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Bis[(*R*)-1-phenylethylaminium] μ-oxalato-$\kappa^4O,O':O''O'''$-bis[diaqua(oxalato-κ^2O,O')cobaltate(II)]

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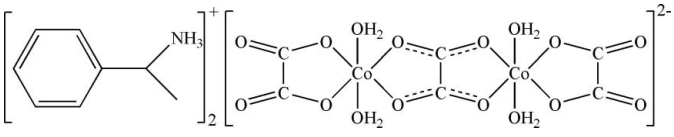
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.012 Å; R factor = 0.033; wR factor = 0.102; data-to-parameter ratio = 11.8.

The title compound, $(C_8H_{12}N)_2[Co_2(C_2O_4)_3(H_2O)_4]$, was prepared under hydrothermal conditions. The structure consists of chiral organic cations and complex anions. Hydrogen bonds and C–H$\cdots\pi$(aryl) interactions give a herring-bone arrangement of the cations along the *a* axis.

Related literature

For a related structure, see: Shan & Huang (2001).



Experimental

Crystal data

$(C_8H_{12}N)_2[Co_2(C_2O_4)_3(H_2O)_4]$
M_r = 698.36
Monoclinic, $C2$
a = 10.973 (2) Å
b = 7.5560 (15) Å
c = 17.067 (3) Å
β = 90.33 (3)°
V = 1415.0 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.25 mm^{-1}
T = 296 K
0.24 × 0.12 × 0.04 mm

Data collection

Siemens SMART CCD diffractometer
Absorption correction: none
3658 measured reflections
2268 independent reflections
1938 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.102
S = 1.13
2268 reflections
193 parameters
38 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.52 e Å^{-3}
Absolute structure: Flack (1983), 850 Friedel pairs
Flack parameter: 0.11 (3)

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N1—H1$C\cdots$O4	0.89	1.95	2.843 (8)	176
N1—H1$B\cdots$O3^{i}	0.89	2.05	2.918 (8)	166
N1—H1$A\cdots$O6^{ii}	0.89	2.23	2.976 (7)	141
N1—H1$A\cdots$O5^{ii}	0.89	2.21	2.973 (7)	143
O8—H8$B\cdots$O2^{iii}	0.93	1.81	2.737 (9)	172
O8—H8$A\cdots$O1^{iv}	0.96	1.77	2.728 (9)	172
O7—H7$B\cdots$O5^{v}	0.90	1.82	2.678 (8)	157
O7—H7$A\cdots$O6^{ii}	0.94	1.81	2.712 (8)	159

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HY2056).

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supplementary materials

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Bis[(*R*)-1-phenylethylaminium] μ-oxalato-$\kappa^4O,O':O''O'''$-bis[diaqua(oxalato-κ^2O,O')cobaltate(II)]

X. Yang, J. Li, H.-W. Wang, X.-H. Zhao and Y.-K. Shan

Comment

The crystal structure of the title compound, (I)(Fig. 1), consists of bicobalt oxalate complex anions $[Co_2(C_2O_4)_3(H_2O)_4]^{2-}$ and organic cations $(C_8H_{12}N)^+$, joined into a two-dimensional sheet by hydrogen bonds. Owing to the presence of the chiral organic cation, the compound crystallizes in the polar space group C2. In the anion, there are two types of oxalates. One as tetradentate ligand bridges two Co atoms, each is coordinated by another type of oxalate as bidentate ligand in the same plane. Each Co atom is also bonded by two water molecules above and below the equatorial plane to produce a negative step-lamella. The bond angles around the Co atom range from 81.01 (9)° to 100.2 (2) °, and from 178.1 (2)° to 178.7 (2)°. The Co—O distances span from 2.062 (5)Å to 2.114 (3) Å. The organic cation contains a stereogenic center in its *R* configuration and has a normal structure similar to that described elsewhere (Shan & Huang, 2001). The cations are anchored in the pockets on the both faces of the puckered anionic lamella through hydrogen bonds with the distances ranging from 2.678 (8)Å to 2.976 (7)Å (Table 1), thus resulting in the formation of cation–anion–cation slab-sandwich layers with an arrangement of the cations held together by close C–H···π(aryl) interactions in a herring bone motif (Fig.2).

Experimental

Compound (I) was prepared by the hydrothermal reaction of $Co(C_2O_4)\cdot 2H_2O$(0.130 g, 0.71 mmol), (*R*)-(+)-1-phenylethylamine (0.062 g, 0.51 mmol) and water (0.6 ml) at 383 K for 3 d in a sealed thick-walled Pyrex tube. Light pink crystals of (I) were obtained in 40–50% yield.

