

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

Chloridobis(ethane-1,2-diamine)(4-methylaniline)cobalt(III) dichloride monohydrate

K. Ravichandran,^a P. Ramesh,^a C. Maharaja Mahalakshmi,^b K. Anbalagan^b and M. N. Ponnuswamy^{a*}

^aCentre of Advanced Study in Crystallography and Biophysics, University of Madras, Guindy Campus, Chennai 600 025, India, and ^bDepartment of Chemistry, Pondicherry University, Pondicherry 605 014, India
Correspondence e-mail: mnpys2004@yahoo.com

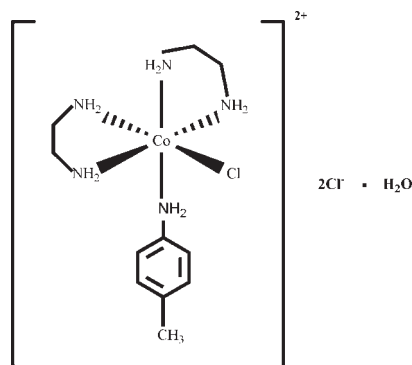
Received 30 September 2009; accepted 20 October 2009

Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.032; wR factor = 0.091; data-to-parameter ratio = 36.1.

In the title compound, $[\text{CoCl}(\text{C}_2\text{H}_8\text{N}_2)_2(\text{C}_7\text{H}_9\text{N})]\text{Cl}_2 \cdot \text{H}_2\text{O}$, the Co^{III} ion has a distorted octahedral coordination environment and is surrounded by four N atoms in an equatorial plane, with the other N and Cl atoms occupying the axial positions. The crystal packing is stabilized by $\text{N}-\text{H} \cdots \text{O}$, $\text{N}-\text{H} \cdots \text{Cl}$ and $\text{O}-\text{H} \cdots \text{Cl}$ interactions.

Related literature

For the biological activity and potential applications of mixed ligand cobalt(III) complexes, see: Arslan *et al.* (2009); Delehanty *et al.* (2008); Sayed *et al.* (1992); Teicher *et al.* (1990). For Co–N and Co–Cl bond lengths in related complexes, see: Anbalagan *et al.* (2009); Lee *et al.* (2007); Ramesh *et al.* (2008); Ravichandran *et al.* (2009). For the preparation of dichlorobis(1,2-diaminoethane)cobalt(III) chloride, see: Bailer & Clapp (1945). For asymmetry parameters, see: Nardelli (1983). For puckering parameters, see: Cremer & Pople (1975).



Experimental

Crystal data

$[\text{CoCl}(\text{C}_2\text{H}_8\text{N}_2)_2(\text{C}_7\text{H}_9\text{N})]\text{Cl}_2 \cdot \text{H}_2\text{O}$
 $M_r = 410.66$
 Triclinic, $P\bar{1}$
 $a = 7.3796$ (2) Å
 $b = 10.8367$ (3) Å
 $c = 12.1789$ (3) Å
 $\alpha = 75.201$ (1)°
 $\beta = 74.671$ (2)°
 $\gamma = 78.862$ (1)°
 $V = 899.86$ (4) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 1.40$ mm⁻¹
 $T = 293$ K
 $0.20 \times 0.20 \times 0.15$ mm

