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Crystal structure of a salt with a protonated sugar cation and a cobalt(II) complex anion: (GlcN–H, K)[Co(NCS)$_4$]·2H$_2$O

Tim Peppel,[a] Sabine M. L. Detert,[b] Christian Vogel[b] and Martin Köckerling[c]*

[a]Leibniz-Institut für Katalyse e.V., Heterogene Photokatalyse, Albert-Einstein-Str. 29a, D-18059 Rostock, Germany, [b]Universität Rostock, Institut für Chemie, Organische Chemie, Albert-Einstein-Str. 3a, D-18059 Rostock, Germany, and [c]Universität Rostock, Institut für Chemie, Anorganische Festkörperchemie, Albert-Einstein-Str. 3a, D-18059 Rostock, Germany. *Correspondence e-mail: Martin.Koeckerling@uni-rostock.de

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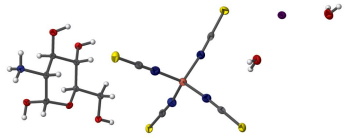
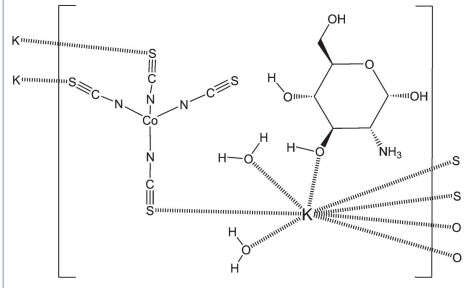
The title compound, D-(+)-glucosammonium potassium tetrathiocyanatocobaltate(II) dihydrate, K(C$_6$H$_{14}$NO$_5$)[Co(NCS)$_4$]·2H$_2$O or (GlcNH)(K)[Co(NCS)$_4$]·2H$_2$O, has been obtained as a side product of an incomplete salt metathesis reaction of D-(+)-glucosamine hydrochloride (GlcN·HCl) and K$_2$[Co(NCS)$_4$]. The asymmetric unit contains a D-(+)-glucosammonium cation, a potassium cation, a tetraisothiocyanatocobalt(II) complex anion and two water molecules. The water molecules coordinate to the potassium cation, which is further coordinated *via* three short K$^+\cdots$SCN$^-$ contacts involving three [Co(NCS)$_4$]$^{2-}$ complex anions and *via* three O atoms of two D-(+)-glucosammonium cations, leading to an overall eightfold coordination around the potassium cation. Hydrogen-bonding interactions between the building blocks consolidate the three-dimensional arrangement.

3D view



Chemical scheme



Structure description

Over about the last two decades, ionic liquids containing paramagnetic complex anions (magnetic ionic liquids, MIL) have attracted great interest because of their unique properties and possible applications (Santos *et al.*, 2014; Clark *et al.*, 2016). During our ongoing efforts to synthesize cobalt-based ionic liquids with low melting points (Kozlova *et al.*, 2009; Geppert-Rybczyńska *et al.*, 2010; Peppel *et al.*, 2010), the title compound was obtained as a side product in an attempted synthesis of new low-melting transition-metal systems containing protonated bio-molecules, *i.e.* sugar-based cations.

Table 1
Selected bond lengths (Å).

Co1—N3	1.944 (2)	N4—C4	1.162 (3)
Co1—N4	1.958 (2)	C4—S4	1.629 (2)
Co1—N2	1.968 (2)	S1—K1^{i}	3.3256 (7)
Co1—N1	1.970 (2)	S2—K1	3.3287 (8)
N1—C1	1.153 (3)	S4—K1^{ii}	3.5399 (7)
C1—S1	1.638 (2)	K1—O6	2.765 (2)
N2—C2	1.171 (3)	K1—O1^{iii}	2.812 (1)
C2—S2	1.613 (2)	K1—O7	2.860 (2)
N3—C3	1.163 (3)	K1—O3^{iv}	2.864 (1)
C3—S3	1.630 (2)	K1—O5^{iii}	2.902 (2)

Symmetry codes: (i) $-x+\frac{3}{2}, -y+2, z-\frac{1}{2}$; (ii) $-x+1, y-\frac{1}{2}, -z+\frac{3}{2}$; (iii) $x-\frac{1}{2}, -y+\frac{3}{2}, -z+1$; (iv) $x+\frac{1}{2}, -y+\frac{3}{2}, -z+1$.

