1899 (2)	0.6788 (3)	0.0737 (15)
C15	0.8560 (7)	−0.2399 (3)	0.6427 (4)	0.130 (3)
H15A	0.8681	−0.2498	0.6833	0.156*
H15B	0.9399	−0.2252	0.6277	0.156*
C16	0.8152 (9)	−0.2930 (4)	0.6107 (3)	0.127 (3)
H16A	0.8815	−0.3236	0.6149	0.191*
H16B	0.8053	−0.2831	0.5705	0.191*
H16C	0.7318	−0.3072	0.6256	0.191*
Cl2	0.3288 (3)	0.21423 (11)	0.52934 (9)	0.1438 (9)
Cl3	0.2763 (2)	0.24725 (11)	0.64763 (12)	0.1451 (11)
Cl4	0.0658 (2)	0.24578 (13)	0.56700 (13)	0.1782 (13)
C17	0.2161 (6)	0.2156 (3)	0.5858 (2)	0.0785 (15)
H17A	0.1983	0.1736	0.5954	0.094*
H2	0.406 (6)	0.064 (3)	0.568 (3)	0.105 (19)*
H1	0.053 (6)	−0.060 (3)	0.683 (3)	0.10 (2)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co1	0.0347 (3)	0.0443 (3)	0.0253 (3)	−0.0059 (2)	0.00124 (18)	0.00086 (19)
Cl1	0.0387 (5)	0.0631 (6)	0.0401 (5)	0.0045 (4)	−0.0007 (4)	0.0016 (4)
N2	0.0389 (17)	0.0465 (18)	0.0306 (17)	−0.0038 (14)	0.0041 (12)	0.0030 (13)
N4	0.0354 (16)	0.0430 (16)	0.0308 (16)	−0.0006 (13)	−0.0008 (12)	0.0025 (13)
O2	0.0527 (17)	0.0618 (17)	0.0321 (14)	−0.0127 (14)	0.0075 (12)	0.0021 (12)
N3	0.0389 (17)	0.0536 (19)	0.0296 (16)	−0.0037 (14)	0.0033 (13)	0.0034 (13)
N5	0.0424 (17)	0.0409 (17)	0.0384 (17)	−0.0040 (14)	−0.0010 (13)	0.0012 (13)
O4	0.0460 (15)	0.0562 (16)	0.0398 (15)	−0.0164 (13)	0.0049 (11)	0.0074 (12)
O3	0.0506 (17)	0.0802 (19)	0.0366 (15)	−0.0215 (15)	0.0116 (12)	0.0071 (14)
C4	0.0384 (19)	0.0416 (19)	0.035 (2)	0.0047 (16)	−0.0036 (15)	0.0002 (15)
N1	0.0430 (18)	0.0542 (19)	0.0358 (18)	−0.0111 (15)	−0.0005 (13)	0.0029 (14)
C2	0.052 (2)	0.051 (2)	0.0293 (18)	−0.0015 (18)	−0.0021 (17)	−0.0057 (17)

