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Crystal structure of diaquabis(2,6-dimethylpyrazine-κN)bis(thiocyanato-κN)-cobalt(II) 2,5-dimethylpyrazine trisolvate

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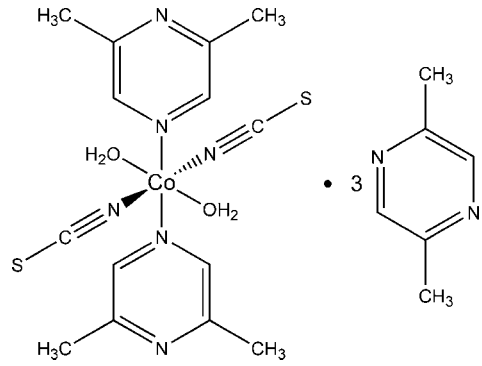
In the crystal structure of the title compound, $[Co(NCS)_2(C_6H_8N_2)_2(H_2O)_2]\cdot 3C_6H_8N_2$, the Co^{II} cation is coordinated by two terminally *N*-bound thiocyanate anions, two water molecules and two 2,6-dimethylpyrazine ligands, forming a discrete complex with a slightly distorted octahedral N_4O_2 coordination environment. The asymmetric unit contains one Co^{II} cation and three halves of 2,5-dimethylpyrazine solvate molecules, all entities being completed by inversion symmetry, as well as one thiocyanate anion, an aqua ligand and a 2,6-dimethylpyrazine ligand, all in general positions. In the crystal, discrete complexes are arranged in a way that cavities are formed where the noncoordinating 2,5-dimethylpyrazine molecules are located. The coordination of the latter to the metal is prevented due to the bulky methyl groups in vicinal positions to the N atoms, leading to a preferential coordination through the 2,6-dimethylpyrazine ligands. The complex molecules are linked by O—H···N hydrogen bonds between the water H atoms and the N atoms of 2,5-dimethylpyrazine solvent molecules, leading to a layered structure extending parallel to (100).

Keywords: crystal structure; coordination compound; octahedral coordination; cobalt(II); dimethylpyrazine.

CCDC reference: 1442780

1. Related literature

The crystal structure of the 2,5-dimethylpyrazine monosolvate of the title compound was reported recently (Suckert *et al.*, 2015*b*). For the structures of other metal thiocyanates with 2,5-dimethylpyrazine or 2,6-dimethylpyrazine, see: Otieno *et al.* (2003); Mahmoudi & Morsali (2009); Suckert *et al.* (2015*a*).



2. Experimental

2.1. Crystal data

$[Co(NCS)_2(C_6H_8N_2)_2(H_2O)_2]\cdot C_6H_8N_2$
M_r = 751.84
Triclinic, $P\overline{1}$
a = 9.3296 (8) Å
b = 10.8407 (8) Å
c = 11.3906 (9) Å
α = 103.231 (9)°
β = 111.888 (9)°
γ = 104.123 (9)°
V = 968.99 (15) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.60 mm^{-1}
T = 200 K
0.17 × 0.13 × 0.06 mm

2.2. Data collection

Stoe IPDS-1 diffractometer
Absorption correction: numerical (*X-SHAPE* and *X-RED32*; Stoe, 2008)
T_{min} = 0.909, T_{max} = 0.963
8838 measured reflections
4092 independent reflections
3540 reflections with $I > 2\sigma(I)$
R_{int} = 0.049

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.130
S = 1.05
4092 reflections
228 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.79 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1*O1*···N30	0.82	1.98	2.796 (2)	172
O1—H2*O1*···N40^{i}	0.82	2.00	2.816 (2)	174

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

Data collection: *X-AREA* (Stoe, 2008); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2015); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008) and *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *publCIF* (Westrip, 2010).

Acknowledgements

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Schleswig–Holstein. We thank Professor Dr Wolfgang Bensch for access to his experimental facilities.

Supporting information for this paper is available from the IUCr electronic archives (Reference: WM5255).