Refinement

H atoms on C and N atoms were positioned geometrically and refined as riding atoms, with C—H = 0.93Å (aryl), 0.98Å (CH) and 0.96Å (CH_3) and N—H = 0.89 Å and U_{iso}(H) = 1.2U_{eq}(C,*N*). H atoms on water molecules were located in a difference Fourier map and fixed with U_{iso}(H) = 1.2U_{eq}(O).

Figures

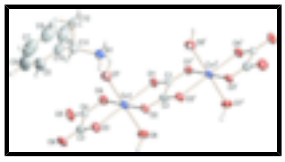


Fig. 1. The structure of (I), with atom labels and 30% probability displacement ellipsoids for non-H atoms. [Symmetry code: (i) $1-x, y, 1-z$.]

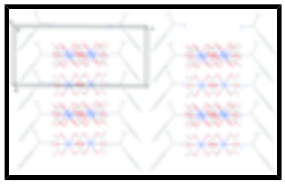
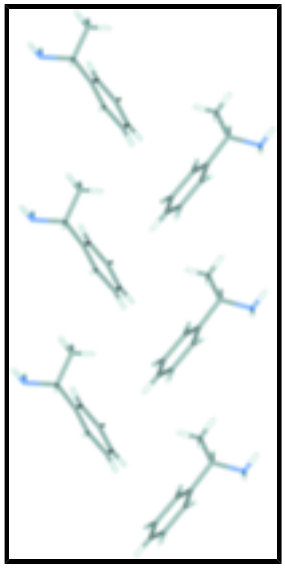


Fig. 2. A packing diagram of (I), showing the cation–anion–cation slab-sandwich layers with a herring-bone arrangement of the cations.



(*R*)-1-Phenylethylaminium μ-oxalato-κ^4*O*,*O*':*O*''*O*'''-bis[diaqua(oxalato-κ^2*O*,*O*')cobalt(II)]

Crystal data

$(C_8H_{12}N)_2[Co_2(C_2O_4)_3(H_2O)_4]$	F_{000} = 720
M_r = 698.36	D_x = 1.639 Mg m^{-3}
Monoclinic, *C*2	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: C 2y	Cell parameters from 4453 reflections
a = 10.973 (2) Å	θ = 1.2–27.1º
b = 7.5560 (15) Å	μ = 1.25 mm^{-1}
c = 17.067 (3) Å	*T* = 296 K
β = 90.33 (3)º	Plate, pink
V = 1415.0 (5) Å^3	0.24 × 0.12 × 0.04 mm
Z = 2	

Data collection

Siemens Quantum CCD diffractometer	1938 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.020
Monochromator: graphite	θ_{max} = 25.5º
T = 296 K	θ_{min} = 1.2º
ω scans	*h* = −13→10
Absorption correction: none	*k* = −9→8
3658 measured reflections	*l* = −20→19
2268 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.033$	$w = 1/[\sigma^2(F_o^2) + (0.0589P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.102$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.13$	$\Delta\rho_{max} = 0.45$ e Å^{-3}
2268 reflections	$\Delta\rho_{min} = -0.52$ e Å^{-3}
193 parameters	Extinction correction: none
38 restraints	Absolute structure: Flack (1983), 850 Fridel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.11 (3)
Secondary atom site location: difference Fourier map	

