

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

K. Ravichandran,<sup>a</sup> P. Ramesh,<sup>a</sup> C. Maharaja Mahalakshmi,<sup>b</sup> K. Anbalagan<sup>b</sup> and M. N. Ponnuswamy<sup>a\*</sup>

<sup>a</sup>Centre of Advanced Study in Crystallography and Biophysics, University of Madras, Guindy Campus, Chennai 600 025, India, and <sup>b</sup>Department of Chemistry, Pondicherry University, Pondicherry 605 014, India  
Correspondence e-mail: mnpsy2004@yahoo.com

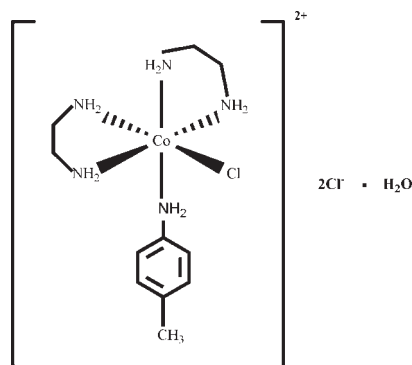
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Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å;  $R$  factor = 0.032;  $wR$  factor = 0.091; data-to-parameter ratio = 36.1.

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  $\text{Co}^{\text{III}}$  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) Å<sup>3</sup>  
 $Z = 2$   
 Mo  $K\alpha$  radiation  
 $\mu = 1.40$  mm<sup>-1</sup>  
 $T = 293$  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<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.33$  e Å<sup>-3</sup>

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).

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

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## supporting information

*Acta Cryst.* (2009). E65, m1458–m1459 [https://doi.org/10.1107/S1600536809043323]

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

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### S1. 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 B<sub>12</sub>, 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 B<sub>12</sub> 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<sup>III</sup> ion can be described as a slightly distorted octahedral. The Co<sup>III</sup> 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:  $q_2 = 0.430(1) \text{ \AA}$ ,  $\varphi = 100.4(1)^\circ$  and  $\Delta_2(\text{Co1}) = 9.92(9)^\circ$ ; and for the ring Co1/N5/C6/C7/N8 are:  $q_2 = 0.428(2) \text{ \AA}$ ,  $\varphi = 272.0(2)^\circ$  and  $\Delta_2(\text{Co1}) = 1.9(1)^\circ$ .

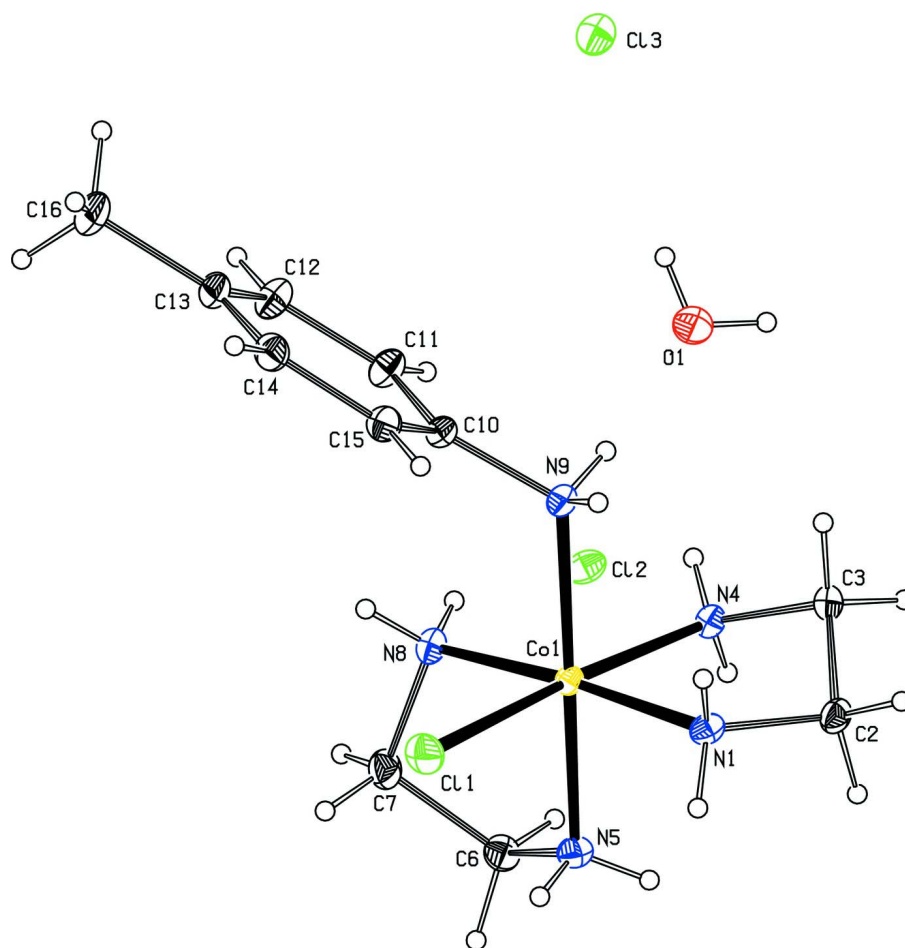
The crystal packing is controlled by N—H $\cdots$ O, N—H $\cdots$ Cl and O—H $\cdots$ Cl interactions.