Fig. 1 shows the molecular structures of the three parts present in the asymmetric unit. The title compound consists of a potassium cation that is bonded in an eightfold fashion to two water molecules, three O atoms of two neighbouring D-(+)-glucosammonium cations, and to three S atoms of three

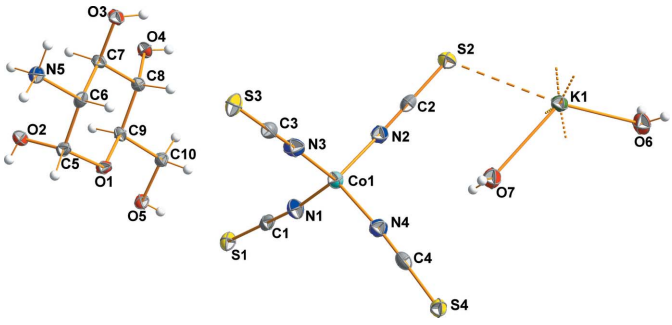


Figure 1
A view of the molecular structures of the cation–cation–anion triple present in the title compound, with atoms being presented as 50% displacement ellipsoids and with atom labelling.

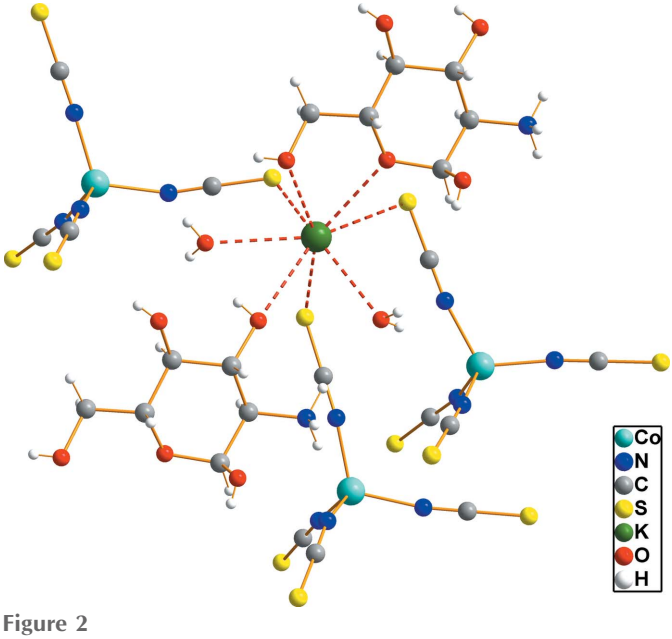


Figure 2
View of the coordination environment of the potassium cation in (GlcNH)(K)[Co(NCS)$_4$]·2H$_2$O.

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3A···O7^{iii}	0.85 (1)	1.84 (1)	2.657 (2)	160 (3)
N5—H5C···O5^{v}	0.91	2.09	2.967 (2)	162
O6—H6C···O4^{vi}	0.85 (1)	2.20 (1)	3.010 (2)	160 (2)
O2—H2A···O4^{vii}	0.85 (1)	2.20 (1)	3.015 (2)	162 (3)
N5—H5B···N2^{viii}	0.91	2.26	3.145 (2)	166
N5—H5D···O6^{iii}	0.91	2.30	3.175 (2)	160
O5—H5A···S1	0.85 (1)	2.39 (1)	3.233 (2)	176 (2)
O4—H4A···S4^{iii}	0.85 (1)	2.43 (1)	3.266 (2)	170 (3)
O7—H7C···S1^{iii}	0.85 (1)	2.49 (1)	3.298 (2)	159 (2)

Symmetry codes: (iii) $x-\frac{1}{2}, -y+\frac{3}{2}, -z+1$; (v) $x-\frac{1}{2}, -y+\frac{3}{2}, -z$; (vi) $x, y, z+1$; (vii) $x+\frac{1}{2}, -y+\frac{3}{2}, -z$; (viii) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$.

[Co(NCS)$_4$]$^{2-}$ complex anions (Fig. 2). All bond lengths and angles are in the expected ranges (Table 1).

In the crystal structure, hydrogen bonds additionally connect all the structural units. All hydrogen atoms that are attached to the N and O atoms (except one H atom of O6 that represents a water O atom) are involved in hydrogen bonding. Table 2 lists all relevant interactions up to $D\cdots A$ distances of 3.3 Å. Fig. 3 shows a cut-out of the structure with hydrogen bonds shown as red dashed lines. The three-dimensional structure can be described as a sequence of anionic and cationic layers extending parallel to (011), stacked along [011], as shown in Fig. 4.