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Co	0.29291 (4)	0.98018 (18)	0.41036 (3)	0.02253 (17)
O1	0.4159 (6)	0.8027 (5)	0.4631 (3)	0.0242 (13)
O2	0.4163 (5)	1.1583 (6)	0.4624 (3)	0.0266 (13)
O3	0.1732 (5)	1.1586 (6)	0.3606 (3)	0.0262 (14)
O4	0.1735 (6)	0.8017 (6)	0.3563 (3)	0.0286 (15)
O5	−0.0023 (6)	0.8057 (7)	0.2907 (3)	0.0379 (16)
O6	−0.0019 (5)	1.1607 (6)	0.2925 (3)	0.0341 (15)
O7	0.3966 (2)	0.9858 (9)	0.30858 (15)	0.0317 (6)
H7A	0.4480	0.8882	0.2992	0.038*
H7B	0.4491	1.0760	0.3010	0.038*
O8	0.1915 (2)	0.9809 (10)	0.51531 (15)	0.0314 (6)
H8A	0.1465	1.0892	0.5220	0.038*
H8B	0.1487	0.8760	0.5244	0.038*
N1	0.2561 (3)	0.4834 (10)	0.28541 (18)	0.0278 (7)
H1A	0.3371	0.4822	0.2833	0.042*
H1B	0.2306	0.3950	0.3156	0.042*
H1C	0.2312	0.5860	0.3054	0.042*
C1	0.5000	0.8799 (16)	0.5000	0.019 (2)
C2	0.5000	1.0861 (15)	0.5000	0.021 (3)
C4	0.0833 (8)	0.8815 (10)	0.3241 (4)	0.0194 (16)
C3	0.0844 (9)	1.0873 (11)	0.3269 (5)	0.0275 (19)
C9	0.3493 (6)	0.6631 (9)	0.1348 (3)	0.0599 (19)
H9	0.4134	0.5978	0.1560	0.072*
C8	0.3753 (9)	0.8048 (11)	0.0842 (4)	0.081 (2)
H8	0.4553	0.8345	0.0722	0.098*
C7	0.2813 (10)	0.8964 (11)	0.0536 (5)	0.086 (3)
H7	0.2975	0.9898	0.0198	0.103*
C6	0.1660 (9)	0.8583 (11)	0.0701 (4)	0.083 (3)

H6	0.1033	0.9241	0.0477	0.100*
C5	0.1388 (7)	0.7174 (10)	0.1216 (4)	0.067 (2)
H5	0.0582	0.6921	0.1337	0.080*
C10	0.2306 (8)	0.6182 (9)	0.1538 (4)	0.0395 (18)
C11	0.2044 (4)	0.4618 (12)	0.2043 (2)	0.0307 (12)
H11	0.1157	0.4517	0.2089	0.037*
C12	0.2513 (8)	0.2897 (8)	0.1688 (4)	0.045 (2)
H12A	0.2189	0.2763	0.1168	0.068*
H12B	0.2258	0.1917	0.2005	0.068*
H12C	0.3387	0.2928	0.1667	0.068*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co	0.0218 (3)	0.0158 (3)	0.0299 (3)	−0.0009 (6)	−0.00417 (19)	0.0013 (6)
O1	0.034 (3)	0.010 (3)	0.029 (3)	−0.001 (3)	−0.008 (3)	0.003 (2)
O2	0.018 (3)	0.023 (3)	0.039 (3)	0.002 (3)	−0.006 (2)	0.005 (3)
O3	0.023 (3)	0.018 (3)	0.038 (3)	−0.004 (2)	−0.007 (3)	0.002 (2)
O4	0.033 (4)	0.016 (3)	0.037 (3)	−0.001 (3)	−0.007 (3)	−0.001 (2)
O5	0.038 (4)	0.031 (4)	0.044 (3)	−0.004 (3)	−0.020 (3)	0.005 (3)
O6	0.032 (4)	0.021 (3)	0.050 (3)	0.010 (3)	−0.006 (3)	0.000 (3)
O7	0.0339 (13)	0.0203 (14)	0.0412 (15)	−0.001 (3)	0.0077 (11)	0.007 (3)
O8	0.0361 (13)	0.0150 (12)	0.0432 (15)	−0.001 (3)	0.0063 (11)	−0.002 (3)
N1	0.0297 (15)	0.0213 (15)	0.0323 (16)	−0.002 (4)	0.0005 (13)	0.003 (4)
C1	0.011 (5)	0.029 (5)	0.017 (4)	0.000	−0.001 (4)	0.000
C2	0.030 (6)	0.005 (4)	0.029 (5)	0.000	0.004 (5)	0.000
C4	0.014 (4)	0.024 (4)	0.019 (4)	0.001 (3)	−0.002 (3)	0.000 (3)
C3	0.039 (5)	0.015 (3)	0.029 (4)	0.004 (4)	0.002 (4)	−0.001 (3)
C9	0.073 (5)	0.070 (5)	0.037 (3)	−0.032 (4)	0.000 (3)	0.005 (3)
C8	0.109 (6)	0.082 (5)	0.053 (4)	−0.043 (5)	0.008 (4)	0.003 (4)
C7	0.149 (7)	0.053 (4)	0.056 (4)	−0.010 (5)	0.007 (5)	−0.001 (3)
C6	0.132 (7)	0.062 (4)	0.056 (4)	0.045 (5)	−0.011 (5)	0.010 (4)
C5	0.086 (5)	0.064 (4)	0.050 (4)	0.038 (4)	−0.003 (4)	0.009 (4)
C10	0.055 (4)	0.031 (4)	0.033 (3)	−0.003 (3)	−0.007 (3)	−0.008 (3)
C11	0.0267 (18)	0.032 (4)	0.034 (2)	−0.005 (3)	−0.0019 (16)	0.001 (3)
C12	0.076 (5)	0.027 (4)	0.033 (4)	0.002 (4)	−0.003 (4)	−0.010 (3)

