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Bis[tris(oxamide dioxime-κ^2N,N')-cobalt(III)] oxalate bis(sulfate) dodecahydrate

Michel M. Belombe,[a]* Justin Nenwa,[a] Gouet Bebga,[a] Boniface P. T. Fokwa[b] and Richard Dronskowski[b]

[a]Department of Inorganic Chemistry, University of Yaounde I, POB 812 Yaounde, Cameroon, and [b]Institut für Anorganische Chemie, RWTH Aachen, D-52056 Aachen, Germany
Correspondence e-mail: belombe2000@yahoo.fr

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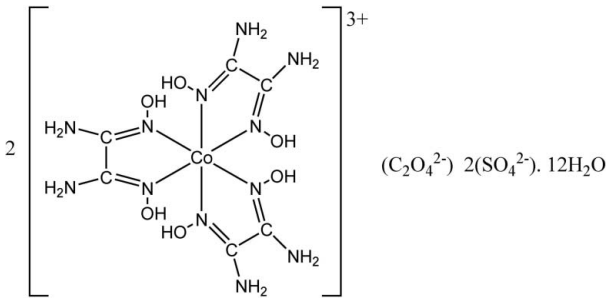
Key indicators: single-crystal X-ray study; T = 193 K; mean σ(C–C) = 0.005 Å; disorder in solvent or counterion; R factor = 0.062; wR factor = 0.158; data-to-parameter ratio = 19.6.

In the title compound, $[Co(C_2H_6N_4O_2)_3]_2(C_2O_4)(SO_4)_2\cdot 12H_2O$, the Co^{3+} ion adopts a distorted octahedral coordination involving six imino N atoms of three bidentate oxamide dioxime ligands. The oxalate ion is centrosymmetric. The bulk structure is consolidated by a network of O—H···O and N—H···O hydrogen bonds, interconnecting the building blocks in such a manner that the framework delineates infinite channels parallel to [100]. The 12 water molecules are lodged inside the channels, six of them being O—H···O and N—H···O bonded to the ionic species, whilst the other six, located along the central axis of the channel, form infinite cyclohexameric water tapes. Two of the water molecules are disordered over two sites, in 0.786 (7):0.214 (7) and 0.637 (8):0.363 (8) ratios.

Related literature

For related literature, see: Bélombé *et al.* (1993, 2007); Bailar & Jones (1939); Bekaroglu *et al.* (1978); Infantes & Motherwell (2002); Ludwig (2001); Mascal *et al.* (2006); Nenwa (2004).



Experimental

Crystal data

$[Co(C_2H_6N_4O_2)_3]_2(C_2O_4)(SO_4)_2\cdot 12H_2O$
M_r = 1314.78
Triclinic, $P\overline{1}$
a = 9.4298 (10) Å
b = 11.7820 (12) Å
c = 12.8118 (13) Å
α = 65.494 (2)°
β = 83.088 (2)°
γ = 86.735 (2)°
V = 1285.7 (2) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.85 mm^{-1}
T = 193 (2) K
0.25 × 0.15 × 0.10 mm

Data collection

Bruker APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.816, T_{max} = 0.919
20047 measured reflections
7453 independent reflections
5173 reflections with $I > 2\sigma(I)$
R_{int} = 0.056

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.158
S = 1.10
7453 reflections
381 parameters
12 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.91 e Å^{-3}
$\Delta\rho_{min}$ = −0.53 e Å^{-3}

Table 1
Selected bond lengths (Å).

Co—N21	1.900 (3)	Co—N11	1.920 (3)
Co—N14	1.906 (3)	Co—N34	1.922 (3)
Co—N31	1.917 (3)	Co—N24	1.932 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
N16—H16B···O42^{i}	0.88	1.99	2.864 (4)	172
N17—H17B···O54^{ii}	0.88	2.06	2.926 (4)	167
N17—H17A···O42^{i}	0.88	2.00	2.835 (4)	159
N27—H27A···O54^{iii}	0.88	2.20	3.069 (4)	170
N26—H26B···O54^{iii}	0.88	2.06	2.900 (4)	158
N36—H36B···O28^{iv}	0.88	2.23	2.952 (4)	139
N37—H37B···O52^{v}	0.88	2.51	2.998 (4)	115
N37—H37B···O38^{vi}	0.88	2.54	3.321 (4)	149
O15—H15···O43^{vii}	0.84	1.77	2.584 (4)	162
O18—H18···O54^{iv}	0.84	1.88	2.709 (4)	172
O18—H18···O52^{iv}	0.84	2.56	3.124 (4)	125
O28—H28···O51^{iv}	0.84	1.78	2.617 (4)	177
O35—H35···O42^{viii}	0.84	1.88	2.667 (4)	155
O35—H35···O43^{vii}	0.84	2.30	2.881 (4)	127
O38—H38···O52^{iv}	0.84	1.76	2.602 (4)	177
N16—H16A···O1^{ix}	0.88	2.18	3.016 (4)	159
N27—H27B···O2^{iv}	0.88	2.02	2.865 (4)	162
N36—H36A···O3	0.88	2.10	2.971 (5)	170
N37—H37A···O2	0.88	2.07	2.879 (5)	152
O25—H25···O1^{iii}	0.84	1.83	2.662 (4)	169
O1—H2O···O53^{v}	0.80 (3)	2.19 (4)	2.931 (4)	156 (6)
O2—H3O···O53^{v}	0.83 (3)	2.08 (3)	2.890 (4)	165 (6)
O3—H6O···O51	0.84 (3)	1.87 (4)	2.653 (5)	157 (7)
O1—H1O···O5A	0.79 (3)	2.11 (3)	2.879 (7)	164 (6)
O2—H4O···O6A	0.85 (3)	1.96 (3)	2.799 (8)	166 (6)
O3—H5O···O4	0.84 (3)	2.16 (6)	2.831 (8)	137 (7)
O4—H7O···O6A^{x}	1.01 (3)	1.99 (6)	2.736 (9)	128 (5)
O4—H8O···O5A^{x}	0.85 (3)	2.59 (4)	3.375 (8)	153 (6)