Synthesis and crystallization

The title compound, (GlcNH)(K)[Co(NCS)$_4$]·2H$_2$O, was obtained as a side product in an incomplete salt metathesis reaction of 2 eq. D-(+)-glucosamine hydrochloride

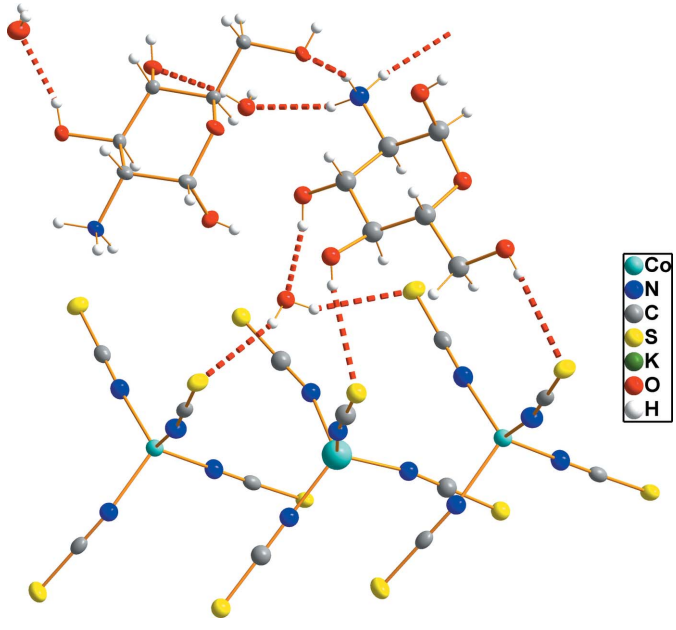


Figure 3
Hydrogen-bonding contacts between the GlcNH$^+$ cation, the (K(H$_2$O)$_2$)$^+$ cation and the [Co(NCS)$_4$]$^{2-}$ anion.

Table 3
Experimental details.

Crystal data	
Chemical formula	$[KCo(C_6H_{14}NO_5)(NCS)_4(H_2O)_2]$
M_r	546.56
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	173
a, b, c (Å)	9.3713 (2), 14.1059 (3), 15.7347 (4)
V (Å^3)	2079.98 (8)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	1.47
Crystal size (mm)	0.65 × 0.07 × 0.05
Data collection	
Diffractometer	Bruker *APEX*-X8 CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2005)
No. of measured, independent and observed $[I > 2\sigma(I)]$ reflections	24428, 9699, 7503
R_{int}	0.031
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.834
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.038, 0.058, 0.98
No. of reflections	9699
No. of parameters	285
No. of restraints	10
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.59, −0.68
Absolute structure	Flack x determined using 2715 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	0.004 (5)

Computer programs: *APEX2* and *SAINT* (Bruker, 2005), *SHELXS2014* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *DIAMOND* (Crystal Impact, 2014) and *ciftab2016* (Köckerling, 2016).

(GlcN·HCl) and 1 eq. $K_2[Co(NCS)_4]$ (Peppel *et al.*, 2010). $K_2[Co(NCS)_4]$ was obtained by heating KSCN (15.0 g, 154.0 mmol, 4 eq.) and anhydrous $CoCl_2$ (5.0 g, 38.5 mmol, 1 eq.) under reflux in 250 ml acetone for 2 h. The solvent was completely removed *in vacuo* and the residue was thoroughly extracted with ethyl acetate until the filtrate became colourless. The solvent of the combined filtrates was removed *in vacuo* and the resulting deep-blue solid was dried overnight at 393 K (14.0 g, 98%). Dry $K_2[Co(NCS)_4]$ (1.0 g, 2.7 mmol, 1 eq.) and GlcN·HCl (1.2 g, 5.4 mmol, 2 eq.) were heated under reflux in 50 ml of ethanol overnight. The hot solution was filtered and the filtrate was slowly cooled to room temperature. Deep-blue single crystals of $(GlcNH)(K)[Co(NCS)_4]\cdot 2H_2O$ were deposited at the bottom of the flask.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. A few low-angle reflections were omitted from the refinement because their intensities were affected by the beam stop.