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supporting information

Acta Cryst. (2015). E**71**, m269–m270 [https://doi.org/10.1107/S2056989015024184]

Crystal structure of diaquabis(2,6-dimethylpyrazine-κN)bis(thiocyanato-κN)cobalt(II) 2,5-dimethylpyrazine trisolvate

Stefan Suckert, Susanne Wöhlert, Inke Jess and Christian Näther

S1. Synthesis and crystallization

$Co(SCN)_2$ and 2,5-dimethylpyrazine (97%) were purchased from Alfa Aesar. The title compound was prepared by the reaction of 28.9 mg (0.15 mmol) $Co(NCS)_2 \cdot H_2O$ in 195.0 μl (1.8 mmol) 2,5-dimethylpyrazine at room temperature. After a few days, plate-like crystals of the title compound were obtained that contained 2,6-dimethylpyrazine in addition. Later it was found that the commercially available 2,5-dimethylpyrazine contains about 3%$_{wt}$ of 2,6-dimethylpyrazine as a contamination.

S2. Refinement

C-bound hydrogen atoms were positioned with idealized geometry (methyl H atoms were allowed to rotate but not to tip) and were refined with U_{eq}(H) = 1.2U_{eq}(C) (1.5 for methyl H atoms) using a riding model with C—H = 0.95 Å for aromatic H atoms and C—H = 0.98 Å for methyl H atoms. The O-bound hydrogen atoms were located in a difference map. The O—H bond length was constrained to 0.84 Å and H atoms were refined with U_{iso}(H) = 1.5U_{eq}(O) using a riding model.

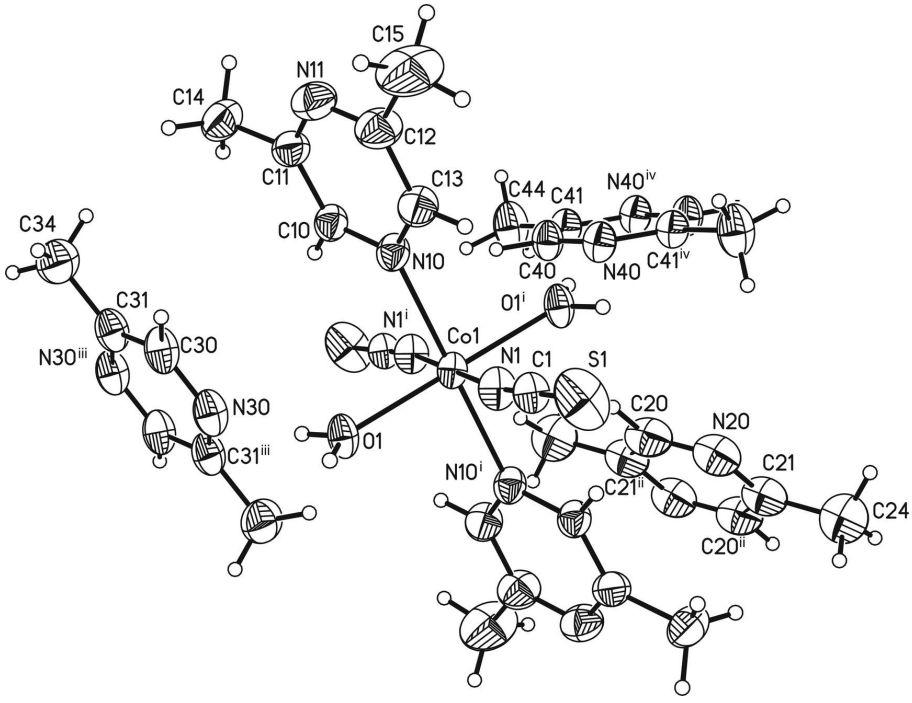

Figure 1

The structures of the molecular entities of the title compound. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry codes: (i) -*x* + 1, *y* + 1, -*z* + 1; (ii) -*x* + 1, -*y* + 1, -*z*; (iii) -*x* + 1, -*y* + 2, -*z* + 1; (iv) -*x* + 1, -*y* + 1, -*z* + 2.]