Geometric parameters (Å, °)

Co—O3	2.062 (5)	C1—C2	1.558 (6)
Co—O7	2.083 (3)	C2—O2^{i}	1.243 (7)
Co—O4	2.091 (5)	C4—C3	1.556 (5)
Co—O1	2.102 (5)	C9—C10	1.387 (10)
Co—O2	2.103 (6)	C9—C8	1.406 (10)
Co—O8	2.114 (3)	C9—H9	0.9300
O1—C1	1.257 (8)	C8—C7	1.345 (12)
O2—C2	1.243 (7)	C8—H8	0.9300
O3—C3	1.250 (10)	C7—C6	1.329 (12)

O4—C4	1.281 (9)	C7—H7	0.9300
O5—C4	1.237 (9)	C6—C5	1.414 (11)
O6—C3	1.242 (10)	C6—H6	0.9300
O7—H7A	0.9420	C5—C10	1.368 (10)
O7—H7B	0.9020	C5—H5	0.9300
O8—H8A	0.9630	C10—C11	1.491 (11)
O8—H8B	0.9349	C11—C12	1.525 (11)
N1—C11	1.503 (5)	C11—H11	0.9800
N1—H1A	0.8900	C12—H12A	0.9600
N1—H1B	0.8900	C12—H12B	0.9600
N1—H1C	0.8900	C12—H12C	0.9600
C1—O1^{i}	1.257 (8)		
O3—Co—O7	89.6 (2)	O5—C4—O4	124.3 (8)
O3—Co—O4	81.01 (9)	O5—C4—C3	118.9 (9)
O7—Co—O4	89.4 (2)	O4—C4—C3	116.8 (9)
O3—Co—O1	178.7 (2)	O6—C3—O3	128.0 (8)
O7—Co—O1	91.0 (2)	O6—C3—C4	115.2 (9)
O4—Co—O1	100.2 (2)	O3—C3—C4	116.8 (9)
O3—Co—O2	99.4 (2)	C10—C9—C8	121.6 (8)
O7—Co—O2	89.2 (2)	C10—C9—H9	119.2
O4—Co—O2	178.5 (3)	C8—C9—H9	119.2
O1—Co—O2	79.45 (9)	C7—C8—C9	118.2 (8)
O3—Co—O8	90.6 (2)	C7—C8—H8	120.9
O7—Co—O8	178.1 (2)	C9—C8—H8	120.9
O4—Co—O8	92.5 (2)	C6—C7—C8	122.4 (8)
O1—Co—O8	88.7 (2)	C6—C7—H7	118.8
O2—Co—O8	88.9 (2)	C8—C7—H7	118.8
C1—O1—Co	112.7 (6)	C7—C6—C5	119.9 (8)
C2—O2—Co	114.2 (6)	C7—C6—H6	120.0
C3—O3—Co	113.6 (5)	C5—C6—H6	120.0
C4—O4—Co	111.5 (5)	C10—C5—C6	120.4 (8)
Co—O7—H7A	117.1	C10—C5—H5	119.8
Co—O7—H7B	119.1	C6—C5—H5	119.8
H7A—O7—H7B	100.6	C5—C10—C9	117.5 (7)
Co—O8—H8A	112.0	C5—C10—C11	121.5 (7)
Co—O8—H8B	113.9	C9—C10—C11	120.9 (7)
H8A—O8—H8B	116.2	C10—C11—N1	111.9 (6)
C11—N1—H1A	109.5	C10—C11—C12	112.3 (4)
C11—N1—H1B	109.5	N1—C11—C12	109.4 (6)
H1A—N1—H1B	109.5	C10—C11—H11	107.7
C11—N1—H1C	109.5	N1—C11—H11	107.7
H1A—N1—H1C	109.5	C12—C11—H11	107.7
H1B—N1—H1C	109.5	C11—C12—H12A	109.5
O1—C1—O1^{i}	124.7 (12)	C11—C12—H12B	109.5
O1—C1—C2	117.6 (6)	H12A—C12—H12B	109.5
O1^{i}—C1—C2	117.6 (6)	C11—C12—H12C	109.5
O2^{i}—C2—O2	128.0 (12)	H12A—C12—H12C	109.5
O2^{i}—C2—C1	116.0 (6)	H12B—C12—H12C	109.5