Data collection

Bruker Kappa APEXII CCD diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2004)
 $T_{\text{min}} = 0.767$, $T_{\text{max}} = 0.817$
 27923 measured reflections
 8633 independent reflections
 6851 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.025$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.032$
 $wR(F^2) = 0.091$
 $S = 1.10$
 8633 reflections
 239 parameters
 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.50$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.33$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H} \cdots A$	$D-\text{H}$	$\text{H} \cdots A$	$D \cdots A$	$D-\text{H} \cdots A$
$\text{N1}-\text{H1B} \cdots \text{Cl2}^{\text{i}}$	0.873 (17)	2.465 (17)	3.2553 (11)	150.8 (14)
$\text{N4}-\text{H4B} \cdots \text{Cl2}^{\text{ii}}$	0.797 (17)	2.602 (18)	3.3299 (11)	152.5 (15)
$\text{O1}-\text{H1W} \cdots \text{Cl3}^{\text{iii}}$	0.79 (3)	2.34 (3)	3.1339 (14)	174 (2)
$\text{O1}-\text{H2W} \cdots \text{Cl3}^{\text{iv}}$	0.77 (3)	2.49 (3)	3.2470 (14)	170 (2)
$\text{N9}-\text{H9A} \cdots \text{Cl3}$	0.87 (2)	2.45 (2)	3.3152 (11)	168.9 (17)
$\text{N8}-\text{H8B} \cdots \text{Cl2}$	0.82 (2)	2.57 (2)	3.3046 (12)	150.8 (18)
$\text{N9}-\text{H9B} \cdots \text{O1}$	0.841 (17)	2.090 (17)	2.9250 (16)	172.1 (15)
$\text{N1}-\text{H1A} \cdots \text{Cl3}$	0.88 (2)	2.39 (2)	3.2467 (11)	163.5 (17)
$\text{N4}-\text{H4A} \cdots \text{O1}$	0.801 (17)	2.461 (17)	3.0754 (17)	134.3 (14)

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

Data collection: APEX2 (Bruker, 2004); cell refinement: SAINT (Bruker, 2004); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: SHELXL97 and PLATON (Spek, 2009).

KR thanks Dr Babu Varghese, SAIF, IIT-Madras, India, for his help with the data collection, and the management of Kandaswami Kandar's College, Velur, Namakkal, TN, India, for the encouragement to pursue the programme. KA records his sincere thanks to the Council of Scientific and Industrial Research, New Delhi, and the Department of Science and Technology-SERC, New Delhi, for financial support through major research projects.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5082).

References

- Anbalagan, K., Tamilselvan, M., Nirmala, S. & Sudha, L. (2009). *Acta Cryst. E* **65**, m836–m837.
- Arslan, H., Duran, N., Borekci, G., Ozer, C. K. & Akbay, C. (2009). *Molecules*, **14**, 519–527.
- Bailer, J. C. & Clapp, L. B. (1945). *J. Am. Chem. Soc.* **67**, 171–175.
- Bruker (2004). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
- Delehanty, J. B., Bongard, J. E., Thach, C. D., Knight, D. A., Hickey, T. E. & Chang, E. L. (2008). *Bioorg. Med. Chem.* **16**, 830–837.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Lee, D. N., Lee, E. Y., Kim, C., Kim, S.-J. & Kim, Y. (2007). *Acta Cryst. E* **63**, m1949–m1950.
- Nardelli, M. (1983). *Acta Cryst. C* **39**, 1141–1142.
- Ramesh, P., SubbiahPandi, A., Jothi, P., Revathi, C. & Dayalan, A. (2008). *Acta Cryst. E* **64**, m300–m301.
- Ravichandran, K., Ramesh, P., Tamilselvan, M., Anbalagan, K. & Ponnuswamy, M. N. (2009). *Acta Cryst. E* **65**, m1174–m1175.
- Sayed, G. H., Radwan, A., Mohamed, S. M., Shiba, S. A. & Kalil, M. (1992). *Chin. J. Chem.* **10**, 475–480.
- Sheldrick, G. M. (2008). *Acta Cryst. A* **64**, 112–122.
- Spek, A. L. (2009). *Acta Cryst. D* **65**, 148–155.
- Teicher, B. A., Abrams, M. J., Rosbe, K. W. & Herman, T. S. (1990). *Cancer Res.* **50**, 6971–6975.

supplementary materials

Acta Cryst. (2009). E65, m1458-m1459 [doi:10.1107/S1600536809043323]

Chloridobis(ethane-1,2-diamine)(4-methylaniline)cobalt(III) dichloride monohydrate

K. Ravichandran, P. Ramesh, C. M. Mahalakshmi, K. Anbalagan and M. N. Ponnuswamy

Comment

Mixed ligand cobalt(III) complexes find potential applications in the fields of antitumor, antibacterial, antimicrobial, radiosensitization and cytotoxicity activities (Sayed *et al.*, 1992; Teicher *et al.*, 1990; Arslan *et al.*, 2009; Delehanty *et al.*, 2008). Cobalt is an essential and integral component of vitamin B₁₂, therefore it is physiologically found in most tissues. Complexes of cobalt are useful for nutritional supplementation to provide cobalt in a form which effectively increases the bioavailability, for instance, vitamin B₁₂ by microorganisms present in the gut. In addition, cobalt(III) complexes are known for electron transfer and ligand substitution reactions, which find applications in chemical and biological systems. Against this background and to ascertain the molecular conformation, the structure determination of the title compound has been carried out.