### S2. 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<sup>III</sup>(en)<sub>2</sub>Cl<sub>2</sub>]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.

### S3. 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  $1.5U_{\text{eq}}(\text{C})$  for methyl H and  $1.2 U_{\text{eq}}(\text{C})$  for other H atoms.



**Figure 1**

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

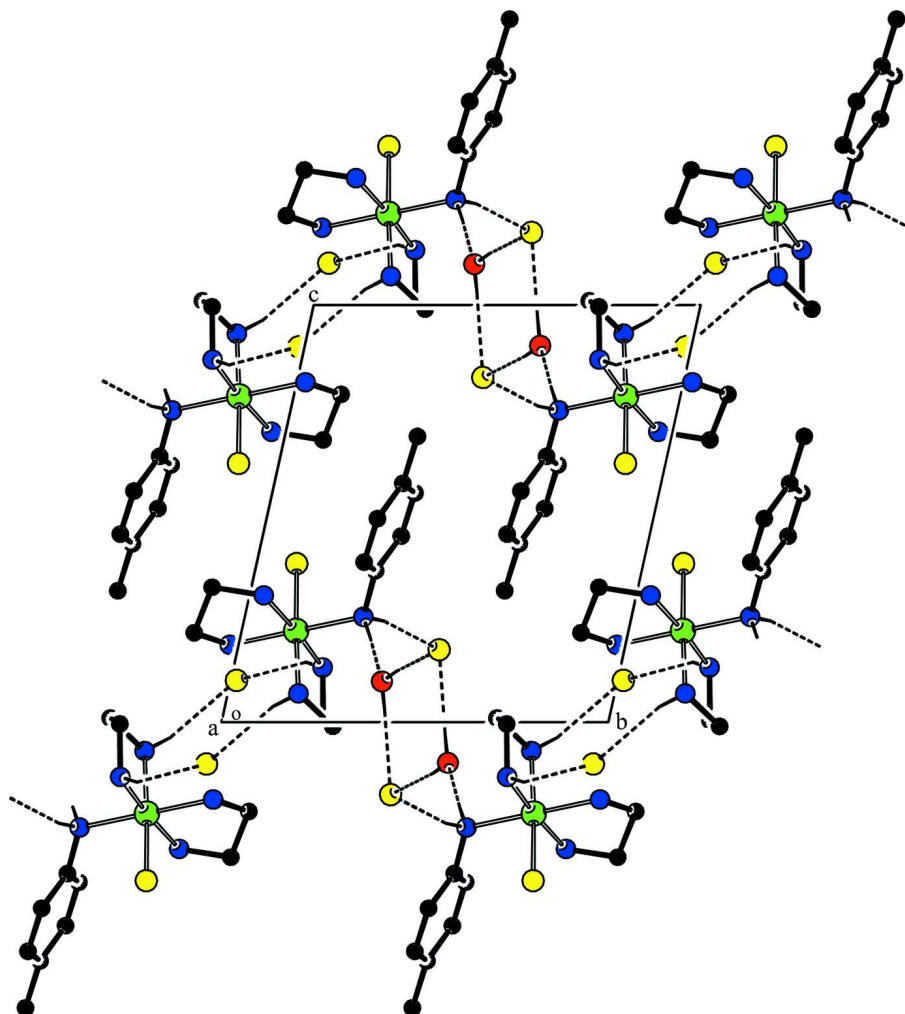


Figure 2

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

$[\text{CoCl}(\text{C}_2\text{H}_8\text{N}_2)_2(\text{C}_7\text{H}_9\text{N})]\text{Cl}_2 \cdot \text{H}_2\text{O}$

$M_r = 410.66$

Triclinic,  $P\bar{1}$

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) Å<sup>3</sup>

$Z = 2$

$F(000) = 428$

$D_x = 1.516$  Mg m<sup>-3</sup>

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

Cell parameters from 5716 reflections

$\theta = 2.4\text{--}32.8^\circ$

$\mu = 1.40$  mm<sup>-1</sup>

$T = 293$  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  
Graphite monochromator  
 $\omega$  and  $\varphi$  scans  
Absorption correction: multi-scan  
(*SADABS*; Bruker, 2004)  
 $T_{\min} = 0.767$ ,  $T_{\max} = 0.817$

27923 measured reflections  
8633 independent reflections  
6851 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.025$   
 $\theta_{\text{max}} = 37.6^\circ$ ,  $\theta_{\text{min}} = 1.8^\circ$   
 $h = -11 \rightarrow 11$   
 $k = -18 \rightarrow 18$   
 $l = -20 \rightarrow 20$

*Refinement*

Refinement on  $F^2$   
Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.032$   
 $wR(F^2) = 0.091$   
 $S = 1.10$   
8633 reflections  
