

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

1-(4-Hydroxyphenyl)piperazine-1,4-dium tetrachloridocobalt(II) monohydrate

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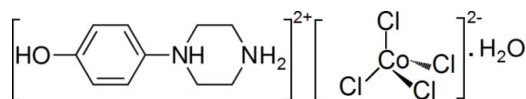
Received 25 November 2013; accepted 24 January 2014

Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.037; wR factor = 0.102; data-to-parameter ratio = 14.6.

The asymmetric unit of the title inorganic-organic hybrid compound, $(\text{C}_{10}\text{H}_{16}\text{N}_2\text{O})[\text{CoCl}_4]\cdot\text{H}_2\text{O}$, consists of a tetrahedral $[\text{CoCl}_4]^{2-}$ anion, together with a $[\text{C}_{10}\text{H}_{18}\text{N}_2\text{O}]^{2+}$ cation and a water molecule. Crystal cohesion is achieved through $\text{N}-\text{H}\cdots\text{Cl}$, $\text{O}-\text{H}\cdots\text{Cl}$ and $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds between organic cations, inorganic anions and the water molecules, building up a three-dimensional network.

Related literature

For spectroscopic and electrochemical properties of hybrid compounds, see: Bu *et al.* (2001). For a similar structural arrangement, see: Azadbakht *et al.* (2012). For the coordination of cobalt, see: Reiss (2013); Oh *et al.* (2011).



Experimental

Crystal data

 $(\text{C}_{10}\text{H}_{16}\text{N}_2\text{O})[\text{CoCl}_4]\cdot\text{H}_2\text{O}$ $M_r = 398.99$ Triclinic, $P\bar{1}$ $a = 7.455$ (1) Å $b = 8.002$ (2) Å $c = 14.105$ (1) Å $\alpha = 91.72$ (1)° $\beta = 96.98$ (1)° $\gamma = 99.19$ (1)° $V = 823.4$ (2) Å³ $Z = 2$ Mo $K\alpha$ radiation $\mu = 1.69$ mm⁻¹ $T = 298$ K $0.6 \times 0.3 \times 0.2$ mm

Data collection

Enraf-Nonius CAD-4 diffractometer

Absorption correction: ψ scan (North *et al.*, 1968) $T_{\min} = 0.607$, $T_{\max} = 0.712$

4220 measured reflections

3586 independent reflections

3110 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.027$

2 standard reflections every 120 min

intensity decay: 1%

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.037$ $wR(F^2) = 0.102$ $S = 1.08$

3586 reflections

245 parameters

All H-atom parameters refined

 $\Delta\rho_{\text{max}} = 0.73$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.45$ e Å⁻³

Table 1

Selected bond lengths (Å).

Co—Cl2	2.2475 (7)	Co—Cl1	2.2777 (7)
Co—Cl4	2.2772 (7)	Co—Cl3	2.2868 (7)

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
OW—HWA \cdots Cl3 ⁱ	0.87 (4)	2.59 (4)	3.381 (3)	150 (4)
OW—HWB \cdots Cl1 ⁱⁱ	0.78 (4)	2.51 (4)	3.264 (3)	164 (4)
O—H1 \cdots Cl3 ⁱⁱⁱ	0.79 (4)	2.41 (4)	3.177 (3)	165 (4)
N1—H1N \cdots Cl4 ^{iv}	0.94 (3)	2.24 (3)	3.171 (2)	170 (2)
N2—H2NA \cdots OW	0.83 (4)	2.06 (4)	2.807 (2)	151 (3)
N2—H2NB \cdots Cl1 ^v	0.91 (3)	2.47 (3)	3.267 (2)	147 (2)
N2—H2NB \cdots Cl2 ^v	0.91 (3)	2.81 (3)	3.324 (2)	117 (2)

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

Data collection: *CAD-4 EXPRESS* (Enraf-Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2008); software used to prepare material for publication: *pubCIF* (Westrip, 2010).

Supporting information for this paper is available from the IUCr electronic archives (Reference: CQ2009).

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supplementary materials

Acta Cryst. (2014). E70, m75 [doi:10.1107/S1600536814001767]

1-(4-Hydroxyphenyl)piperazine-1,4-dium tetrachloridocobalt(II) monohydrate

Marwa Mghandef and Habib Boughzala

1. Comment

Recently, the synthesis of organic-inorganic hybrid compounds has attracted increasing interest, not only from a structural point of view, but also because of their diverse optical properties and various applications in catalysis, electrical conductivity and photochemistry (Bu *et al.*, 2001).