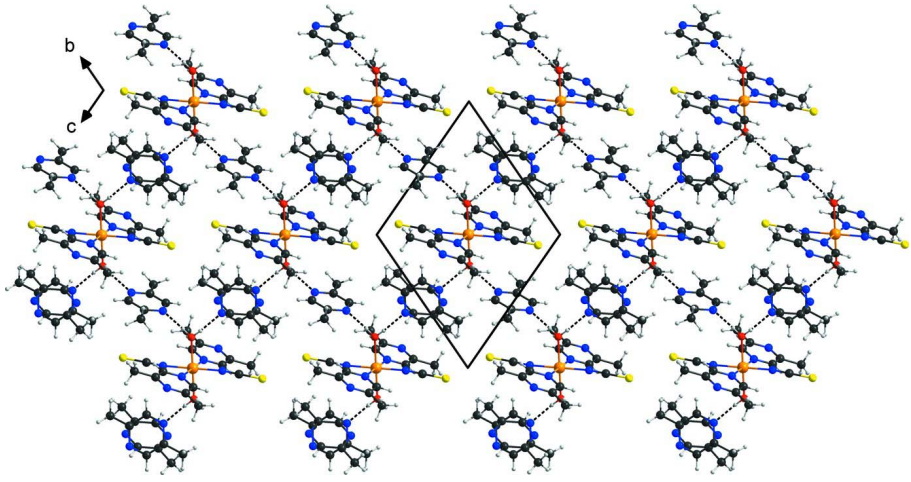

Figure 2

The crystal packing of the title compound in a view along [100]. Hydrogen bonding is shown as dashed lines.

Diaquabis(2,6-dimethylpyrazine-κ*N*)bis(thiocyanato-κ*N*)cobalt(II) 2,5-dimethylpyrazine trisolvate

Crystal data

$[Co(NCS)_2(C_6H_8N_2)_2(H_2O)_2]\cdot C_6H_8N_2$
M_r = 751.84
Triclinic, $P\overline{1}$
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c = 11.3906 (9) Å
α = 103.231 (9)°
β = 111.888 (9)°
γ = 104.123 (9)°
V = 968.99 (15) Å^3

$Z = 1$
$F(000) = 395$
$D_x = 1.288$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 8844 reflections
$\theta = 2.6$–$22.0°$
$\mu = 0.60$ mm^{-1}
$T = 200$ K
Plate, purple
0.17 × 0.13 × 0.06 mm

Data collection

Stoe IPDS-1 diffractometer
Radiation source: fine-focus sealed tube
phi scans
Absorption correction: numerical (*X-SHAPE* and *X-RED32*; Stoe, 2008)
$T_{min} = 0.909$, $T_{max} = 0.963$
8838 measured reflections
4092 independent reflections
3540 reflections with $I > 2\sigma(I)$
$R_{int} = 0.049$
$\theta_{max} = 27.4°$, $\theta_{min} = 2.6°$
$h = -11 \rightarrow 11$
$k = -13 \rightarrow 13$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.130$
$S = 1.05$
4092 reflections
228 parameters
0 restraints
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0772P)^2 + 0.2726P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.032$
$\Delta\rho_{max} = 0.37$ e Å^{-3}
$\Delta\rho_{min} = -0.79$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Co1	0.5000	0.5000	0.5000	0.02727 (14)
N1	0.4584 (2)	0.32856 (19)	0.34740 (18)	0.0352 (4)
C1	0.4289 (3)	0.2251 (2)	0.2698 (2)	0.0337 (4)
S1	0.38697 (12)	0.08006 (6)	0.15982 (8)	0.0628 (2)
N10	0.2366 (2)	0.47166 (18)	0.40932 (18)	0.0339 (4)
C10	0.1832 (3)	0.5739 (2)	0.4318 (2)	0.0351 (4)
H10	0.2616	0.6618	0.4927	0.042*
C11	0.0166 (3)	0.5558 (3)	0.3691 (2)	0.0397 (5)
C12	−0.0429 (3)	0.3307 (3)	0.2646 (3)	0.0492 (6)
C13	0.1238 (3)	0.3506 (2)	0.3258 (2)	0.0416 (5)
H13	0.1579	0.2757	0.3073	0.050*
C14	−0.0406 (3)	0.6725 (3)	0.3916 (3)	0.0533 (6)
H14A	−0.0693	0.6993	0.3118	0.080*
H14B	0.0483	0.7496	0.4710	0.080*
H14C	−0.1384	0.6451	0.4069	0.080*
C15	−0.1686 (4)	0.1922 (4)	0.1715 (4)	0.0830 (12)
H15A	−0.2500	0.1654	0.2047	0.125*