The ORTEP diagram of the title compound is shown in Fig.1. The coordination geometry around the Co^{III} ion can be described as a slightly distorted octahedral. The Co^{III} ion and the four N atoms almost lie in the same plane, whereas the other N and Cl atoms are approximately perpendicular to this plane. The Co—N and Co—Cl bond lengths are comparable with related complexes (Lee *et al.*, 2007; Ramesh *et al.*, 2008; Anbalagan *et al.*, 2009; Ravichandran *et al.*, 2009). One of the five membered rings in the molecule adopts an envelope conformation, whereas the other ring adopts a twist conformation with the puckering parameters (Cremer & Pople, 1975) and the asymmetry parameters (Nardelli, 1983) for the ring Co1/N1/C2/C3/N4 are: $q_2 = 0.430$ (1) Å, $\varphi = 100.4$ (1)° and $\Delta_2(\text{Co1}) = 9.92$ (9)°; and for the ring Co1/N5/C6/C7/N8 are: $q_2 = 0.428$ (2) Å, $\varphi = 272.0$ (2)° and $\Delta_2(\text{Co1}) = 1.9$ (1)°.

The crystal packing is controlled by N—H···O, N—H···Cl and O—H···Cl interactions.

Experimental

The complex was synthesized using dichlorobis(1,2-diamino ethane) cobalt(III) chloride by the reported method (Bailer & Clapp, 1945). A paste was prepared in a mortar with 2 g of *trans*-[Co^{III}(en)₂Cl₂]Cl crystals in 3–4 drops of deionized water. To the solid mass, about 2.5 g of 4-methyl aniline dissolved in ethanol was added in drops for 20 min. The grinding was continued for half an hour and the color was found to change from dull green to red. The reaction mixture was set aside until no further change was observed and the product was allowed to stand overnight. Finally, the solid was washed with 3–4 times using pure ethanol. The final complex was dissolved in 5–10 ml of deionized water pre-heated to 70°C. The cobalt(III) complex was recrystallized out on addition of few drops of hot conc. HCl and 2 ml of water and cooled. The crystals were filtered, washed with ethanol and dried over vacuum.

Refinement

Nitrogen and Oxygen H atoms were freely refined. Other H atoms were positioned geometrically (C—H = 0.93–0.97 Å) and allowed to ride on their parent atoms, with 1.5 $U_{\text{eq}}(\text{C})$ for methyl H and 1.2 $U_{\text{eq}}(\text{C})$ for other H atoms.

Figures

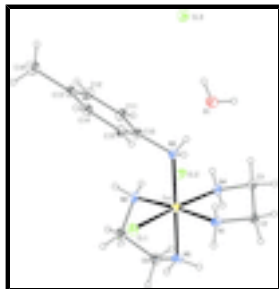


Fig. 1. Perspective view of the molecule showing the displacement ellipsoids at the 30% probability level.

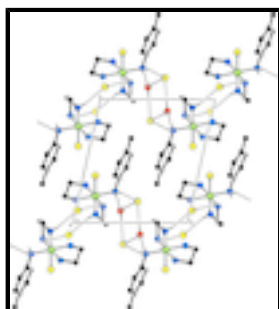


Fig. 2. The crystal packing of the molecules viewed down the a-axis. H atoms not involved in hydrogen bonding have been omitted for clarity.

Chloridobis(ethane-1,2-diamine)(4-methylaniline)cobalt(III) dichloride monohydrate

Crystal data

[CoCl(C₂H₈N₂)₂(C₇H₉N)]Cl₂·H₂O

$M_r = 410.66$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 7.3796$ (2) Å

$b = 10.8367$ (3) Å

$c = 12.1789$ (3) Å

$\alpha = 75.201$ (1)°

$\beta = 74.671$ (2)°

$\gamma = 78.862$ (1)°

$V = 899.86$ (4) Å³

$Z = 2$

$F_{000} = 428$

$D_x = 1.516$ Mg m⁻³

Mo K α radiation, $\lambda = 0.71073$ Å

Cell parameters from 5716 reflections

$\theta = 2.4$ – 32.8 °

$\mu = 1.40$ mm⁻¹

$T = 293$ K

Block, red

$0.20 \times 0.20 \times 0.15$ mm

Data collection

Bruker Kappa APEXII CCD diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293$ K