Here we report the synthesis and structural characterisation of the organic-inorganic hybrid compound, (1-hydroxyphenyl)piperazine-1,4-dium tetrachloridocobalt(II) monohydrate, $[\text{C}_{10}\text{H}_{18}\text{N}_2\text{O}] [\text{CoCl}_4] \cdot \text{H}_2\text{O}$.

The asymmetric unit of this compound is composed of one tetrachlorocobalt(II) anion, one organic cation and one isolated water molecule, as shown in Figure 1. The coordination geometry of the Co(II) ion is tetrahedral with Co—Cl bond lengths ranging from 2.2475 (7) to 2.2868 (7) Å, as observed in similar compounds (Oh *et al.*; 2011), (Reiss; 2013), (Azadbakht *et al.*; 2012).

The CoCl_4 groups are isolated (0-D anionic network) and connected to three organic cations by N—H \cdots Cl and O—H \cdots Cl hydrogen bonds and to two water molecules by O—H \cdots Cl hydrogen bonds.

The organic cation, $[\text{C}_{10}\text{H}_{18}\text{N}_2\text{O}]^{2+}$, contains a piperazindium ring in a chair conformation and a planar aromatic ring (r.m.s. deviation = 0.0119 Å). The angle between the mean planes of the phenyl and piperazindium rings is about 78.0°. The crystal structure can be described as alternating stacking of organic and inorganic layers along [011], as shown in Figure 2. Water molecules link adjacent inorganic sheets.

The stability and the cohesion between the different components of the structure are assured by the water molecules connected to the organic cations through N—H \cdots O hydrogen bonds and to the tetrahedral vertices of the tetrachlorocobalt (II) anions to build up a three-dimensional network.

2. Experimental

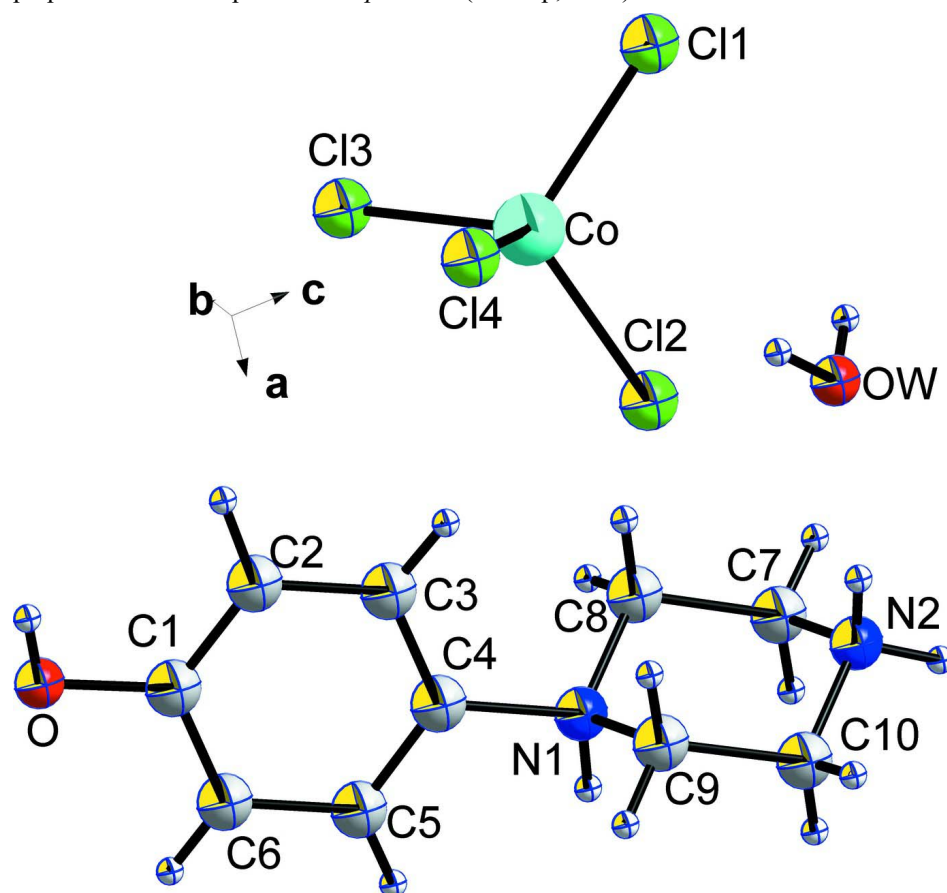
A mixture of chloride cobalt (II) $\text{CoCl}_2 \cdot \text{H}_2\text{O}$ (0.24 g) and 1-acetyl-4-(4-hydroxyphenyl)piperazine ($\text{C}_{12}\text{H}_{16}\text{N}_2\text{O}_2$) (0.11 g) (molar ratio 1:1) was dissolved in an aqueous solution of hydrochloric acid. The mixture was stirred then kept at room temperature. Blue crystals of the title compound were obtained two weeks later. The (1-hydroxyphenyl) cations are formed by loss of the acetyl group on acid hydrolysis.