H15B	−0.1129	0.1265	0.1684	0.125*
H15C	−0.2252	0.1939	0.0804	0.125*
N11	−0.0949 (2)	0.4341 (2)	0.2867 (2)	0.0482 (5)
C20	0.8651 (3)	0.4951 (3)	0.9060 (3)	0.0539 (7)
H20	0.7663	0.4936	0.8381	0.065*
N20	0.8981 (3)	0.3820 (3)	0.8896 (2)	0.0532 (6)
C24	1.0768 (4)	0.2597 (4)	0.9700 (4)	0.0691 (8)
H24A	0.9786	0.1815	0.9460	0.104*
H24B	1.1671	0.2702	1.0554	0.104*
H24C	1.1115	0.2448	0.8982	0.104*
N30	0.5186 (3)	0.87945 (19)	0.4487 (2)	0.0425 (4)
C30	0.3732 (3)	0.8941 (2)	0.4031 (2)	0.0431 (5)
H30	0.2788	0.8192	0.3329	0.052*
C31	0.3532 (3)	1.0149 (2)	0.4541 (2)	0.0423 (5)
C21	1.0360 (3)	0.3855 (3)	0.9857 (3)	0.0515 (6)
C34	0.1905 (4)	1.0312 (3)	0.4020 (4)	0.0596 (7)
H34A	0.1321	1.0032	0.4529	0.089*
H34B	0.1241	0.9743	0.3059	0.089*
H34C	0.2076	1.1267	0.4126	0.089*
N40	0.4624 (3)	0.4506 (2)	0.86521 (18)	0.0390 (4)
C40	0.4770 (3)	0.5764 (2)	0.9248 (2)	0.0398 (5)
H40	0.4610	0.6333	0.8726	0.048*
C41	0.5148 (3)	0.6284 (2)	1.0603 (2)	0.0369 (4)
C44	0.5314 (4)	0.7707 (3)	1.1266 (3)	0.0566 (7)
H44A	0.4322	0.7690	1.1384	0.085*
H44B	0.5434	0.8240	1.0697	0.085*
H44C	0.6294	0.8122	1.2152	0.085*
O1	0.5467 (2)	0.62397 (14)	0.39186 (14)	0.0351 (3)
H1O1	0.5451	0.7009	0.4059	0.053*
H2O1	0.5481	0.5992	0.3192	0.053*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co1	0.0321 (2)	0.02219 (19)	0.0215 (2)	0.00973 (14)	0.00805 (14)	0.00454 (14)
N1	0.0432 (9)	0.0284 (8)	0.0262 (8)	0.0120 (7)	0.0122 (7)	0.0035 (7)
C1	0.0383 (10)	0.0302 (10)	0.0301 (10)	0.0121 (8)	0.0140 (8)	0.0094 (8)
S1	0.0962 (6)	0.0294 (3)	0.0568 (4)	0.0147 (3)	0.0435 (4)	−0.0014 (3)
N10	0.0333 (8)	0.0325 (9)	0.0306 (8)	0.0121 (7)	0.0091 (7)	0.0114 (7)
C10	0.0354 (10)	0.0360 (10)	0.0334 (10)	0.0142 (8)	0.0129 (8)	0.0145 (9)
C11	0.0358 (10)	0.0493 (13)	0.0389 (11)	0.0175 (9)	0.0186 (9)	0.0196 (10)
C12	0.0354 (11)	0.0461 (13)	0.0484 (14)	0.0073 (10)	0.0107 (10)	0.0086 (11)
C13	0.0364 (11)	0.0353 (11)	0.0411 (12)	0.0093 (9)	0.0104 (9)	0.0094 (9)
C14	0.0482 (13)	0.0617 (16)	0.0614 (16)	0.0317 (12)	0.0263 (12)	0.0268 (14)
C15	0.0432 (15)	0.0558 (18)	0.094 (3)	−0.0011 (13)	0.0064 (16)	−0.0079 (18)
N11	0.0344 (9)	0.0547 (12)	0.0478 (11)	0.0135 (9)	0.0140 (8)	0.0158 (10)
C20	0.0376 (12)	0.0704 (18)	0.0329 (12)	0.0066 (12)	0.0059 (9)	0.0141 (12)
N20	0.0433 (11)	0.0593 (14)	0.0330 (10)	0.0024 (10)	0.0077 (8)	0.0089 (10)