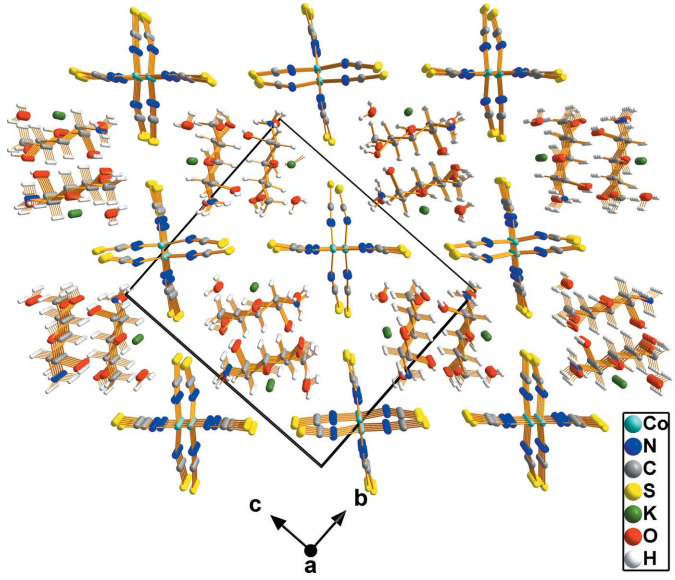


Figure 4
The packing of the ions in the crystal structure of the title compound.

Acknowledgements

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full crystallographic data

IUCrData (2019). **4**, x191142 [https://doi.org/10.1107/S2414314619011428]

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Tim Peppel, Sabine M. L. Detert, Christian Vogel and Martin Köckerling

Poly[diaqua[*μ*-*D*-(+)-glucosammonium]tri-*μ*-thiocyanato-thiocyanatocobalt(II)potassium(I)]

Crystal data

$[KCo(C_6H_{14}NO_5)(NCS)_4(H_2O)_2]$
M_r = 546.56
Orthorhombic, $P2_12_12_1$
a = 9.3713 (2) Å
b = 14.1059 (3) Å
c = 15.7347 (4) Å
V = 2079.98 (8) Å^3
Z = 4
$F(000)$ = 1116
D_x = 1.745 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9954 reflections
θ = 2.6–36.0°
μ = 1.47 mm^{-1}
T = 173 K
Block, blue
0.65 × 0.07 × 0.05 mm

Data collection

Bruker APEX-X8 CCD diffractometer
Radiation source: sealed tube
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2005)

24428 measured reflections

9699 independent reflections
7503 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 36.4°, θ_{min} = 2.6°
h = −14→15
k = −23→13
l = −26→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.058
S = 0.98
9699 reflections
285 parameters
10 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0187P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.59 e Å^{-3}
$\Delta\rho_{min}$ = −0.68 e Å^{-3}
Absolute structure: Flack x determined using 2715 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.004 (5)

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	0.62109 (3)	0.76616 (2)	0.48158 (2)	0.01938 (7)
N1	0.7606 (2)	0.8376 (1)	0.4133 (1)	0.0268 (4)
C1	0.8322 (2)	0.8742 (2)	0.3636 (1)	0.0191 (4)
S1	0.93387 (6)	0.92611 (4)	0.29276 (4)	0.0223 (1)
N2	0.5171 (2)	0.8625 (1)	0.5476 (1)	0.0232 (4)
C2	0.4283 (2)	0.8943 (2)	0.5915 (1)	0.0189 (4)
S2	0.30724 (6)	0.94110 (5)	0.65107 (4)	0.0305 (1)
N3	0.4981 (2)	0.6893 (1)	0.4099 (1)	0.0274 (4)
C3	0.4293 (2)	0.6383 (2)	0.3688 (1)	0.0200 (4)
S3	0.33187 (6)	0.56658 (4)	0.31185 (4)	0.0257 (1)
N4	0.7071 (2)	0.6805 (1)	0.5650 (1)	0.0250 (4)
C4	0.7734 (2)	0.6367 (1)	0.6135 (1)	0.0185 (4)
S4	0.86663 (6)	0.57317 (4)	0.67975 (3)	0.0233 (1)
K1	0.37801 (5)	0.88532 (3)	0.85226 (3)	0.02172 (9)
O1	0.5999 (1)	0.6576 (1)	0.09269 (9)	0.0165 (3)
O2	0.5319 (2)	0.6440 (1)	−0.05053 (9)	0.0199 (3)
H2A	0.615 (1)	0.635 (2)	−0.070 (2)	0.06 (1)*
O3	0.1598 (1)	0.6684 (1)	0.0965 (1)	0.0196 (3)
H3A	0.150 (3)	0.693 (2)	0.1456 (7)	0.049 (9)*
O4	0.3236 (2)	0.8390 (1)	0.12991 (9)	0.0170 (3)
H4A	0.324 (3)	0.865 (2)	0.1788 (7)	0.039 (8)*
O5	0.7725 (1)	0.8081 (1)	0.14516 (9)	0.0189 (3)
H5A	0.819 (2)	0.839 (2)	0.182 (1)	0.030 (7)*
N5	0.2920 (2)	0.5419 (1)	−0.0135 (1)	0.0168 (3)
H5B	0.3322	0.4843	−0.0242	0.025*
H5C	0.2942	0.5777	−0.0616	0.025*
H5D	0.1999	0.5338	0.0033	0.025*
C5	0.5299 (2)	0.6025 (2)	0.0295 (1)	0.0161 (4)
H5E	0.5759	0.5386	0.0263	0.019*
C6	0.3738 (2)	0.5909 (1)	0.0554 (1)	0.0141 (3)
H6A	0.3684	0.5524	0.1086	0.017*
C7	0.3019 (2)	0.6859 (1)	0.0699 (1)	0.0136 (4)
H7A	0.2983	0.7202	0.0144	0.016*
C8	0.3854 (2)	0.7460 (1)	0.1321 (1)	0.0126 (3)
H8A	0.3768	0.7189	0.1906	0.015*
C9	0.5425 (2)	0.7510 (1)	0.1063 (1)	0.0130 (4)
H9A	0.5522	0.7892	0.0530	0.016*
C10	0.6299 (2)	0.7957 (1)	0.1758 (1)	0.0169 (4)
H10A	0.5886	0.8577	0.1919	0.020*