ω and φ scans

Absorption correction: multi-scan (SADABS; Bruker, 2004)

$T_{\min} = 0.767$, $T_{\max} = 0.817$

8633 independent reflections

6851 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.025$

$\theta_{\text{max}} = 37.6$ °

$\theta_{\text{min}} = 1.8$ °

$h = -11 \rightarrow 11$

$k = -18 \rightarrow 18$

27923 measured reflections

$l = -20 \rightarrow 20$

Refinement

Refinement on F^2

Secondary atom site location: difference Fourier map

Least-squares matrix: full

Hydrogen site location: inferred from neighbouring sites

$R[F^2 > 2\sigma(F^2)] = 0.032$

H atoms treated by a mixture of independent and constrained refinement

$wR(F^2) = 0.091$

$$w = 1/[\sigma^2(F_o^2) + (0.0444P)^2 + 0.0729P]$$

where $P = (F_o^2 + 2F_c^2)/3$

$S = 1.10$

$(\Delta/\sigma)_{\max} < 0.001$

8633 reflections

$\Delta\rho_{\max} = 0.50 \text{ e } \text{\AA}^{-3}$

239 parameters

$\Delta\rho_{\min} = -0.32 \text{ e } \text{\AA}^{-3}$

Primary atom site location: structure-invariant direct methods

Extinction correction: none

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	0.22560 (5)	0.10374 (4)	0.37990 (3)	0.03929 (8)
C12	-0.29764 (4)	0.01499 (3)	0.10024 (3)	0.03307 (7)
C13	0.25518 (5)	0.52141 (3)	0.17368 (3)	0.03977 (8)
Co1	0.11254 (2)	0.140646 (14)	0.218880 (12)	0.02132 (4)
O1	-0.30863 (18)	0.39142 (13)	0.09756 (12)	0.0472 (3)
H1W	-0.287 (3)	0.415 (2)	0.029 (2)	0.061 (7)*
H2W	-0.408 (4)	0.424 (2)	0.122 (2)	0.061 (7)*
N1	0.32886 (14)	0.23025 (10)	0.13118 (9)	0.02693 (18)
H1A	0.319 (3)	0.3017 (19)	0.1556 (17)	0.047 (5)*
H1B	0.435 (2)	0.1842 (16)	0.1437 (14)	0.031 (4)*
C2	0.33739 (18)	0.26181 (12)	0.00430 (11)	0.0304 (2)
H2A	0.4046	0.1897	-0.0302	0.036*
H2B	0.4031	0.3362	-0.0337	0.036*
C3	0.13641 (18)	0.29018 (12)	-0.01003 (10)	0.0305 (2)
H3A	0.0761	0.3714	0.0109	0.037*
H3B	0.1334	0.2954	-0.0902	0.037*