3. Refinement

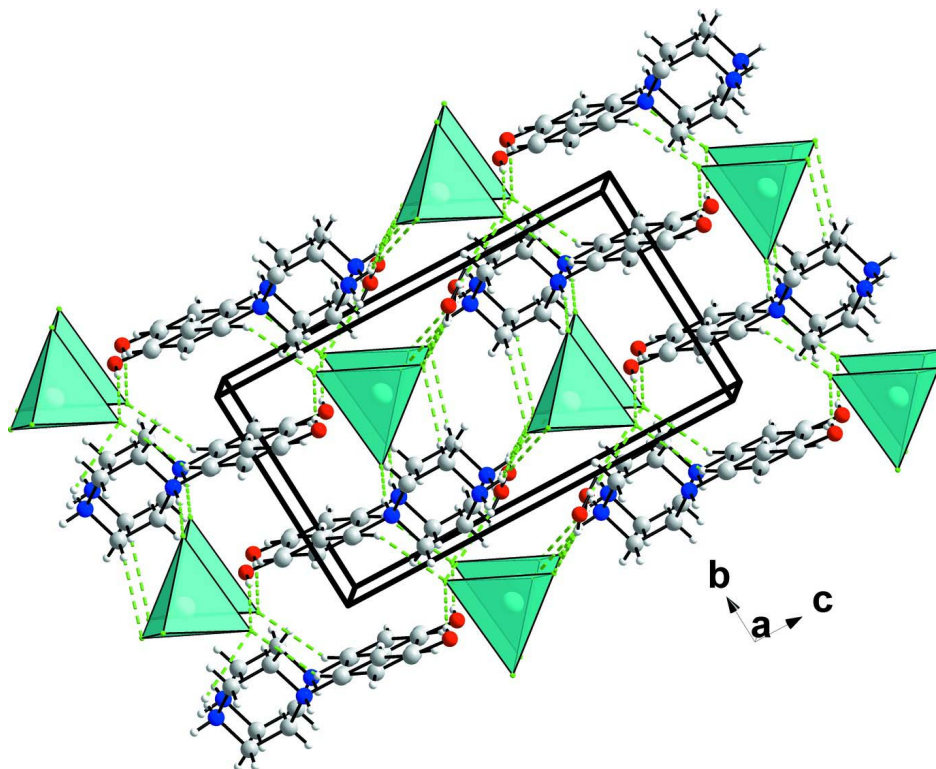
The hydrogen atoms were located in difference Fourier maps. Those attached to carbon were placed in calculated positions (C—H = 0.86 – 1.00 Å) while those attached to nitrogen and oxygen were placed in the experimental positions and their coordinates adjusted to give N—H = 0.83 Å and O—H = 0.81 Å. All were included as riding contributions with isotropic displacement parameters 1.2 - 1.5 times those of the attached atoms.

Computing details

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2008); software used to prepare material for publication: *publCIF* (Westrip, 2010).

**Figure 1**

An *ORTEP* of the molecular entities of [C₁₀H₁₈N₂O][CoCl₄]·H₂O showing the atom numbering scheme. Displacement ellipsoids are drawn at 50% probability level. Symmetry codes: (i) $x, y - 1, z$; (ii) $-x, -y + 1, -z + 1$; (iii) $-x, -y + 1, -z$; (iv) $x + 1, y, z$; (v) $-x + 1, -y + 1, -z + 1$.


Figure 2

Projection of the $[\text{C}_{10}\text{H}_{18}\text{N}_2\text{O}][\text{CoCl}_4]\cdot\text{H}_2\text{O}$ structure showing the hydrogen bonds as dashed lines and the alternating stacking of organic and inorganic layers along $[011]$.