H10B	0.6301	0.7543	0.2267	0.020*
O6	0.4544 (2)	0.9728 (1)	1.0028 (1)	0.0300 (4)
H6B	0.437 (3)	1.0320 (4)	1.001 (2)	0.048 (9)*
H6C	0.411 (3)	0.948 (2)	1.045 (1)	0.10 (2)*
O7	0.5753 (2)	0.7830 (1)	0.7483 (1)	0.0294 (4)
H7B	0.635 (2)	0.811 (1)	0.716 (1)	0.07 (1)*
H7C	0.559 (3)	0.7274 (8)	0.730 (2)	0.06 (1)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co1	0.0206 (1)	0.0181 (1)	0.0194 (1)	−0.0007 (1)	0.0012 (1)	0.0005 (1)
N1	0.028 (1)	0.027 (1)	0.026 (1)	−0.0017 (9)	0.0034 (8)	0.0009 (9)
C1	0.0209 (9)	0.017 (1)	0.019 (1)	0.0008 (8)	−0.0048 (8)	−0.0036 (8)
S1	0.0236 (2)	0.0243 (3)	0.0191 (2)	−0.0077 (2)	−0.0008 (2)	0.0002 (2)
N2	0.0247 (9)	0.022 (1)	0.0229 (9)	0.0007 (8)	−0.0002 (8)	0.0010 (8)
C2	0.0215 (9)	0.018 (1)	0.018 (1)	−0.0027 (8)	−0.0057 (8)	0.0033 (9)
S2	0.0288 (3)	0.0370 (3)	0.0257 (3)	0.0123 (3)	0.0029 (2)	0.0007 (3)
N3	0.027 (1)	0.023 (1)	0.032 (1)	0.0023 (8)	−0.0017 (9)	−0.0026 (9)
C3	0.0221 (9)	0.018 (1)	0.020 (1)	0.0030 (8)	0.0028 (8)	0.0033 (9)
S3	0.0340 (3)	0.0222 (3)	0.0210 (3)	−0.0055 (2)	−0.0040 (2)	0.0022 (2)
N4	0.0235 (9)	0.026 (1)	0.025 (1)	0.0006 (8)	0.0017 (8)	0.0020 (8)
C4	0.0188 (9)	0.017 (1)	0.020 (1)	−0.0031 (8)	0.0052 (8)	−0.0037 (8)
S4	0.0280 (3)	0.0238 (3)	0.0180 (2)	0.0046 (2)	−0.0015 (2)	−0.0023 (2)
K1	0.0173 (2)	0.0256 (2)	0.0223 (2)	−0.0020 (2)	0.0021 (2)	−0.0012 (2)
O1	0.0141 (6)	0.0158 (7)	0.0196 (7)	0.0024 (5)	−0.0052 (5)	−0.0050 (6)
O2	0.0163 (7)	0.0268 (9)	0.0164 (7)	0.0006 (6)	0.0022 (6)	−0.0016 (6)
O3	0.0133 (6)	0.0248 (8)	0.0207 (8)	−0.0039 (6)	0.0027 (6)	−0.0065 (7)
O4	0.0200 (7)	0.0124 (7)	0.0187 (7)	0.0028 (6)	−0.0009 (6)	−0.0040 (6)
O5	0.0130 (6)	0.0270 (8)	0.0168 (7)	−0.0062 (6)	−0.0004 (6)	−0.0045 (6)
N5	0.0175 (7)	0.0137 (8)	0.0191 (8)	−0.0017 (6)	−0.0013 (7)	−0.0029 (7)
C5	0.0156 (8)	0.0164 (9)	0.016 (1)	0.0025 (7)	−0.0039 (7)	−0.0040 (8)
C6	0.0175 (8)	0.0123 (9)	0.0125 (8)	−0.0011 (8)	−0.0028 (8)	−0.0001 (7)
C7	0.0111 (8)	0.0145 (9)	0.0153 (9)	−0.0009 (7)	0.0007 (7)	−0.0011 (7)
C8	0.0138 (7)	0.0113 (9)	0.0126 (8)	0.0002 (7)	0.0008 (7)	0.0002 (6)
C9	0.0145 (8)	0.0111 (9)	0.0134 (8)	−0.0003 (7)	0.0011 (7)	−0.0012 (7)
C10	0.0137 (8)	0.022 (1)	0.0149 (9)	−0.0027 (8)	0.0001 (8)	−0.0043 (8)
O6	0.0395 (9)	0.0252 (9)	0.025 (1)	0.0058 (8)	0.0028 (8)	−0.0036 (7)
O7	0.0303 (8)	0.035 (1)	0.0233 (8)	−0.0009 (8)	−0.0002 (7)	−0.0037 (8)