Symmetry codes: (i) $-x+2, -y+2, -z+1$; (ii) $x+1, y+1, z$; (iii) $x, y+1, z$; (iv) $-x+1, -y+1, -z+1$; (v) $x+1, y, z$; (vi) $-x+2, -y+1, -z+1$; (vii) $x, y, z-1$; (viii) $-x+1, -y+2, -z+1$; (ix) $-x+2, -y+1, -z$; (x) $-x+1, -y+1, -z$.

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Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2446).

References

Bailar, J. C. & Jones, E. M. (1939). *Inorg. Synth.* **1**, 35–38.
Bekaroglu, Ö., Sarisaban, S., Koray, A. R., Nuber, B., Weidenhammer, K., Weiss, J. & Ziegler, M. L. (1978). *Acta Cryst.* B**34**, 3591–3593.
Bélombé, M. M., Jokwi, I., Ngameni, E., Roux, R. & Nuber, B. (1993). *Z. Naturforsch. Teil B*, **48**, 1719–1722.
Bélombé, M. M., Nenwa, J., Mbiangué, Y.-A., Gouet Bebga, Majoumo- Mbé, F., Hey-Hawkins, E. & Lönnecke, P. (2007). *Inorg. Chim. Acta*, doi:10.1016/j.ica.2007.03.003.
Brandenburg, K. (1999). *DIAMOND*. Release 2.1c. Crystal Impact GbR, Bonn, Germany.
Bruker (1998). *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2000). *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2001). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Infantes, L. & Motherwell, S. (2002). *CrystEngComm*, **4**, 454–461.
Ludwig, R. (2001). *Angew. Chem. Int. Ed.* **40**, 1808.
Mascal, M., Infantes, L. & Chisholm, J. (2006). *Angew. Chem. Int. Ed.* **45**, 32.
Nenwa, J. (2004). PhD dissertation, University of Yaounde I, Cameroon.
Sheldrick, G. M. (1997). *SHELXS97*and *SHELXL97*. University of Göttingen, Germany.

supplementary materials

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Bis[tris(oxamide dioxime-κ^2N,N')cobalt(III)] oxalate bis(sulfate) dodecahydrate

M. M. Belombe, J. Nenwa, G. Bebga, B. P. T. Fokwa and R. Dronskowski

Comment

Water clusters, encapsulated as crystal hydrates in various solid networks, were recently introduced as a new field of considerable scientific relevance, both in theoretical and in experimental studies (Ludwig, 2001; Infantes & Motherwell, 2002; Mascal *et al.*, 2006). The point at stake relates to the need for a comprehensive analysis of hydrogen-bonded water clusters, $(H_2O)_n$, where n > 1. Infantes and Motherwell (2002) have selected the cluster patterns and sorted their structures into five broad classes designated D, *R*, C, T and *L*.

We describe here compound (**I**) as a salt of the rare complex cation, $[Co(H_2oxado)_3]^{3+}$ (Bekaroglu *et al.*, 1978; Bélombé *et al*., 1993; Nenwa, 2004). It is a transition metal complex system that crystallizes in a nanochannelled lattice encapsulating infinite tapes of cyclic water hexamers, and it provides, therefore, another well documented example to be added to category T of the aforementioned classification.

The constituent parts of (**I**) are depicted in Fig. 1. The pseudo-octahedral coordination in the complex cation is similar to the chiral geometries, and the bond lengths and angles compare within experimental error with those reported previously (Nenwa, 2004; Bekaroglu *et al.*, 1978; Bélombé *et al.*, 1993). The host lattice of the structure is realised by the ionic partners which are linked together *via* a three dimensional network of O–H···O and N–H···O hydrogen bonds. The ions of each kind pile up in an eclipsed sequence to generate the corresponding charged stacks. The electrically neutral scaffold thus constructed is characterized by infinite channels (*ca* 6.4 Å wide) oriented parallel to [100], and encapsulating twelve water molecules of crystallization per unit cell.

The projection of a unit cell of (**I**) in Fig. 2 shows the positioning of the water molecules within the channels, reminiscent of the structure of a silver salt reported recently (Bélombé *et al.*, 2007). The oxalate ion is centrosymmetric. Thus, only one oxalate ion is present in the unit cell, the other species being represented twice. A short segment of a water tape in (**I**) viewed down [010] is shown in Fig. 3. One distinguishes, from the ellipsoid size, two types of water molecules per asymmetric unit. Those containing atoms O1, O2 and O3, positioned close to the periphery of the channels – due to their involvement in H-bonding to the ionic building blocks of the host lattice – may be dubbed "peripheral" waters. The reduced ellipsoid size of these O atoms reveals that they are well ordered. The water molecules with the atoms O4, O5A and O6A may be referred to as "central" waters, as they are located around the central axis of the channel. The ellipsoid extent of these O atoms indicates their high disorder, probably due to their increased mobility since they are less strongly bonded than their "peripheral" congeners.