239 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 atoms treated by a mixture of independent  
and constrained refinement  
 $w = 1/[\sigma^2(F_o^2) + (0.0444P)^2 + 0.0729P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.50 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.33 \text{ e } \text{\AA}^{-3}$

*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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.22560 (5)	0.10374 (4)	0.37990 (3)	0.03929 (8)
Cl2	-0.29764 (4)	0.01499 (3)	0.10024 (3)	0.03307 (7)
Cl3	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*
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 ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
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—C11	89.09 (3)	H7A—C7—H7B	108.6
N5—Co1—C11	89.51 (4)	C7—N8—Co1	109.73 (9)
N8—Co1—C11	90.95 (4)	C7—N8—H8A	106.8 (13)
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</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1 <i>B</i> ...C12 <sup>i</sup>	0.873 (17)	2.465 (17)	3.2553 (11)	150.8 (14)
N4—H4 <i>B</i> ...C12 <sup>ii</sup>	0.797 (17)	2.602 (18)	3.3299 (11)	152.5 (15)
O1—H1 <i>W</i> ...C13 <sup>iii</sup>	0.79 (3)	2.34 (3)	3.1339 (14)	174 (2)
O1—H2 <i>W</i> ...C13 <sup>iv</sup>	0.77 (3)	2.49 (3)	3.2470 (14)	170 (2)
N9—H9 <i>A</i> ...C13	0.87 (2)	2.45 (2)	3.3152 (11)	168.9 (17)
N8—H8 <i>B</i> ...C12	0.82 (2)	2.57 (2)	3.3046 (12)	150.8 (18)
N9—H9 <i>B</i> ...O1	0.841 (17)	2.090 (17)	2.9250 (16)	172.1 (15)
N1—H1 <i>A</i> ...C13	0.88 (2)	2.39 (2)	3.2467 (11)	163.5 (17)
N4—H4 <i>A</i> ...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)  $x-1, y, z$ .