supplementary materials

N4	0.03717 (15)	0.18257 (11)	0.06885 (8)	0.02559 (18)
H4A	-0.075 (3)	0.1988 (15)	0.0745 (14)	0.028 (4)*
H4B	0.065 (2)	0.1235 (17)	0.0370 (15)	0.033 (4)*
N5	0.25627 (16)	-0.02271 (11)	0.18526 (11)	0.0299 (2)
H5A	0.325 (3)	-0.043 (2)	0.2275 (18)	0.051 (6)*
H5B	0.319 (3)	-0.0173 (19)	0.1170 (18)	0.045 (5)*
C6	0.1287 (2)	-0.12166 (12)	0.21421 (12)	0.0336 (2)
H6A	0.1999	-0.2072	0.2270	0.040*
H6B	0.0666	-0.1125	0.1511	0.040*
C7	-0.0154 (2)	-0.10131 (13)	0.32353 (13)	0.0383 (3)
H7A	-0.1160	-0.1536	0.3392	0.046*
H7B	0.0437	-0.1251	0.3896	0.046*
N8	-0.09254 (16)	0.03689 (11)	0.30385 (10)	0.02891 (19)
H8A	-0.146 (3)	0.0534 (18)	0.3710 (18)	0.047 (5)*
H8B	-0.177 (3)	0.0465 (18)	0.2693 (17)	0.043 (5)*
N9	-0.04520 (15)	0.30641 (10)	0.25264 (9)	0.02624 (18)
H9A	0.032 (3)	0.3621 (19)	0.2425 (17)	0.045 (5)*
H9B	-0.113 (2)	0.3357 (15)	0.2038 (14)	0.028 (4)*
C10	-0.17753 (17)	0.31173 (11)	0.36291 (10)	0.0269 (2)
C11	-0.35600 (19)	0.27735 (14)	0.38350 (12)	0.0352 (3)
H11	-0.3898	0.2479	0.3273	0.042*
C12	-0.4850 (2)	0.28674 (16)	0.48792 (13)	0.0411 (3)
H12	-0.6044	0.2621	0.5017	0.049*
C13	-0.4384 (2)	0.33237 (14)	0.57219 (11)	0.0382 (3)
C14	-0.2609 (2)	0.36910 (13)	0.54924 (11)	0.0370 (3)
H14	-0.2289	0.4018	0.6041	0.044*
C15	-0.12911 (19)	0.35829 (13)	0.44591 (11)	0.0320 (2)
H15	-0.0093	0.3821	0.4324	0.038*
C16	-0.5802 (3)	0.34315 (19)	0.68492 (14)	0.0541 (4)
H16A	-0.5368	0.3933	0.7250	0.081*
H16B	-0.5937	0.2587	0.7330	0.081*
H16C	-0.7005	0.3844	0.6686	0.081*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.04434 (18)	0.04594 (18)	0.03245 (15)	-0.00318 (14)	-0.01775 (13)	-0.00983 (13)
C12	0.02439 (13)	0.03889 (16)	0.03991 (15)	-0.00327 (11)	-0.00643 (11)	-0.01715 (13)
C13	0.04419 (18)	0.03590 (16)	0.04088 (17)	-0.01063 (13)	-0.00336 (13)	-0.01388 (13)
Co1	0.02056 (7)	0.02343 (7)	0.02069 (7)	-0.00416 (5)	-0.00377 (5)	-0.00610 (5)
O1	0.0374 (6)	0.0573 (7)	0.0410 (6)	0.0044 (5)	-0.0122 (5)	-0.0047 (5)
N1	0.0222 (4)	0.0276 (5)	0.0325 (5)	-0.0052 (4)	-0.0036 (4)	-0.0106 (4)
C2	0.0274 (5)	0.0302 (6)	0.0301 (5)	-0.0087 (4)	0.0021 (4)	-0.0057 (4)
C3	0.0310 (6)	0.0320 (6)	0.0246 (5)	-0.0060 (5)	-0.0022 (4)	-0.0018 (4)
N4	0.0240 (4)	0.0301 (5)	0.0236 (4)	-0.0064 (4)	-0.0034 (3)	-0.0075 (4)
N5	0.0272 (5)	0.0278 (5)	0.0347 (5)	-0.0025 (4)	-0.0055 (4)	-0.0094 (4)
C6	0.0392 (7)	0.0252 (5)	0.0365 (6)	-0.0066 (5)	-0.0068 (5)	-0.0068 (5)
C7	0.0456 (7)	0.0292 (6)	0.0343 (6)	-0.0101 (5)	-0.0008 (5)	-0.0012 (5)

N8	0.0285 (5)	0.0319 (5)	0.0253 (4)	-0.0084 (4)	-0.0011 (4)	-0.0064 (4)
N9	0.0258 (4)	0.0286 (5)	0.0235 (4)	-0.0034 (4)	-0.0022 (3)	-0.0078 (4)
C10	0.0276 (5)	0.0274 (5)	0.0245 (5)	-0.0015 (4)	-0.0029 (4)	-0.0081 (4)
C11	0.0319 (6)	0.0441 (7)	0.0317 (6)	-0.0065 (5)	-0.0021 (5)	-0.0161 (5)
C12	0.0330 (6)	0.0486 (8)	0.0384 (7)	-0.0093 (6)	0.0045 (5)	-0.0139 (6)
C13	0.0453 (8)	0.0352 (7)	0.0258 (5)	0.0013 (6)	0.0020 (5)	-0.0075 (5)
C14	0.0499 (8)	0.0350 (6)	0.0262 (5)	0.0001 (6)	-0.0087 (5)	-0.0111 (5)
C15	0.0355 (6)	0.0333 (6)	0.0295 (5)	-0.0036 (5)	-0.0085 (5)	-0.0104 (5)
C16	0.0601 (10)	0.0575 (10)	0.0318 (7)	0.0018 (8)	0.0083 (7)	-0.0135 (7)