Table 2 summarizes the three categories of H-bonds that are effective in this structure. O–H···O and N–H···O bridges interconnect the ionic partners amongst themselves into the three dimensional host lattice. Note that these bridges represent the most efficient ones, with the shortest O···O separation of 2.584 (4)Å linking an oxamide dioxime to an oxalate O atom, *viz*. O15···O43^x (see Table 2 for symmetry code). Then, O–H···O and N–H···O bridges of medium efficiency interlink the

"peripheral" water molecules to the ionic partners. Finally, O–H···O bridges weakly interconnect water molecules amongst themselves within the nanochannels.

Experimental

Commercial $CoSO_4·7H_2O$, freshly prepared H_2oxado (Nenwa, 2004), and $K_3[Co(C_2O_4)_3]·3H_2O$ (Bailar & Jones, 1939), were mixed together in a ratio of 0.56 g (2 mmol):0.95 g (8 mmol):0.99 g (2 mmol) in water (120 ml) with stirring at room temperature. The resulting red-brown precipitate was discarded by filtration. Concentration of the filtrate by slow evaporation in the hood over three weeks yielded dark-red prisms of (I) that were filtered off and dried in air at room temperature.

Refinement

The non-water H atoms were positioned geometrically (O—H = 0.84 Å, N—H = 0.86 Å) and refined as riding with U_{iso}(H) = 1.2 U_{eq}(N) or 1.5U_{eq}(O). All the water H atoms were first located in a difference Fourier map and then refined with distance restraints of O–H = 0.85 (3) Å and H···H = 1.39 (3) Å, and with equal U_{iso}(H). The highest peak and deepest hole in the final difference map are 0.55 Å from atom H6A and 0.29 Å from O5A, respectively.

Due to the disorder observed for the two oxygen sites (O5 and O6), no hydrogen atoms could be located for these O atoms.

Figures

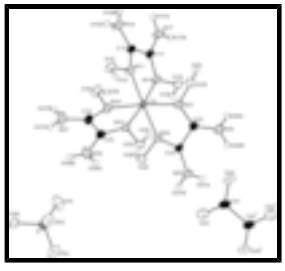

Fig. 1. Structures of the ions $[Co(H_2oxado)_3]^{3+}$, $C_2O_4^{2-}$ and SO_4^{2-} in (**I**) with ellipsoids drawn at 50% probability. Symmetry code: (i) $1-x, 2-y, 2-z$.

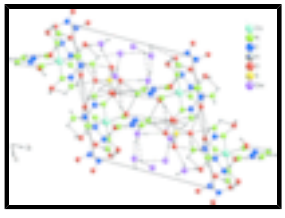

Fig. 2. Projection down [100] of the unit cell of (**I**) showing the positioning of water molecules in the channels, and the extended network of O–H···O and N–H···O bridgings (dashed lines).

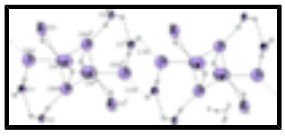

Fig. 3. View down [010] of a segment (encompassing two adjacent unit cells) of a water tape in (**I**), brought about by O–H···O bridgings within the nanochannel, with O atom numbering and ellipsoids drawn at 50% probability. Symmetry code: (iii) $1-x, 1-y, -z$

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Crystal data

$[Co(C_2H_6N_4O_2)_3]_2(C_2O_4)(SO_4)_2·12H_2O$ $Z = 1$

M_r = 1314.78	F_{000} = 678
Triclinic, $P\bar{1}$	D_x = 1.698 Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 9.4298 (10) Å	Cell parameters from 20047 reflections
b = 11.7820 (12) Å	θ = 1.8–30.0º
c = 12.8118 (13) Å	μ = 0.85 mm^{-1}
α = 65.494 (2)º	T = 193 (2) K
β = 83.088 (2)º	Prism, red
γ = 86.735 (2)º	0.25 × 0.15 × 0.10 mm
V = 1285.7 (2) Å^3	

Data collection

Bruker APEX CCD diffractometer	7453 independent reflections
Radiation source: fine-focus sealed tube	5173 reflections with $I > 2s(I)$
Monochromator: graphite	R_{int} = 0.056
T = 193(2) K	θ_{max} = 30.0º
ω scans	θ_{min} = 1.8º
Absorption correction: multi-scan (SADABS; Bruker, 2001)	h = −13→13
T_{min} = 0.816, T_{max} = 0.919	k = −16→16
20047 measured reflections	l = −18→18