O1	0.065 (2)	0.078 (2)	0.0422 (17)	−0.0358 (17)	0.0040 (14)	0.0046 (16)
C3	0.037 (2)	0.056 (2)	0.0287 (18)	0.0056 (17)	−0.0011 (15)	−0.0013 (17)
C1	0.052 (2)	0.055 (2)	0.037 (2)	−0.0067 (19)	−0.0063 (17)	−0.0052 (18)
C6	0.075 (3)	0.090 (4)	0.033 (2)	−0.014 (3)	0.003 (2)	−0.013 (2)
C7	0.057 (3)	0.086 (3)	0.031 (2)	−0.006 (2)	0.0015 (17)	−0.005 (2)
C9	0.047 (2)	0.051 (2)	0.056 (3)	0.0002 (19)	0.0069 (19)	0.0105 (19)
C8	0.056 (2)	0.056 (2)	0.044 (2)	−0.007 (2)	−0.0107 (19)	−0.0058 (18)
C10	0.049 (2)	0.054 (2)	0.068 (3)	0.004 (2)	0.004 (2)	0.002 (2)
C13	0.059 (3)	0.054 (2)	0.042 (2)	−0.001 (2)	0.0035 (18)	0.0085 (19)
C5	0.081 (3)	0.070 (3)	0.053 (3)	−0.029 (3)	−0.013 (2)	−0.007 (2)
C11	0.061 (3)	0.048 (2)	0.055 (3)	0.003 (2)	−0.015 (2)	−0.002 (2)
C12	0.080 (3)	0.052 (2)	0.048 (3)	0.001 (2)	−0.006 (2)	0.012 (2)
O5	0.074 (3)	0.078 (3)	0.164 (5)	0.031 (2)	0.017 (3)	0.036 (3)
O6	0.180 (5)	0.134 (4)	0.089 (3)	0.091 (4)	−0.008 (3)	0.028 (3)
C14	0.084 (4)	0.057 (3)	0.081 (4)	0.016 (3)	−0.028 (3)	0.000 (3)
C15	0.086 (5)	0.098 (5)	0.207 (9)	0.052 (4)	0.016 (5)	0.038 (6)
C16	0.151 (8)	0.114 (6)	0.118 (6)	0.072 (6)	0.003 (5)	0.020 (5)
Cl2	0.159 (2)	0.167 (2)	0.1055 (15)	0.0384 (17)	0.0411 (14)	0.0400 (13)
Cl3	0.1212 (16)	0.178 (2)	0.1355 (19)	0.0474 (15)	−0.0562 (14)	−0.0717 (16)
Cl4	0.1211 (17)	0.229 (3)	0.185 (3)	0.0840 (18)	−0.0778 (17)	−0.087 (2)
C17	0.081 (4)	0.079 (4)	0.075 (4)	−0.007 (3)	−0.009 (3)	0.001 (3)

Geometric parameters (Å, °)

Co1—N3	1.891 (3)	C7—H7C	0.9600
Co1—N4	1.898 (3)	C9—C10	1.370 (6)
Co1—N2	1.901 (3)	C9—H9A	0.9300
Co1—N1	1.902 (3)	C8—H8A	0.9600
Co1—N5	1.965 (3)	C8—H8B	0.9600
Co1—Cl1	2.2375 (12)	C8—H8C	0.9600
N2—C2	1.294 (5)	C10—C11	1.389 (6)
N2—O2	1.339 (4)	C10—H10A	0.9300
N4—C4	1.293 (5)	C13—C12	1.374 (6)
N4—O4	1.344 (4)	C13—H13A	0.9300
O2—H2	1.14 (6)	C5—H5A	0.9600
N3—C3	1.292 (5)	C5—H5B	0.9600
N3—O3	1.347 (4)	C5—H5C	0.9600
N5—C13	1.343 (5)	C11—C12	1.371 (6)
N5—C9	1.345 (5)	C11—C14	1.487 (6)
O4—H2	1.37 (6)	C12—H12A	0.9300
C4—C3	1.471 (5)	O5—C14	1.325 (7)
C4—C8	1.492 (5)	O5—C15	1.454 (7)
N1—C1	1.292 (5)	O6—C14	1.194 (7)
N1—O1	1.342 (4)	C15—C16	1.456 (11)
C2—C1	1.468 (6)	C15—H15A	0.9700
C2—C6	1.497 (5)	C15—H15B	0.9700
O1—H1	0.89 (6)	C16—H16A	0.9600
C3—C7	1.492 (5)	C16—H16B	0.9600
C1—C5	1.493 (6)	C16—H16C	0.9600