Geometric parameters (Å, º)

Co1—N3	1.944 (2)	O3—H3A	0.850 (1)
Co1—N4	1.958 (2)	O4—C8	1.434 (2)
Co1—N2	1.968 (2)	O4—H4A	0.850 (1)
Co1—N1	1.970 (2)	O5—C10	1.432 (2)
N1—C1	1.153 (3)	O5—K1^{iv}	2.902 (2)
C1—S1	1.638 (2)	O5—H5A	0.850 (1)

N2—C2	1.171 (3)	N5—C6	1.497 (2)
C2—S2	1.613 (2)	N5—H5B	0.9100
N3—C3	1.163 (3)	N5—H5C	0.9100
C3—S3	1.630 (2)	N5—H5D	0.9100
N4—C4	1.162 (3)	C5—C6	1.527 (3)
C4—S4	1.629 (2)	C5—H5E	1.0000
S1—K1^{i}	3.3256 (7)	C6—C7	1.517 (3)
S2—K1	3.3287 (8)	C6—H6A	1.0000
S4—K1^{ii}	3.5399 (7)	C7—C8	1.513 (2)
K1—O6	2.765 (2)	C7—H7A	1.0000
K1—O1^{iii}	2.812 (1)	C8—C9	1.529 (2)
K1—O7	2.860 (2)	C8—H8A	1.0000
K1—O3^{iv}	2.864 (1)	C9—C10	1.505 (3)
K1—O5^{iii}	2.902 (2)	C9—H9A	1.0000
K1—C10^{iii}	3.481 (2)	C10—K1^{iv}	3.481 (2)
O1—C5	1.422 (2)	C10—H10A	0.9900
O1—C9	1.440 (2)	C10—H10B	0.9900
O1—K1^{iv}	2.812 (1)	O6—H6B	0.850 (1)
O2—C5	1.389 (2)	O6—H6C	0.850 (1)
O2—H2A	0.850 (1)	O7—H7B	0.850 (1)
O3—C7	1.417 (2)	O7—H7C	0.850 (1)
O3—K1^{iii}	2.864 (1)		