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.062	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.158	$w = 1/[\sigma^2(F_o^2) + (0.0654P)^2 + 0.8468P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.10	$(\Delta/\sigma)_{max} < 0.001$
7453 reflections	$\Delta\rho_{max}$ = 0.91 e Å^{-3}
381 parameters	$\Delta\rho_{min}$ = −0.53 e Å^{-3}
12 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Co	0.72701 (5)	0.84049 (4)	0.30836 (4)	0.01529 (12)	
N24	0.6030 (3)	0.8386 (3)	0.4406 (2)	0.0171 (6)	
N31	0.5850 (3)	0.7379 (3)	0.2960 (3)	0.0189 (6)	
N14	0.8825 (3)	0.9315 (3)	0.3172 (2)	0.0178 (6)	
N11	0.8492 (3)	0.8491 (3)	0.1742 (2)	0.0189 (6)	
N34	0.8108 (3)	0.6788 (3)	0.3867 (3)	0.0202 (6)	
N21	0.6281 (3)	0.9941 (3)	0.2389 (2)	0.0189 (6)	
C23	0.4943 (3)	0.9156 (3)	0.4158 (3)	0.0174 (6)	
C12	0.9796 (3)	0.8860 (3)	0.1652 (3)	0.0181 (7)	
C22	0.5210 (3)	1.0192 (3)	0.2997 (3)	0.0191 (7)	
C13	0.9951 (3)	0.9473 (3)	0.2431 (3)	0.0180 (7)	
C32	0.6064 (4)	0.6183 (3)	0.3411 (3)	0.0207 (7)	
C33	0.7511 (4)	0.5856 (3)	0.3794 (3)	0.0215 (7)	
N27	0.3745 (3)	0.9059 (3)	0.4837 (3)	0.0241 (7)	
H27B	0.3629	0.8434	0.5522	0.029*	
H27A	0.3062	0.9620	0.4604	0.029*	
N16	1.0876 (3)	0.8716 (3)	0.0946 (3)	0.0262 (7)	
H16A	1.0736	0.8348	0.0494	0.031*	
H16B	1.1731	0.8989	0.0931	0.031*	
N17	1.1079 (3)	1.0139 (3)	0.2339 (3)	0.0261 (7)	
H17B	1.1107	1.0519	0.2800	0.031*	
H17A	1.1803	1.0203	0.1815	0.031*	
N36	0.5106 (4)	0.5328 (3)	0.3572 (3)	0.0321 (8)	
H36A	0.4242	0.5558	0.3366	0.039*	
H36B	0.5334	0.4531	0.3886	0.039*	
N26	0.4420 (3)	1.1219 (3)	0.2664 (3)	0.0265 (7)	
H26A	0.4604	1.1814	0.1969	0.032*	
H26B	0.3711	1.1307	0.3139	0.032*	
N37	0.8072 (4)	0.4730 (3)	0.4041 (3)	0.0344 (8)	
H37B	0.8927	0.4556	0.4280	0.041*	
H37A	0.7589	0.4155	0.3967	0.041*	
O28	0.5724 (3)	0.7321 (2)	0.5422 (2)	0.0207 (5)	
H28	0.6444	0.7116	0.5790	0.031*	
O35	0.4425 (2)	0.7706 (2)	0.2803 (2)	0.0227 (5)	
H35	0.4373	0.8282	0.2145	0.034*	
O15	0.8358 (3)	0.7791 (3)	0.1115 (2)	0.0252 (6)	
H15	0.7532	0.7885	0.0908	0.038*	
O38	0.9540 (3)	0.6557 (3)	0.4085 (2)	0.0253 (6)	
H38	0.9732	0.6906	0.4504	0.038*	
O18	0.8796 (3)	1.0069 (2)	0.3777 (2)	0.0227 (5)	