Geometric parameters (Å, °)

Cl1—Co1	2.2444 (3)	C6—H6B	0.9700
Co1—N1	1.9493 (10)	C7—N8	1.4777 (18)
Co1—N5	1.9600 (11)	C7—H7A	0.9700
Co1—N8	1.9648 (11)	C7—H7B	0.9700
Co1—N4	1.9673 (10)	N8—H8A	0.86 (2)
Co1—N9	2.0167 (10)	N8—H8B	0.82 (2)
O1—H1W	0.79 (3)	N9—C10	1.4439 (15)
O1—H2W	0.77 (3)	N9—H9A	0.87 (2)
N1—C2	1.4818 (17)	N9—H9B	0.841 (17)
N1—H1A	0.88 (2)	C10—C11	1.3797 (18)
N1—H1B	0.873 (17)	C10—C15	1.3876 (16)
C2—C3	1.5027 (18)	C11—C12	1.3866 (19)
C2—H2A	0.9700	C11—H11	0.9300
C2—H2B	0.9700	C12—C13	1.388 (2)
C3—N4	1.4849 (16)	C12—H12	0.9300
C3—H3A	0.9700	C13—C14	1.379 (2)
C3—H3B	0.9700	C13—C16	1.506 (2)
N4—H4A	0.801 (17)	C14—C15	1.3883 (19)
N4—H4B	0.797 (17)	C14—H14	0.9300
N5—C6	1.4768 (17)	C15—H15	0.9300
N5—H5A	0.78 (2)	C16—H16A	0.9600
N5—H5B	0.83 (2)	C16—H16B	0.9600
C6—C7	1.505 (2)	C16—H16C	0.9600
C6—H6A	0.9700		
N1—Co1—N5	90.39 (5)	N5—C6—H6A	110.4
N1—Co1—N8	175.15 (5)	C7—C6—H6A	110.4
N5—Co1—N8	84.75 (5)	N5—C6—H6B	110.4
N1—Co1—N4	84.77 (4)	C7—C6—H6B	110.4
N5—Co1—N4	90.54 (5)	H6A—C6—H6B	108.6
N8—Co1—N4	95.17 (4)	N8—C7—C6	106.93 (11)
N1—Co1—N9	91.51 (4)	N8—C7—H7A	110.3
N5—Co1—N9	177.58 (4)	C6—C7—H7A	110.3
N8—Co1—N9	93.34 (4)	N8—C7—H7B	110.3
N4—Co1—N9	88.15 (4)	C6—C7—H7B	110.3
N1—Co1—Cl1	89.09 (3)	H7A—C7—H7B	108.6
N5—Co1—Cl1	89.51 (4)	C7—N8—Co1	109.73 (9)
N8—Co1—Cl1	90.95 (4)	C7—N8—H8A	106.8 (13)