C6—H6A	0.9600	Cl2—C17	1.728 (6)
C6—H6B	0.9600	Cl3—C17	1.705 (6)
C6—H6C	0.9600	Cl4—C17	1.712 (6)
C7—H7A	0.9600	C17—H17A	0.9800
C7—H7B	0.9600		
N3—Co1—N4	81.36 (13)	H7A—C7—H7B	109.5
N3—Co1—N2	177.80 (13)	C3—C7—H7C	109.5
N4—Co1—N2	98.96 (13)	H7A—C7—H7C	109.5
N3—Co1—N1	98.27 (13)	H7B—C7—H7C	109.5
N4—Co1—N1	179.30 (14)	N5—C9—C10	122.6 (4)
N2—Co1—N1	81.39 (13)	N5—C9—H9A	118.7
N3—Co1—N5	91.17 (13)	C10—C9—H9A	118.7
N4—Co1—N5	90.15 (13)	C4—C8—H8A	109.5
N2—Co1—N5	91.00 (13)	C4—C8—H8B	109.5
N1—Co1—N5	90.45 (14)	H8A—C8—H8B	109.5
N3—Co1—Cl1	89.10 (10)	C4—C8—H8C	109.5
N4—Co1—Cl1	89.28 (9)	H8A—C8—H8C	109.5
N2—Co1—Cl1	88.73 (10)	H8B—C8—H8C	109.5
N1—Co1—Cl1	90.12 (11)	C9—C10—C11	119.8 (4)
N5—Co1—Cl1	179.33 (10)	C9—C10—H10A	120.1
C2—N2—O2	121.4 (3)	C11—C10—H10A	120.1
C2—N2—Co1	116.3 (3)	N5—C13—C12	122.7 (4)
O2—N2—Co1	122.3 (2)	N5—C13—H13A	118.6
C4—N4—O4	121.5 (3)	C12—C13—H13A	118.6
C4—N4—Co1	116.6 (2)	C1—C5—H5A	109.5
O4—N4—Co1	121.9 (2)	C1—C5—H5B	109.5
N2—O2—H2	102 (3)	H5A—C5—H5B	109.5
C3—N3—O3	121.0 (3)	C1—C5—H5C	109.5
C3—N3—Co1	116.5 (3)	H5A—C5—H5C	109.5
O3—N3—Co1	122.4 (2)	H5B—C5—H5C	109.5
C13—N5—C9	117.3 (4)	C12—C11—C10	117.6 (4)
C13—N5—Co1	121.5 (3)	C12—C11—C14	119.2 (5)
C9—N5—Co1	121.2 (3)	C10—C11—C14	123.2 (5)
N4—O4—H2	102 (2)	C11—C12—C13	120.0 (4)
N4—C4—C3	112.5 (3)	C11—C12—H12A	120.0
N4—C4—C8	124.3 (3)	C13—C12—H12A	120.0
C3—C4—C8	123.2 (3)	C14—O5—C15	117.3 (5)
C1—N1—O1	120.8 (3)	O6—C14—O5	125.3 (5)
C1—N1—Co1	116.2 (3)	O6—C14—C11	122.5 (6)
O1—N1—Co1	123.0 (2)	O5—C14—C11	112.1 (5)
N2—C2—C1	112.9 (3)	O5—C15—C16	109.1 (7)
N2—C2—C6	122.1 (4)	O5—C15—H15A	109.9
C1—C2—C6	125.0 (4)	C16—C15—H15A	109.9
N1—O1—H1	99 (4)	O5—C15—H15B	109.9
N3—C3—C4	112.9 (3)	C16—C15—H15B	109.9
N3—C3—C7	122.8 (4)	H15A—C15—H15B	108.3
C4—C3—C7	124.3 (3)	C15—C16—H16A	109.5
N1—C1—C2	113.1 (3)	C15—C16—H16B	109.5
N1—C1—C5	122.9 (4)	H16A—C16—H16B	109.5