H18	0.8734	0.9618	0.4488	0.034*	
O25	0.6574 (3)	1.0865 (2)	0.1272 (2)	0.0235 (5)	
H25	0.7287	1.1276	0.1244	0.035*	
S	0.09424 (9)	0.24928 (9)	0.35902 (8)	0.0222 (2)	
O54	0.1647 (3)	0.1248 (2)	0.3908 (2)	0.0251 (6)	
O53	0.0244 (3)	0.2839 (3)	0.2537 (2)	0.0303 (6)	
O51	0.2052 (3)	0.3409 (3)	0.3414 (3)	0.0377 (7)	
O52	−0.0110 (3)	0.2428 (3)	0.4557 (3)	0.0329 (7)	
C41	0.5668 (3)	0.9661 (4)	0.9842 (3)	0.0222 (7)	
O42	0.6460 (3)	1.0302 (3)	0.8951 (2)	0.0261 (6)	
O43	0.5845 (3)	0.8550 (3)	1.0492 (2)	0.0274 (6)	
O1	0.8832 (3)	0.2297 (3)	0.0879 (3)	0.0339 (7)	
O2	0.7212 (3)	0.3062 (3)	0.3112 (2)	0.0296 (6)	
O3	0.2201 (4)	0.5849 (4)	0.2797 (4)	0.0605 (11)	
O4	0.4136 (7)	0.6487 (6)	0.0773 (5)	0.110 (2)	
O5A	0.8644 (7)	0.4954 (6)	−0.0420 (6)	0.0863 (19)*	0.786 (7)
O5B	0.989 (3)	0.449 (2)	−0.092 (2)	0.0863 (19)*	0.214 (7)
O6A	0.5585 (8)	0.3823 (8)	0.1243 (6)	0.0709 (17)*	0.637 (8)
O6B	0.6096 (14)	0.4563 (13)	0.1069 (11)	0.0709 (17)*	0.363 (8)
H1O	0.874 (7)	0.299 (3)	0.042 (4)	0.094 (9)*	
H2O	0.936 (6)	0.227 (6)	0.133 (4)	0.094 (9)*	
H3O	0.804 (3)	0.289 (6)	0.292 (5)	0.094 (9)*	
H4O	0.685 (6)	0.334 (6)	0.247 (4)	0.094 (9)*	
H5O	0.234 (8)	0.616 (5)	0.207 (2)	0.094 (9)*	
H6O	0.191 (7)	0.513 (3)	0.296 (5)	0.094 (9)*	
H7O	0.480 (6)	0.657 (6)	0.006 (4)	0.094 (9)*	
H8O	0.366 (6)	0.594 (5)	0.069 (6)	0.094 (9)*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co	0.0097 (2)	0.0186 (2)	0.0200 (2)	0.00156 (16)	−0.00084 (16)	−0.01075 (18)
N24	0.0145 (13)	0.0192 (14)	0.0173 (14)	0.0002 (11)	−0.0005 (11)	−0.0076 (11)
N31	0.0127 (13)	0.0198 (15)	0.0249 (15)	0.0013 (11)	−0.0013 (11)	−0.0103 (12)
N14	0.0132 (13)	0.0215 (15)	0.0239 (15)	0.0004 (11)	0.0001 (11)	−0.0152 (13)
N11	0.0162 (13)	0.0250 (15)	0.0209 (15)	−0.0005 (11)	−0.0022 (11)	−0.0147 (13)
N34	0.0161 (14)	0.0233 (15)	0.0242 (15)	0.0058 (11)	−0.0059 (11)	−0.0123 (13)
N21	0.0152 (13)	0.0212 (15)	0.0187 (14)	−0.0010 (11)	−0.0010 (11)	−0.0068 (12)
C23	0.0133 (15)	0.0219 (17)	0.0209 (17)	0.0023 (12)	−0.0022 (12)	−0.0127 (14)
C12	0.0144 (15)	0.0211 (17)	0.0202 (16)	0.0016 (12)	−0.0018 (12)	−0.0102 (14)
C22	0.0134 (15)	0.0216 (17)	0.0245 (18)	0.0003 (12)	−0.0044 (13)	−0.0111 (14)
C13	0.0136 (15)	0.0207 (17)	0.0219 (17)	0.0040 (12)	−0.0035 (12)	−0.0110 (14)
C32	0.0203 (17)	0.0244 (18)	0.0188 (17)	0.0002 (13)	−0.0007 (13)	−0.0107 (14)
C33	0.0201 (17)	0.0228 (18)	0.0250 (18)	0.0050 (13)	−0.0061 (14)	−0.0128 (15)
N27	0.0159 (14)	0.0283 (17)	0.0256 (16)	0.0050 (12)	0.0021 (12)	−0.0105 (13)
N16	0.0123 (13)	0.043 (2)	0.0336 (18)	−0.0032 (13)	0.0039 (12)	−0.0273 (16)
N17	0.0149 (14)	0.0404 (19)	0.0333 (18)	−0.0059 (13)	0.0038 (12)	−0.0264 (16)
N36	0.0270 (17)	0.0210 (16)	0.047 (2)	−0.0032 (13)	−0.0104 (15)	−0.0103 (15)

N26	0.0236 (16)	0.0239 (16)	0.0265 (17)	0.0089 (13)	−0.0015 (13)	−0.0064 (13)
N37	0.0310 (18)	0.0237 (17)	0.054 (2)	0.0104 (14)	−0.0170 (17)	−0.0198 (17)
O28	0.0161 (11)	0.0223 (13)	0.0199 (12)	0.0009 (10)	−0.0023 (9)	−0.0049 (10)
O35	0.0122 (11)	0.0242 (13)	0.0287 (14)	−0.0004 (9)	−0.0037 (10)	−0.0074 (11)
O15	0.0138 (11)	0.0390 (16)	0.0359 (15)	0.0017 (11)	−0.0035 (11)	−0.0285 (13)
O38	0.0155 (12)	0.0331 (15)	0.0365 (15)	0.0089 (10)	−0.0098 (11)	−0.0227 (13)
O18	0.0224 (12)	0.0263 (13)	0.0261 (13)	−0.0015 (10)	0.0020 (11)	−0.0184 (11)
O25	0.0217 (13)	0.0248 (13)	0.0194 (13)	−0.0037 (10)	−0.0016 (10)	−0.0040 (10)
S	0.0178 (4)	0.0249 (5)	0.0301 (5)	0.0048 (3)	−0.0093 (3)	−0.0163 (4)
O54	0.0202 (12)	0.0260 (14)	0.0330 (15)	0.0061 (10)	−0.0061 (11)	−0.0158 (12)
O53	0.0246 (14)	0.0374 (16)	0.0311 (15)	0.0051 (12)	−0.0116 (11)	−0.0147 (13)
O51	0.0317 (16)	0.0261 (15)	0.055 (2)	−0.0019 (12)	−0.0212 (14)	−0.0120 (14)
O52	0.0243 (14)	0.0493 (18)	0.0365 (16)	0.0101 (13)	−0.0081 (12)	−0.0290 (15)
C41	0.0110 (15)	0.038 (2)	0.0225 (18)	−0.0025 (14)	−0.0052 (13)	−0.0156 (16)
O42	0.0152 (12)	0.0383 (16)	0.0249 (14)	−0.0030 (11)	0.0046 (10)	−0.0143 (12)
O43	0.0189 (13)	0.0346 (15)	0.0282 (14)	−0.0003 (11)	−0.0052 (11)	−0.0118 (12)
O1	0.0319 (16)	0.0387 (17)	0.0354 (17)	−0.0066 (13)	−0.0050 (13)	−0.0183 (14)
O2	0.0196 (13)	0.0339 (16)	0.0347 (16)	0.0010 (11)	0.0004 (11)	−0.0147 (13)
O3	0.047 (2)	0.038 (2)	0.098 (3)	−0.0035 (17)	−0.010 (2)	−0.029 (2)
O4	0.139 (5)	0.105 (5)	0.084 (4)	−0.056 (4)	−0.030 (4)	−0.024 (3)