supplementary materials

N4—Co1—C11	173.86 (3)	Co1—N8—H8A	112.7 (13)
N9—Co1—C11	92.01 (3)	C7—N8—H8B	107.1 (13)
H1W—O1—H2W	108 (2)	Co1—N8—H8B	113.5 (13)
C2—N1—Co1	111.40 (7)	H8A—N8—H8B	106.6 (18)
C2—N1—H1A	109.7 (13)	C10—N9—Co1	122.01 (8)
Co1—N1—H1A	108.8 (13)	C10—N9—H9A	106.4 (13)
C2—N1—H1B	108.9 (11)	Co1—N9—H9A	107.7 (13)
Co1—N1—H1B	111.2 (11)	C10—N9—H9B	103.3 (11)
H1A—N1—H1B	106.7 (17)	Co1—N9—H9B	109.0 (11)
N1—C2—C3	107.07 (10)	H9A—N9—H9B	107.5 (16)
N1—C2—H2A	110.3	C11—C10—C15	119.80 (11)
C3—C2—H2A	110.3	C11—C10—N9	120.15 (11)
N1—C2—H2B	110.3	C15—C10—N9	119.95 (11)
C3—C2—H2B	110.3	C10—C11—C12	120.01 (12)
H2A—C2—H2B	108.6	C10—C11—H11	120.0
N4—C3—C2	106.54 (10)	C12—C11—H11	120.0
N4—C3—H3A	110.4	C11—C12—C13	120.90 (14)
C2—C3—H3A	110.4	C11—C12—H12	119.6
N4—C3—H3B	110.4	C13—C12—H12	119.6
C2—C3—H3B	110.4	C14—C13—C12	118.40 (12)
H3A—C3—H3B	108.6	C14—C13—C16	121.16 (14)
C3—N4—Co1	108.95 (7)	C12—C13—C16	120.43 (15)
C3—N4—H4A	111.1 (12)	C13—C14—C15	121.37 (12)
Co1—N4—H4A	113.9 (11)	C13—C14—H14	119.3
C3—N4—H4B	107.5 (12)	C15—C14—H14	119.3
Co1—N4—H4B	112.7 (12)	C10—C15—C14	119.49 (13)
H4A—N4—H4B	102.4 (16)	C10—C15—H15	120.3
C6—N5—Co1	110.44 (8)	C14—C15—H15	120.3
C6—N5—H5A	108.2 (15)	C13—C16—H16A	109.5
Co1—N5—H5A	104.6 (15)	C13—C16—H16B	109.5
C6—N5—H5B	109.6 (13)	H16A—C16—H16B	109.5
Co1—N5—H5B	114.2 (13)	C13—C16—H16C	109.5
H5A—N5—H5B	110 (2)	H16A—C16—H16C	109.5
N5—C6—C7	106.72 (11)	H16B—C16—H16C	109.5
N5—Co1—N1—C2	82.07 (9)	N5—Co1—N8—C7	15.23 (9)
N8—Co1—N1—C2	81.1 (5)	N4—Co1—N8—C7	105.29 (9)
N4—Co1—N1—C2	-8.43 (8)	N9—Co1—N8—C7	-166.27 (9)
N9—Co1—N1—C2	-96.43 (8)	C11—Co1—N8—C7	-74.20 (9)
C11—Co1—N1—C2	171.58 (8)	N1—Co1—N9—C10	-142.93 (9)
Co1—N1—C2—C3	33.98 (12)	N5—Co1—N9—C10	75.2 (11)
N1—C2—C3—N4	-49.24 (13)	N8—Co1—N9—C10	37.28 (10)
C2—C3—N4—Co1	42.63 (11)	N4—Co1—N9—C10	132.35 (9)
N1—Co1—N4—C3	-19.47 (8)	C11—Co1—N9—C10	-53.79 (9)
N5—Co1—N4—C3	-109.82 (8)	Co1—N9—C10—C11	-81.78 (14)
N8—Co1—N4—C3	165.40 (8)	Co1—N9—C10—C15	101.80 (12)
N9—Co1—N4—C3	72.21 (8)	C15—C10—C11—C12	-1.4 (2)
C11—Co1—N4—C3	-19.4 (4)	N9—C10—C11—C12	-177.80 (13)
N1—Co1—N5—C6	-166.79 (9)	C10—C11—C12—C13	1.0 (2)
N8—Co1—N5—C6	13.13 (9)	C11—C12—C13—C14	0.5 (2)

N4—Co1—N5—C6	-82.01 (9)	C11—C12—C13—C16	179.51 (15)
N9—Co1—N5—C6	-24.9 (12)	C12—C13—C14—C15	-1.5 (2)
C11—Co1—N5—C6	104.13 (9)	C16—C13—C14—C15	179.43 (14)
Co1—N5—C6—C7	-37.90 (13)	C11—C10—C15—C14	0.34 (19)
N5—C6—C7—N8	50.01 (14)	N9—C10—C15—C14	176.77 (12)
C6—C7—N8—Co1	-39.65 (13)	C13—C14—C15—C10	1.1 (2)
N1—Co1—N8—C7	16.2 (6)		