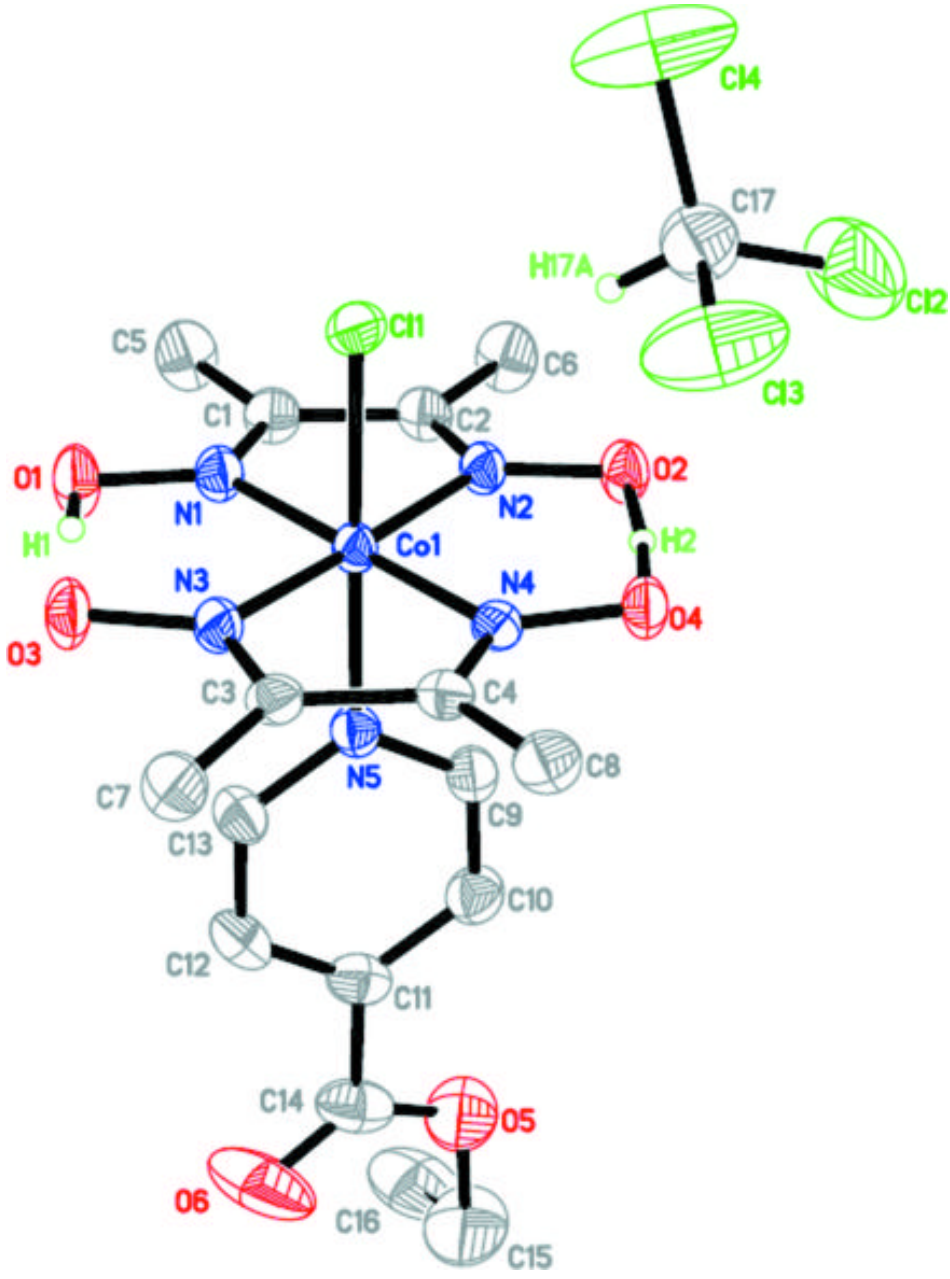
C2—C1—C5	123.9 (4)	C15—C16—H16C	109.5
C2—C6—H6A	109.5	H16A—C16—H16C	109.5
C2—C6—H6B	109.5	H16B—C16—H16C	109.5
H6A—C6—H6B	109.5	Cl3—C17—Cl4	111.2 (3)
C2—C6—H6C	109.5	Cl3—C17—Cl2	114.0 (3)
H6A—C6—H6C	109.5	Cl4—C17—Cl2	113.2 (4)
H6B—C6—H6C	109.5	Cl3—C17—H17A	105.9
C3—C7—H7A	109.5	Cl4—C17—H17A	105.9
C3—C7—H7B	109.5	Cl2—C17—H17A	105.9

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O4	1.14 (6)	1.37 (6)	2.495 (4)	168 (5)
O2—H2···N4	1.14 (6)	2.10 (6)	3.004 (4)	133 (4)
O1—H1···O3	0.89 (6)	1.60 (6)	2.486 (4)	177 (6)
O1—H1···N3	0.89 (6)	2.25 (6)	3.000 (4)	142 (5)
C17—H17A···Cl1	0.98	2.49	3.437 (6)	163
C6—H6C···Cl1^{i}	0.96	2.79	3.675 (5)	153

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

Fig. 1


Cl4
C17
H17A
Cl2
Cl3
Cl1
C5
C6
C1
C2
O1
N1
N2
O2
H1
Co1
H2
N3
N4
O4
O3
C3
C4
N5
C8
C7
C9
C13
C10
C12
C11
C14
O5
O6
C16
C15