Geometric parameters (Å, °)

Co—N21	1.900 (3)	N17—H17B	0.8800
Co—N14	1.906 (3)	N17—H17A	0.8800
Co—N31	1.917 (3)	N36—H36A	0.8800
Co—N11	1.920 (3)	N36—H36B	0.8800
Co—N34	1.922 (3)	N26—H26A	0.8800
Co—N24	1.932 (3)	N26—H26B	0.8800
N24—C23	1.307 (4)	N37—H37B	0.8800
N24—O28	1.396 (4)	N37—H37A	0.8800
N31—C32	1.297 (4)	O28—H28	0.8400
N31—O35	1.391 (3)	O35—H35	0.8400
N14—C13	1.302 (4)	O15—H15	0.8400
N14—O18	1.398 (3)	O38—H38	0.8400
N11—C12	1.303 (4)	O18—H18	0.8400
N11—O15	1.388 (4)	O25—H25	0.8400
N34—C33	1.303 (4)	S—O53	1.465 (3)
N34—O38	1.400 (3)	S—O52	1.469 (3)
N21—C22	1.297 (4)	S—O51	1.477 (3)
N21—O25	1.398 (4)	S—O54	1.489 (3)
C23—N27	1.319 (4)	C41—O43	1.241 (5)
C23—C22	1.485 (5)	C41—O42	1.255 (4)
C12—N16	1.330 (4)	C41—C41^{i}	1.555 (7)
C12—C13	1.478 (5)	O1—H1O	0.79 (3)
C22—N26	1.321 (4)	O1—H2O	0.80 (3)
C13—N17	1.323 (4)	O2—H3O	0.83 (3)
C32—N36	1.327 (5)	O2—H4O	0.85 (3)
C32—C33	1.482 (5)	O3—H5O	0.84 (3)

C33—N37	1.324 (5)	O3—H6O	0.84 (3)
N27—H27B	0.8800	O4—H7O	1.01 (3)
N27—H27A	0.8800	O4—H8O	0.85 (3)
N16—H16A	0.8800	O5A—O5B	1.45 (2)
N16—H16B	0.8800	O6A—O6B	0.950 (13)
N21—Co—N14	88.61 (12)	N17—C13—C12	123.1 (3)
N21—Co—N31	96.04 (12)	N31—C32—N36	125.4 (3)
N14—Co—N31	174.13 (12)	N31—C32—C33	111.9 (3)
N21—Co—N11	97.98 (12)	N36—C32—C33	122.6 (3)
N14—Co—N11	79.99 (12)	N34—C33—N37	126.2 (3)
N31—Co—N11	95.78 (12)	N34—C33—C32	111.9 (3)
N21—Co—N34	174.77 (12)	N37—C33—C32	121.9 (3)
N14—Co—N34	95.29 (12)	C23—N27—H27B	120.0
N31—Co—N34	80.29 (12)	C23—N27—H27A	120.0
N11—Co—N34	86.15 (13)	H27B—N27—H27A	120.0
N21—Co—N24	79.98 (12)	C12—N16—H16A	120.0
N14—Co—N24	99.14 (12)	C12—N16—H16B	120.0
N31—Co—N24	85.24 (12)	H16A—N16—H16B	120.0
N11—Co—N24	177.82 (13)	C13—N17—H17B	120.0
N34—Co—N24	95.93 (12)	C13—N17—H17A	120.0
C23—N24—O28	112.3 (3)	H17B—N17—H17A	120.0
C23—N24—Co	114.8 (2)	C32—N36—H36A	120.0
O28—N24—Co	124.3 (2)	C32—N36—H36B	120.0
C32—N31—O35	113.0 (3)	H36A—N36—H36B	120.0
C32—N31—Co	117.3 (2)	C22—N26—H26A	120.0
O35—N31—Co	125.9 (2)	C22—N26—H26B	120.0
C13—N14—O18	113.8 (3)	H26A—N26—H26B	120.0
C13—N14—Co	117.9 (2)	C33—N37—H37B	120.0
O18—N14—Co	126.8 (2)	C33—N37—H37A	120.0
C12—N11—O15	112.9 (3)	H37B—N37—H37A	120.0
C12—N11—Co	116.3 (2)	N24—O28—H28	109.5
O15—N11—Co	126.0 (2)	N31—O35—H35	109.5
C33—N34—O38	113.0 (3)	N11—O15—H15	109.5
C33—N34—Co	116.2 (2)	N34—O38—H38	109.5
O38—N34—Co	125.5 (2)	N14—O18—H18	109.5
C22—N21—O25	114.6 (3)	N21—O25—H25	109.5
C22—N21—Co	118.1 (2)	O53—S—O52	110.50 (16)
O25—N21—Co	127.3 (2)	O53—S—O51	110.43 (18)
N24—C23—N27	125.7 (3)	O52—S—O51	109.38 (18)
N24—C23—C22	111.6 (3)	O53—S—O54	110.55 (16)
N27—C23—C22	122.7 (3)	O52—S—O54	108.07 (17)
N11—C12—N16	124.9 (3)	O51—S—O54	107.84 (16)
N11—C12—C13	112.0 (3)	O43—C41—O42	126.7 (3)
N16—C12—C13	123.0 (3)	O43—C41—C41^{i}	117.1 (4)
N21—C22—N26	126.4 (3)	O42—C41—C41^{i}	116.2 (4)
N21—C22—C23	111.4 (3)	H1O—O1—H2O	111 (4)
N26—C22—C23	122.2 (3)	H3O—O2—H4O	101 (4)
N14—C13—N17	125.2 (3)	H5O—O3—H6O	103 (4)