Hydrogen-bond geometry (Å, °)

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
N1—H1B...C12 ⁱ	0.873 (17)	2.465 (17)	3.2553 (11)	150.8 (14)
N4—H4B...C12 ⁱⁱ	0.797 (17)	2.602 (18)	3.3299 (11)	152.5 (15)
O1—H1W...C13 ⁱⁱⁱ	0.79 (3)	2.34 (3)	3.1339 (14)	174 (2)
O1—H2W...C13 ^{iv}	0.77 (3)	2.49 (3)	3.2470 (14)	170 (2)
N9—H9A...C13	0.87 (2)	2.45 (2)	3.3152 (11)	168.9 (17)
N8—H8B...C12	0.82 (2)	2.57 (2)	3.3046 (12)	150.8 (18)
N9—H9B...O1	0.841 (17)	2.090 (17)	2.9250 (16)	172.1 (15)
N1—H1A...C13	0.88 (2)	2.39 (2)	3.2467 (11)	163.5 (17)
N4—H4A...O1	0.801 (17)	2.461 (17)	3.0754 (17)	134.3 (14)

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

Fig. 1

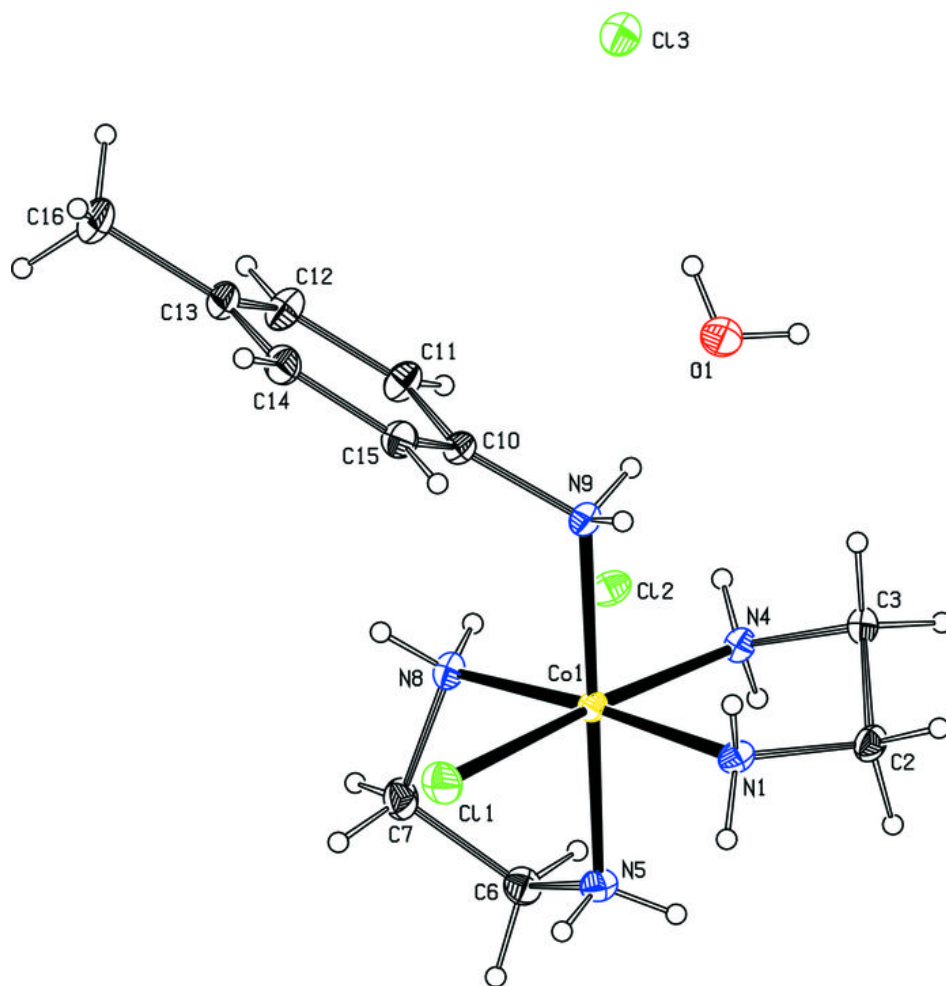


Fig. 2