1-(4-Hydroxyphenyl)piperazine-1,4-dium tetrachloridocobalt(II) monohydrate

Crystal data

$(\text{C}_{10}\text{H}_{16}\text{N}_2\text{O})[\text{CoCl}_4]\cdot\text{H}_2\text{O}$

$M_r = 398.99$

Triclinic, $P\bar{1}$

Hall symbol: $-P\ 1$

$a = 7.455\ (1)\ \text{\AA}$

$b = 8.002\ (2)\ \text{\AA}$

$c = 14.105\ (1)\ \text{\AA}$

$\alpha = 91.72\ (1)^\circ$

$\beta = 96.98\ (1)^\circ$

$\gamma = 99.19\ (1)^\circ$

$V = 823.4\ (2)\ \text{\AA}^3$

$Z = 2$

$F(000) = 406$

$D_x = 1.609\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 25 reflections

$\theta = 10\text{--}15^\circ$

$\mu = 1.69\ \text{mm}^{-1}$

$T = 298\ \text{K}$

Prism, blue

$0.6 \times 0.3 \times 0.2\ \text{mm}$

Data collection

Enraf–Nonius CAD-4

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

non-profiled $\omega/2\theta$ scans

Absorption correction: ψ scan

(North *et al.*, 1968)

$T_{\min} = 0.607$, $T_{\max} = 0.712$

4220 measured reflections

3586 independent reflections

3110 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.027$

$\theta_{\max} = 27.0^\circ$, $\theta_{\min} = 2.6^\circ$

$h = -9 \rightarrow 1$

$k = -10 \rightarrow 10$

$l = -17 \rightarrow 17$

2 standard reflections every 120 min

intensity decay: 1%

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.037$

$wR(F^2) = 0.102$

$S = 1.08$

3586 reflections

245 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

All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0592P)^2 + 0.2936P]$

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

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

$\Delta\rho_{\max} = 0.73 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.45 \text{ e } \text{\AA}^{-3}$

Extinction correction: *SHELXL97* (Sheldrick,
2008), $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.019 (2)

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	$U_{\text{iso}}^*/U_{\text{eq}}$
Co	0.13891 (4)	0.68672 (4)	0.31676 (2)	0.03430 (13)
Cl1	-0.02197 (8)	0.72320 (8)	0.44103 (4)	0.04349 (16)
Cl2	0.43614 (7)	0.72221 (8)	0.37771 (4)	0.04201 (16)
Cl3	0.09100 (10)	0.89884 (9)	0.21635 (5)	0.05405 (19)
Cl4	0.04924 (9)	0.43236 (8)	0.23402 (5)	0.0534 (2)
N1	0.6315 (3)	0.2602 (2)	0.20897 (12)	0.0300 (4)
N2	0.6528 (3)	0.1678 (3)	0.40592 (14)	0.0362 (4)
O	0.3173 (3)	0.2976 (3)	-0.16914 (12)	0.0487 (4)
OW	0.2791 (3)	0.1012 (3)	0.42756 (18)	0.0602 (6)
C1	0.3886 (3)	0.2802 (3)	-0.07645 (16)	0.0375 (5)
C2	0.2788 (4)	0.2306 (4)	-0.00674 (18)	0.0475 (6)
C3	0.3570 (3)	0.2225 (4)	0.08687 (17)	0.0447 (6)
C4	0.5440 (3)	0.2645 (3)	0.10896 (15)	0.0319 (4)
C5	0.6549 (3)	0.3101 (3)	0.03969 (17)	0.0395 (5)
C6	0.5760 (4)	0.3167 (3)	-0.05438 (17)	0.0422 (5)
C7	0.6411 (4)	0.3453 (3)	0.37993 (17)	0.0416 (5)
C8	0.5389 (4)	0.3477 (3)	0.28063 (17)	0.0402 (5)
C9	0.6456 (4)	0.0825 (3)	0.23609 (17)	0.0363 (5)
C10	0.7445 (3)	0.0816 (3)	0.33578 (17)	0.0379 (5)
HWA	0.193 (6)	0.050 (5)	0.384 (3)	0.082 (12)*
HWB	0.227 (6)	0.161 (5)	0.454 (3)	0.080 (13)*
H1	0.213 (5)	0.259 (5)	-0.172 (3)	0.066 (11)*
H2	0.144 (4)	0.201 (4)	-0.019 (2)	0.055 (8)*
H3	0.284 (4)	0.180 (4)	0.134 (2)	0.054 (8)*