N14—C13—C12	111.7 (3)	H7O—O4—H8O	90 (3)

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N16—H16B···O42^{ii}	0.88	1.99	2.864 (4)	172
N17—H17B···O54^{iii}	0.88	2.06	2.926 (4)	167
N17—H17A···O42^{ii}	0.88	2.00	2.835 (4)	159
N27—H27A···O54^{iv}	0.88	2.20	3.069 (4)	170
N26—H26B···O54^{iv}	0.88	2.06	2.900 (4)	158
N36—H36B···O28^{v}	0.88	2.23	2.952 (4)	139
N37—H37B···O52^{vi}	0.88	2.51	2.998 (4)	115
N37—H37B···O38^{vii}	0.88	2.54	3.321 (4)	149
O15—H15···O43^{viii}	0.84	1.77	2.584 (4)	162
O18—H18···O54^{v}	0.84	1.88	2.709 (4)	172
O18—H18···O52^{v}	0.84	2.56	3.124 (4)	125
O28—H28···O51^{v}	0.84	1.78	2.617 (4)	177
O35—H35···O42^{ix}	0.84	1.88	2.667 (4)	155
O35—H35···O43^{viii}	0.84	2.30	2.881 (4)	127
O38—H38···O52^{v}	0.84	1.76	2.602 (4)	177
N16—H16A···O1^{x}	0.88	2.18	3.016 (4)	159
N27—H27B···O2^{v}	0.88	2.02	2.865 (4)	162
N36—H36A···O3	0.88	2.10	2.971 (5)	170
N37—H37A···O2	0.88	2.07	2.879 (5)	152
O25—H25···O1^{iv}	0.84	1.83	2.662 (4)	169
O1—H2O···O53^{vi}	0.80 (3)	2.19 (4)	2.931 (4)	156 (6)
O2—H3O···O53^{vi}	0.83 (3)	2.08 (3)	2.890 (4)	165 (6)
O3—H6O···O51	0.84 (3)	1.87 (4)	2.653 (5)	157 (7)
O1—H1O···O5A	0.79 (3)	2.11 (3)	2.879 (7)	164 (6)
O2—H4O···O6A	0.85 (3)	1.96 (3)	2.799 (8)	166 (6)
O3—H5O···O4	0.84 (3)	2.16 (6)	2.831 (8)	137 (7)
O4—H7O···O6A^{xi}	1.01 (3)	1.99 (6)	2.736 (9)	128 (5)
O4—H8O···O5A^{xi}	0.85 (3)	2.59 (4)	3.375 (8)	153 (6)