C24	0.0691 (19)	0.072 (2)	0.0572 (18)	0.0237 (16)	0.0231 (15)	0.0190 (16)
N30	0.0653 (12)	0.0271 (8)	0.0355 (10)	0.0184 (9)	0.0236 (9)	0.0090 (8)
C30	0.0606 (14)	0.0251 (9)	0.0374 (11)	0.0145 (9)	0.0182 (10)	0.0084 (9)
C31	0.0601 (14)	0.0296 (10)	0.0404 (12)	0.0175 (10)	0.0249 (11)	0.0132 (9)
C21	0.0431 (12)	0.0624 (16)	0.0350 (12)	0.0073 (11)	0.0122 (10)	0.0154 (11)
C34	0.0575 (15)	0.0407 (13)	0.078 (2)	0.0197 (12)	0.0283 (15)	0.0200 (14)
N40	0.0508 (10)	0.0414 (10)	0.0277 (8)	0.0217 (8)	0.0174 (8)	0.0128 (8)
C40	0.0538 (12)	0.0402 (11)	0.0320 (11)	0.0226 (10)	0.0205 (9)	0.0164 (9)
C41	0.0453 (11)	0.0369 (11)	0.0309 (10)	0.0178 (9)	0.0178 (9)	0.0123 (9)
C44	0.090 (2)	0.0413 (13)	0.0464 (14)	0.0312 (14)	0.0351 (14)	0.0140 (12)
O1	0.0549 (9)	0.0257 (7)	0.0277 (7)	0.0181 (6)	0.0194 (6)	0.0101 (6)

Geometric parameters (Å, º)

Co1—N1^{i}	2.0718 (18)	N20—C21	1.327 (4)
Co1—N1	2.0718 (18)	C24—C21	1.494 (5)
Co1—O1	2.0942 (15)	C24—H24A	0.9800
Co1—O1^{i}	2.0942 (15)	C24—H24B	0.9800
Co1—N10^{i}	2.1895 (18)	C24—H24C	0.9800
Co1—N10	2.1895 (18)	N30—C31^{iii}	1.316 (3)
N1—C1	1.153 (3)	N30—C30	1.324 (4)
C1—S1	1.621 (2)	C30—C31	1.393 (3)
N10—C13	1.320 (3)	C30—H30	0.9500
N10—C10	1.333 (3)	C31—N30^{iii}	1.316 (3)
C10—C11	1.387 (3)	C31—C34	1.481 (4)
C10—H10	0.9500	C21—C20^{ii}	1.373 (4)
C11—N11	1.320 (3)	C34—H34A	0.9800
C11—C14	1.494 (4)	C34—H34B	0.9800
C12—N11	1.335 (4)	C34—H34C	0.9800
C12—C13	1.382 (3)	N40—C40	1.326 (3)
C12—C15	1.497 (4)	N40—C41^{iv}	1.336 (3)
C13—H13	0.9500	C40—C41	1.386 (3)
C14—H14A	0.9800	C40—H40	0.9500
C14—H14B	0.9800	C41—N40^{iv}	1.336 (3)
C14—H14C	0.9800	C41—C44	1.495 (3)
C15—H15A	0.9800	C44—H44A	0.9800
C15—H15B	0.9800	C44—H44B	0.9800
C15—H15C	0.9800	C44—H44C	0.9800
C20—N20	1.325 (4)	O1—H1O1	0.8175
C20—C21^{ii}	1.373 (4)	O1—H2O1	0.8152
C20—H20	0.9500		
N1^{i}—Co1—N1	180.0	C11—N11—C12	118.4 (2)
N1^{i}—Co1—O1	88.87 (7)	N20—C20—C21^{ii}	124.4 (3)
N1—Co1—O1	91.13 (7)	N20—C20—H20	117.8
N1^{i}—Co1—O1^{i}	91.13 (7)	C21^{ii}—C20—H20	117.8
N1—Co1—O1^{i}	88.87 (7)	C20—N20—C21	117.1 (2)
O1—Co1—O1^{i}	180.0	C21—C24—H24A	109.5