H5	0.781 (4)	0.344 (4)	0.056 (2)	0.052 (8)*
H6	0.646 (4)	0.344 (4)	-0.102 (2)	0.052 (8)*
H7A	0.578 (5)	0.390 (4)	0.426 (2)	0.064 (9)*
H7B	0.769 (5)	0.410 (4)	0.385 (2)	0.054 (8)*
H8A	0.415 (4)	0.288 (4)	0.278 (2)	0.052 (8)*
H8B	0.531 (4)	0.449 (4)	0.264 (2)	0.051 (8)*
H9A	0.528 (5)	0.019 (4)	0.231 (2)	0.054 (8)*
H9B	0.718 (4)	0.045 (4)	0.191 (2)	0.045 (7)*
H10A	0.749 (4)	-0.027 (4)	0.353 (2)	0.047 (7)*
H10B	0.865 (4)	0.147 (4)	0.340 (2)	0.045 (7)*
H1N	0.752 (4)	0.317 (4)	0.209 (2)	0.046 (7)*
H2NA	0.551 (5)	0.113 (4)	0.411 (2)	0.063 (10)*
H2NB	0.722 (4)	0.175 (4)	0.464 (2)	0.046 (8)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co	0.03205 (18)	0.03383 (18)	0.03517 (19)	0.00247 (12)	0.00115 (12)	0.00040 (12)
C11	0.0365 (3)	0.0511 (3)	0.0436 (3)	0.0088 (2)	0.0072 (2)	-0.0033 (2)
C12	0.0314 (3)	0.0489 (3)	0.0448 (3)	0.0073 (2)	0.0020 (2)	-0.0056 (2)
C13	0.0535 (4)	0.0515 (4)	0.0533 (4)	0.0005 (3)	-0.0020 (3)	0.0208 (3)
C14	0.0400 (3)	0.0473 (3)	0.0682 (4)	-0.0050 (3)	0.0099 (3)	-0.0209 (3)
N1	0.0349 (9)	0.0261 (8)	0.0281 (8)	0.0025 (7)	0.0030 (7)	0.0021 (6)
N2	0.0353 (10)	0.0397 (10)	0.0316 (9)	0.0020 (8)	0.0003 (8)	0.0082 (8)
O	0.0569 (12)	0.0537 (11)	0.0325 (9)	0.0054 (9)	-0.0026 (8)	0.0072 (7)
OW	0.0417 (10)	0.0598 (13)	0.0764 (15)	0.0048 (9)	0.0080 (10)	-0.0254 (11)
C1	0.0479 (13)	0.0318 (10)	0.0322 (11)	0.0078 (9)	0.0015 (9)	0.0000 (8)
C2	0.0400 (13)	0.0566 (15)	0.0408 (13)	-0.0031 (11)	-0.0019 (10)	0.0074 (11)
C3	0.0384 (12)	0.0569 (15)	0.0347 (12)	-0.0052 (11)	0.0037 (10)	0.0090 (10)
C4	0.0378 (11)	0.0266 (9)	0.0301 (10)	0.0036 (8)	0.0014 (8)	0.0009 (7)
C5	0.0371 (12)	0.0469 (13)	0.0350 (11)	0.0074 (10)	0.0053 (9)	0.0024 (9)
C6	0.0466 (13)	0.0491 (13)	0.0322 (11)	0.0070 (11)	0.0107 (10)	0.0042 (10)
C7	0.0589 (15)	0.0334 (11)	0.0323 (11)	0.0088 (11)	0.0046 (10)	-0.0006 (9)
C8	0.0569 (15)	0.0344 (11)	0.0340 (11)	0.0193 (11)	0.0079 (10)	0.0032 (9)
C9	0.0458 (13)	0.0257 (10)	0.0375 (12)	0.0092 (9)	0.0013 (10)	0.0017 (8)
C10	0.0395 (12)	0.0332 (11)	0.0413 (12)	0.0096 (10)	-0.0003 (9)	0.0071 (9)

Geometric parameters (\AA , $^\circ$)