O2—C2—C1	116.0 (6)

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1C···O4	0.89	1.95	2.843 (8)	176
N1—H1B···O3^{ii}	0.89	2.05	2.918 (8)	166
N1—H1A···O6^{iii}	0.89	2.23	2.976 (7)	141
N1—H1A···O5^{iii}	0.89	2.21	2.973 (7)	143
O8—H8B···O2^{iv}	0.93	1.81	2.737 (9)	172
O8—H8A···O1^{v}	0.96	1.77	2.728 (9)	172
O7—H7B···O5^{vi}	0.90	1.82	2.678 (8)	157
O7—H7A···O6^{iii}	0.94	1.81	2.712 (8)	159

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

Fig. 1

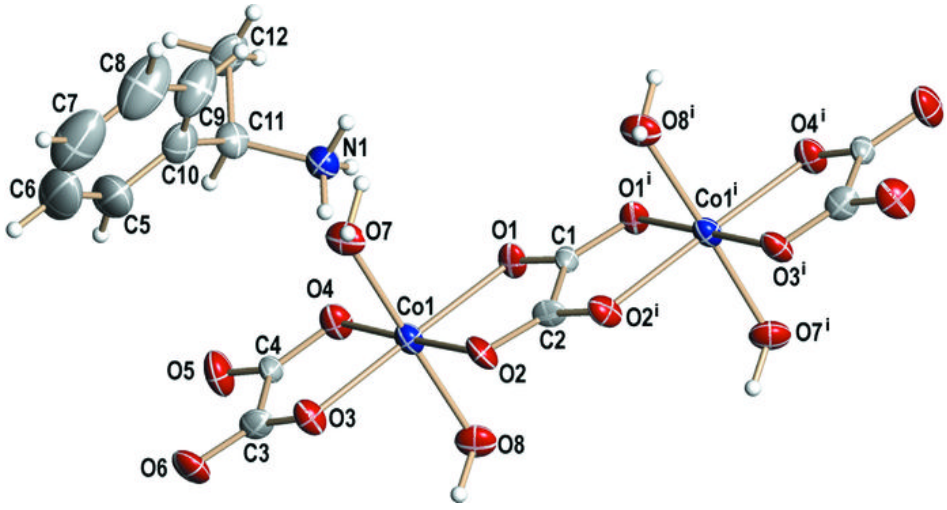

C12
C8
C7
C9
C11
N1
C10
C6
C5
O7
O1
C1
O1i
Co1i
O8i
O4i
O3i
O4
Co1
O2i
C2
O7i
C4
O5
O2
O3
C3
O6
O8

Fig. 2

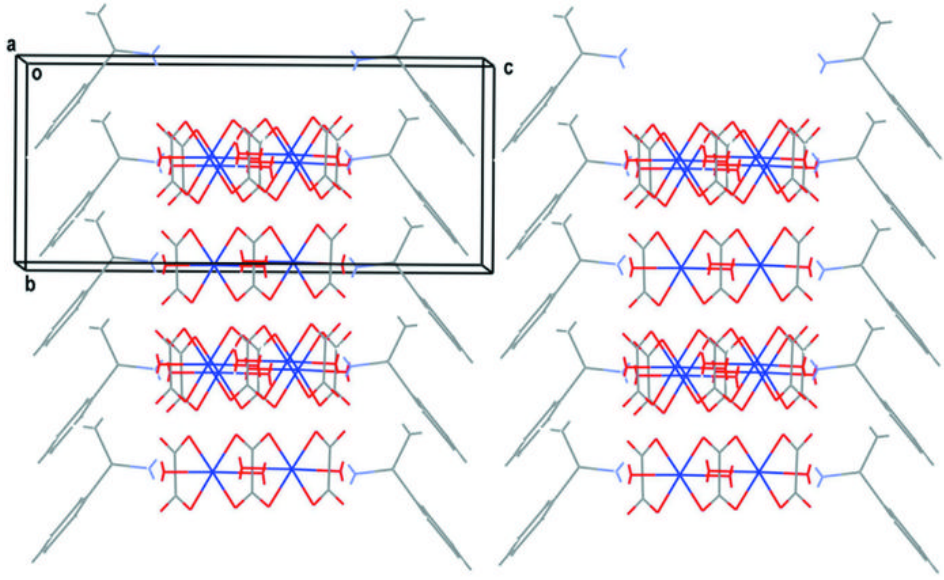


Fig. 3