N1^{i}—Co1—N10^{i}	91.60 (7)	C21—C24—H24B	109.5
N1—Co1—N10^{i}	88.40 (7)	H24A—C24—H24B	109.5
O1—Co1—N10^{i}	88.71 (7)	C21—C24—H24C	109.5
O1^{i}—Co1—N10^{i}	91.29 (7)	H24A—C24—H24C	109.5
N1^{i}—Co1—N10	88.40 (7)	H24B—C24—H24C	109.5
N1—Co1—N10	91.60 (7)	C31^{iii}—N30—C30	117.2 (2)
O1—Co1—N10	91.29 (7)	N30—C30—C31	122.6 (2)
O1^{i}—Co1—N10	88.71 (7)	N30—C30—H30	118.7
N10^{i}—Co1—N10	180.0	C31—C30—H30	118.7
C1—N1—Co1	172.52 (19)	N30^{iii}—C31—C30	120.3 (2)
N1—C1—S1	179.6 (2)	N30^{iii}—C31—C34	117.4 (2)
C13—N10—C10	117.27 (19)	C30—C31—C34	122.4 (2)
C13—N10—Co1	120.17 (16)	N20—C21—C20^{ii}	118.5 (3)
C10—N10—Co1	122.53 (14)	N20—C21—C24	118.3 (3)
N10—C10—C11	122.0 (2)	C20^{ii}—C21—C24	123.2 (3)
N10—C10—H10	119.0	C31—C34—H34A	109.5
C11—C10—H10	119.0	C31—C34—H34B	109.5
N11—C11—C10	120.1 (2)	H34A—C34—H34B	109.5
N11—C11—C14	118.6 (2)	C31—C34—H34C	109.5
C10—C11—C14	121.3 (2)	H34A—C34—H34C	109.5
N11—C12—C13	121.0 (2)	H34B—C34—H34C	109.5
N11—C12—C15	118.8 (2)	C40—N40—C41^{iv}	118.11 (19)
C13—C12—C15	120.3 (3)	N40—C40—C41	122.6 (2)
N10—C13—C12	121.3 (2)	N40—C40—H40	118.7
N10—C13—H13	119.3	C41—C40—H40	118.7
C12—C13—H13	119.3	N40^{iv}—C41—C40	119.3 (2)
C11—C14—H14A	109.5	N40^{iv}—C41—C44	118.5 (2)
C11—C14—H14B	109.5	C40—C41—C44	122.2 (2)
H14A—C14—H14B	109.5	C41—C44—H44A	109.5
C11—C14—H14C	109.5	C41—C44—H44B	109.5
H14A—C14—H14C	109.5	H44A—C44—H44B	109.5
H14B—C14—H14C	109.5	C41—C44—H44C	109.5
C12—C15—H15A	109.5	H44A—C44—H44C	109.5
C12—C15—H15B	109.5	H44B—C44—H44C	109.5
H15A—C15—H15B	109.5	Co1—O1—H1O1	125.2
C12—C15—H15C	109.5	Co1—O1—H2O1	126.2
H15A—C15—H15C	109.5	H1O1—O1—H2O1	107.2
H15B—C15—H15C	109.5		

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) −*x*+2, −*y*+1, −*z*+2; (iii) −*x*+1, −*y*+2, −*z*+1; (iv) −*x*+1, −*y*+1, −*z*+2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*O*1···N30	0.82	1.98	2.796 (2)	172
O1—H2*O*1···N40^{i}	0.82	2.00	2.816 (2)	174

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