Co—C12	2.2475 (7)	C2—C3	1.385 (3)
Co—C14	2.2772 (7)	C2—H2	0.99 (3)
Co—C11	2.2777 (7)	C3—C4	1.376 (3)
Co—C13	2.2868 (7)	C3—H3	0.95 (3)
N1—C4	1.484 (3)	C4—C5	1.376 (3)
N1—C9	1.500 (3)	C5—C6	1.391 (3)
N1—C8	1.506 (3)	C5—H5	0.94 (3)
N1—H1N	0.94 (3)	C6—H6	0.91 (3)
N2—C10	1.482 (3)	C7—C8	1.513 (3)
N2—C7	1.491 (3)	C7—H7A	0.94 (3)
N2—H2NA	0.83 (4)	C7—H7B	1.00 (3)

N2—H2NB	0.91 (3)	C8—H8A	0.96 (3)
O—C1	1.370 (3)	C8—H8B	0.86 (3)
O—H1	0.79 (4)	C9—C10	1.507 (3)
OW—HWA	0.87 (4)	C9—H9A	0.93 (3)
OW—HWB	0.77 (4)	C9—H9B	0.95 (3)
C1—C6	1.376 (4)	C10—H10A	0.92 (3)
C1—C2	1.382 (4)	C10—H10B	0.96 (3)
C12—Co—C14	111.38 (3)	C3—C4—N1	120.36 (19)
C12—Co—C11	106.87 (3)	C4—C5—C6	119.3 (2)
C14—Co—C11	113.97 (3)	C4—C5—H5	120.3 (19)
C12—Co—C13	109.60 (3)	C6—C5—H5	120.3 (19)
C14—Co—C13	108.94 (3)	C1—C6—C5	119.6 (2)
C11—Co—C13	105.87 (3)	C1—C6—H6	119.3 (19)
C4—N1—C9	111.55 (16)	C5—C6—H6	121.1 (19)
C4—N1—C8	113.32 (17)	N2—C7—C8	110.53 (19)
C9—N1—C8	110.62 (17)	N2—C7—H7A	106 (2)
C4—N1—H1N	105.0 (18)	C8—C7—H7A	111 (2)
C9—N1—H1N	106.4 (18)	N2—C7—H7B	108.3 (18)
C8—N1—H1N	109.6 (17)	C8—C7—H7B	111.9 (18)
C10—N2—C7	110.87 (18)	H7A—C7—H7B	109 (3)
C10—N2—H2NA	111 (2)	N1—C8—C7	110.1 (2)
C7—N2—H2NA	112 (2)	N1—C8—H8A	107.9 (19)
C10—N2—H2NB	109.1 (18)	C7—C8—H8A	110.1 (19)
C7—N2—H2NB	106.2 (18)	N1—C8—H8B	109 (2)
H2NA—N2—H2NB	108 (3)	C7—C8—H8B	112 (2)
C1—O—H1	105 (3)	H8A—C8—H8B	107 (3)
HWA—OW—HWB	102 (4)	N1—C9—C10	110.75 (18)
O—C1—C6	117.2 (2)	N1—C9—H9A	108.8 (19)
O—C1—C2	122.2 (2)	C10—C9—H9A	111.0 (19)
C6—C1—C2	120.6 (2)	N1—C9—H9B	103.1 (17)
C1—C2—C3	119.9 (2)	C10—C9—H9B	109.7 (17)
C1—C2—H2	123.5 (18)	H9A—C9—H9B	113 (2)
C3—C2—H2	116.6 (18)	N2—C10—C9	110.96 (19)
C4—C3—C2	119.1 (2)	N2—C10—H10A	108.8 (18)
C4—C3—H3	120.2 (18)	C9—C10—H10A	110.8 (18)
C2—C3—H3	120.5 (18)	N2—C10—H10B	104.4 (17)
C5—C4—C3	121.4 (2)	C9—C10—H10B	110.3 (17)
C5—C4—N1	118.25 (19)	H10A—C10—H10B	111 (2)

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>
OW—HWA...C13 ⁱ	0.87 (4)	2.59 (4)	3.381 (3)	150 (4)
OW—HWB...C11 ⁱⁱ	0.78 (4)	2.51 (4)	3.264 (3)	164 (4)
O—H1...C13 ⁱⁱⁱ	0.79 (4)	2.41 (4)	3.177 (3)	165 (4)
N1—H1N...C14 ^{iv}	0.94 (3)	2.24 (3)	3.171 (2)	170 (2)
N2—H2NA...OW	0.83 (4)	2.06 (4)	2.807 (2)	151 (3)

N2—H2NB...C11 ^v	0.91 (3)	2.47 (3)	3.267 (2)	147 (2)
N2—H2NB...C12 ^v	0.91 (3)	2.81 (3)	3.324 (2)	117 (2)

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