N3—Co1—N4	106.83 (9)	C7—O3—H3A	108 (2)
N3—Co1—N2	113.43 (8)	K1^{iii}—O3—H3A	75 (2)
N4—Co1—N2	106.02 (8)	C8—O4—H4A	111 (2)
N3—Co1—N1	111.26 (9)	C10—O5—K1^{iv}	101.4 (1)
N4—Co1—N1	114.08 (8)	C10—O5—H5A	108 (2)
N2—Co1—N1	105.25 (8)	K1^{iv}—O5—H5A	108 (2)
C1—N1—Co1	170.3 (2)	C6—N5—H5B	109.5
N1—C1—S1	179.9 (2)	C6—N5—H5C	109.5
C1—S1—K1^{i}	118.29 (7)	H5B—N5—H5C	109.5
C2—N2—Co1	158.1 (2)	C6—N5—H5D	109.5
N2—C2—S2	178.4 (2)	H5B—N5—H5D	109.5
C2—S2—K1	108.43 (8)	H5C—N5—H5D	109.5
C3—N3—Co1	175.6 (2)	O2—C5—O1	113.4 (2)
N3—C3—S3	179.5 (2)	O2—C5—C6	107.4 (2)
C4—N4—Co1	171.4 (2)	O1—C5—C6	108.3 (2)
N4—C4—S4	178.6 (2)	O2—C5—H5E	109.2
C4—S4—K1^{ii}	88.48 (7)	O1—C5—H5E	109.2
O6—K1—O1^{iii}	94.13 (5)	C6—C5—H5E	109.2
O6—K1—O7	123.22 (5)	N5—C6—C7	106.8 (2)
O1^{iii}—K1—O7	131.81 (5)	N5—C6—C5	110.3 (2)
O6—K1—O3^{iv}	68.80 (5)	C7—C6—C5	111.8 (2)
O1^{iii}—K1—O3^{iv}	135.31 (4)	N5—C6—H6A	109.3
O7—K1—O3^{iv}	55.32 (5)	C7—C6—H6A	109.3
O6—K1—O5^{iii}	119.72 (5)	C5—C6—H6A	109.3
O1^{iii}—K1—O5^{iii}	58.51 (4)	O3—C7—C8	113.1 (2)

O7—K1—O5^{iii}	75.74 (5)	O3—C7—C6	108.0 (2)
O3^{iv}—K1—O5^{iii}	93.51 (4)	C8—C7—C6	111.3 (2)
O6—K1—S1^{v}	75.35 (4)	O3—C7—H7A	108.1
O1^{iii}—K1—S1^{v}	138.60 (3)	C8—C7—H7A	108.1
O7—K1—S1^{v}	84.26 (4)	C6—C7—H7A	108.1
O3^{iv}—K1—S1^{v}	78.59 (3)	O4—C8—C7	106.8 (1)
O5^{iii}—K1—S1^{v}	159.46 (3)	O4—C8—C9	109.9 (1)
O6—K1—S2	139.53 (4)	C7—C8—C9	110.6 (1)
O1^{iii}—K1—S2	99.16 (3)	O4—C8—H8A	109.9
O7—K1—S2	72.81 (4)	C7—C8—H8A	109.9
O3^{iv}—K1—S2	120.94 (3)	C9—C8—H8A	109.9
O5^{iii}—K1—S2	99.65 (3)	O1—C9—C10	106.7 (2)
S1^{v}—K1—S2	69.44 (2)	O1—C9—C8	110.9 (2)
O6—K1—C10^{iii}	127.51 (5)	C10—C9—C8	110.5 (2)
O1^{iii}—K1—C10^{iii}	42.47 (4)	O1—C9—H9A	109.6
O7—K1—C10^{iii}	89.37 (5)	C10—C9—H9A	109.6
O3^{iv}—K1—C10^{iii}	117.23 (5)	C8—C9—H9A	109.6
O5^{iii}—K1—C10^{iii}	23.78 (4)	O5—C10—C9	108.3 (2)
S1^{v}—K1—C10^{iii}	154.80 (4)	O5—C10—K1^{iv}	54.79 (9)
S2—K1—C10^{iii}	85.38 (4)	C9—C10—K1^{iv}	88.0 (1)
O6—K1—S4^{vi}	87.44 (4)	O5—C10—H10A	110.0
O1^{iii}—K1—S4^{vi}	66.72 (3)	C9—C10—H10A	110.0
O7—K1—S4^{vi}	135.67 (4)	K1^{iv}—C10—H10A	160.4
O3^{iv}—K1—S4^{vi}	146.68 (3)	O5—C10—H10B	110.0
O5^{iii}—K1—S4^{vi}	119.02 (3)	C9—C10—H10B	110.0
S1^{v}—K1—S4^{vi}	72.82 (2)	K1^{iv}—C10—H10B	70.7
S2—K1—S4^{vi}	63.81 (2)	H10A—C10—H10B	108.4
C10^{iii}—K1—S4^{vi}	95.64 (3)	K1—O6—H6B	111 (2)
C5—O1—C9	115.6 (1)	K1—O6—H6C	111 (2)
C5—O1—K1^{iv}	121.7 (1)	H6B—O6—H6C	109.5 (2)
C9—O1—K1^{iv}	119.9 (1)	K1—O7—H7B	122 (2)
C5—O2—H2A	107 (2)	K1—O7—H7C	123 (2)
C7—O3—K1^{iii}	174.6 (1)	H7B—O7—H7C	109.5 (2)