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

Fig. 1

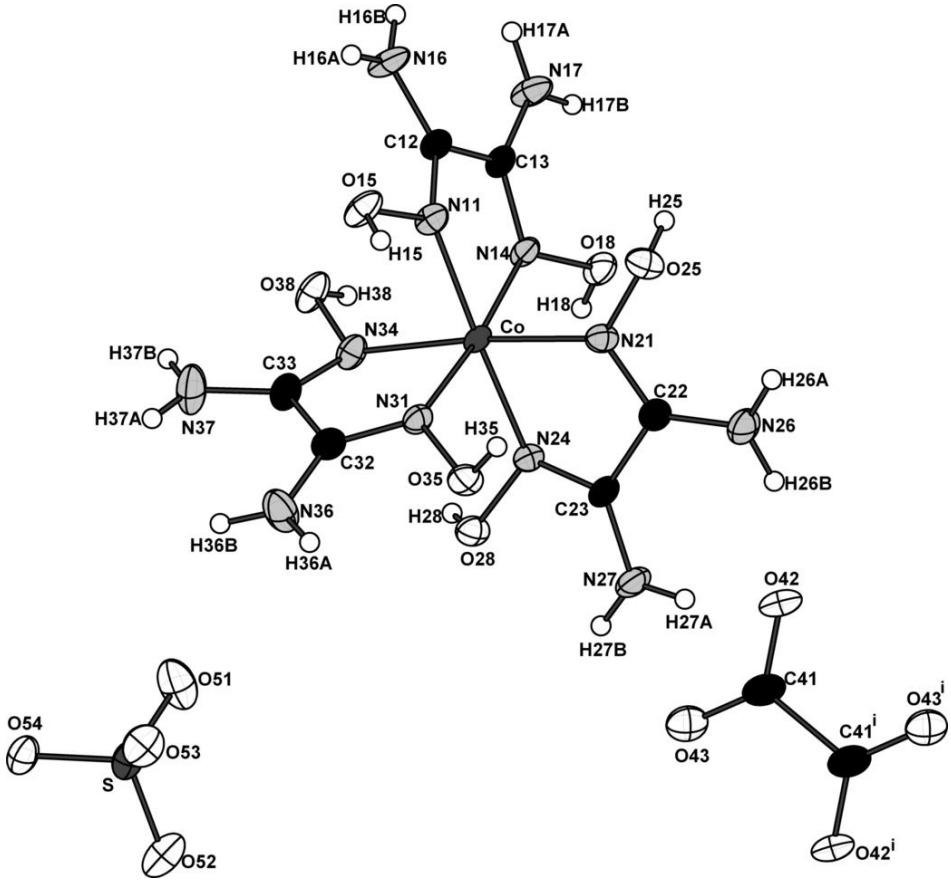

H16B
H17A
H16A
N16
N17
H17B
C12
C13
O15
N11
H25
H15
N14
O18
O25
O38
H38
H18
N34
Co
N21
H37B
C33
C22
H26A
N31
H37A
N37
H35
N24
N26
C32
O35
H26B
N36
H28
C23
H36B
O28
H36A
N27
O42
H27A
H27B
C41
O43i
O51
O54
O53
C41i
O43
S
O52
O42i

Fig. 2

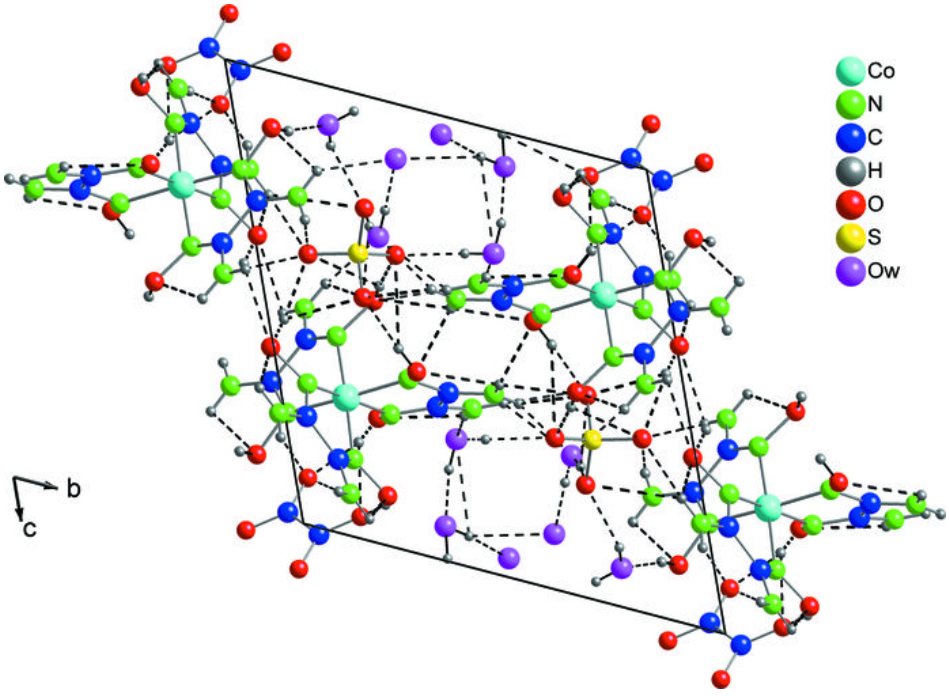
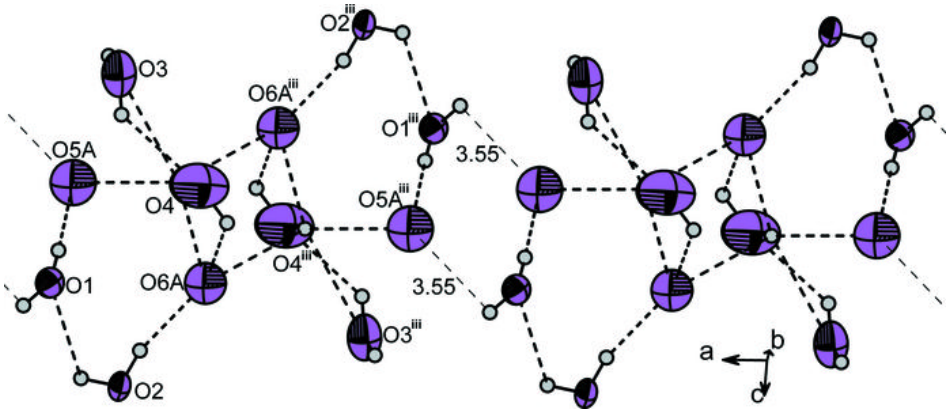


Fig. 3


O2iii
O3
O6Aiii
O1iii
3.55
O5A
O4
O5Aiii
O4iii
O1
O6A
3.55
O3iii
O2
a
b
c