C9—O1—C5—O2	59.7 (2)	C6—C7—C8—C9	50.5 (2)
K1^{iv}—O1—C5—O2	−101.0 (2)	C5—O1—C9—C10	179.1 (2)
C9—O1—C5—C6	−59.5 (2)	K1^{iv}—O1—C9—C10	−19.9 (2)
K1^{iv}—O1—C5—C6	139.9 (1)	C5—O1—C9—C8	58.7 (2)
O2—C5—C6—N5	51.3 (2)	K1^{iv}—O1—C9—C8	−140.2 (1)
O1—C5—C6—N5	174.2 (2)	O4—C8—C9—O1	−169.3 (1)
O2—C5—C6—C7	−67.4 (2)	C7—C8—C9—O1	−51.7 (2)
O1—C5—C6—C7	55.5 (2)	O4—C8—C9—C10	72.6 (2)
N5—C6—C7—O3	61.3 (2)	C7—C8—C9—C10	−169.8 (2)
C5—C6—C7—O3	−177.9 (1)	K1^{iv}—O5—C10—C9	−73.8 (2)
N5—C6—C7—C8	−174.0 (2)	O1—C9—C10—O5	65.5 (2)
C5—C6—C7—C8	−53.2 (2)	C8—C9—C10—O5	−173.8 (2)
O3—C7—C8—O4	−68.2 (2)	O1—C9—C10—K1^{iv}	13.8 (1)

C6—C7—C8—O4	170.0 (2)	C8—C9—C10—K1^{iv}	134.4 (1)
O3—C7—C8—C9	172.3 (2)		

Symmetry codes: (i) −*x*+3/2, −*y*+2, *z*−1/2; (ii) −*x*+1, *y*−1/2, −*z*+3/2; (iii) *x*−1/2, −*y*+3/2, −*z*+1; (iv) *x*+1/2, −*y*+3/2, −*z*+1; (v) −*x*+3/2, −*y*+2, *z*+1/2; (vi) −*x*+1, *y*+1/2, −*z*+3/2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3*A*···O7^{iii}	0.85 (1)	1.84 (1)	2.657 (2)	160 (3)
N5—H5*C*···O5^{vii}	0.91	2.09	2.967 (2)	162
O6—H6*C*···O4^{viii}	0.85 (1)	2.20 (1)	3.010 (2)	160 (2)
O2—H2*A*···O4^{ix}	0.85 (1)	2.20 (1)	3.015 (2)	162 (3)
N5—H5*B*···N2^{x}	0.91	2.26	3.145 (2)	166
N5—H5*D*···O6^{iii}	0.91	2.30	3.175 (2)	160
O5—H5*A*···S1	0.85 (1)	2.38 (1)	3.233 (2)	176 (2)
O4—H4*A*···S4^{iii}	0.85 (1)	2.42 (1)	3.266 (2)	170 (3)
O7—H7*C*···S1^{iii}	0.85 (1)	2.49 (1)	3.298 (2)	159 (2)
O7—H7*B*···S3^{iv}	0.85 (1)	2.57 (1)	3.344 (2)	152 (2)
O6—H6*B*···N3^{vi}	0.85 (1)	2.69 (2)	3.378 (3)	139 (2)
C7—H7*A*···O5^{vii}	1.00	2.55	3.397 (2)	142
C5—H5*E*···S4^{xi}	1.00	2.93	3.560 (2)	122
C9—H9*A*···O3^{ix}	1.00	2.63	3.560 (2)	155
C8—H8*A*···S3	1.00	2.90	3.828 (2)	154

Symmetry codes: (iii) *x*−1/2, −*y*+3/2, −*z*+1; (iv) *x*+1/2, −*y*+3/2, −*z*+1; (vi) −*x*+1, *y*+1/2, −*z*+3/2; (vii) *x*−1/2, −*y*+3/2, −*z*; (viii) *x*, *y*, *z*+1; (ix) *x*+1/2, −*y*+3/2, −*z*; (x) −*x*+1, *y*−1/2, −*z*+1/2; (xi) −*x*+3/2, −*y*+1, *z*−1/2.