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Poly[[aqua(μ_2-oxalato)(μ_2-2-oxidopyridinium-3-carboxylato)dysprosium(III)] monohydrate]

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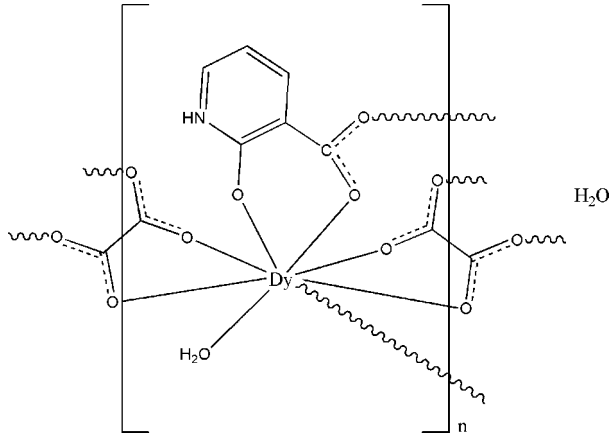
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.011 Å; R factor = 0.030; wR factor = 0.077; data-to-parameter ratio = 11.6.

In the title complex, $\{[Dy(C_6H_4NO_3)(C_2O_4)(H_2O)]\cdot H_2O\}_n$, the Dy^{III} ion is coordinated by seven O atoms from two 2-oxidopyridinium-3-carboxylate ligands, two oxalate ligands and one water molecule, displaying a distorted bicapped trigonal-prismatic geometry. The carboxylate groups of the 2-oxidopyridinium-3-carboxylate and oxalate ligands link dysprosium metal centres, forming layers parallel to (100). These layers are further connected by intermolecular O—H···O hydrogen-bonding interactions involving the coordinated water molecules, forming a three-dimensional supramolecular network. The uncoordinated water molecule is involved in N—H···O and O—H···O hydrogen-bonding interactions within the layer.

Related literature

For background to the molecular self-assembly of supramolecular architectures, see: Moulton & Zaworotko (2001); Zeng *et al.* (2007).



Experimental

Crystal data

$[Dy(C_6H_4NO_3)(C_2O_4)(H_2O)]\cdot H_2O$
M_r = 424.65
Triclinic, $P\overline{1}$
a = 6.5359 (15) Å
b = 9.561 (2) Å
c = 9.734 (2) Å
α = 71.906 (2)°
β = 78.800 (3)°
γ = 80.305 (2)°
V = 563.4 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 6.68 mm^{-1}
T = 296 (2) K
0.17 × 0.16 × 0.14 mm

Data collection

Bruker APEXII area-detector diffractometer
Absorption correction: multi-scan (*APEX2*; Bruker, 2004)
T_{min} = 0.397, T_{max} = 0.455
(expected range = 0.342–0.393)
2926 measured reflections
2003 independent reflections
1888 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.077
S = 1.09
2003 reflections
172 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 2.29 e Å^{-3}
$\Delta\rho_{min}$ = −1.55 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2W	0.86	1.99	2.785 (9)	154
O1W—H1W···O2^{i}	0.85	1.94	2.732 (7)	155
O1W—H2W···O4^{ii}	0.85	2.07	2.751 (7)	137
O2W—H4W···O1^{iii}	0.85	2.26	3.080 (8)	163
O2W—H4W···O6^{iii}	0.85	2.36	2.878 (8)	120

Symmetry codes: (i) $-x+2, -y+1, -z+2$; (ii) $x+1, y, z$; (iii) $x, y, z-1$.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

The authors acknowledge South China Normal University for supporting this work.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2421).

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Zeng, R.-H., Qiu, Y.-C., Cai, Y.-P., Wu, J.-Z. & Deng, H. (2007). *Acta Cryst.* E**63**, m1666.

supplementary materials

Acta Cryst. (2009). E**65**, m177-m178 [**doi:10.1107/S1600536809000580**]

Poly[[aqua(μ_2-oxalato)(μ_2-2-oxidopyridinium-3-carboxylato)dysprosium(III)] monohydrate]

C.-D. Huang, J.-X. Huang, Y.-Y. Wu, Y.-Y. Lian and R.-H. Zeng

Comment

Molecular self-assembly of supramolecular architectures has received much attention during recent decades (Zeng *et al.*, 2007; Moulton & Zaworotko, 2001). The structures and properties of such systems depend on the coordination and geometric preferences of both the central metal ions and the bridging building blocks, as well as the influence of weaker non-covalent interactions, such as hydrogen bonds and π-π stacking interactions. Recently, we obtained the title coordination polymer, which was synthesized under hydrothermal conditions.

In the structure of the title compound, each Dy^{III} centre is in a bicapped trigonal prismatic geometry, defined by seven oxygen atoms from two 2-oxidopyridinium-3-carboxylate ligands, one oxalate ligand, and one water molecule Fig. 1. The Dy^{III} ions are linked by 2-oxidopyridinium-3-carboxylate ligands and oxalate ligands to form a layer in the *bc* plane, and the adjacent Dy···Dy separations are 5.858 (4), 6.186 (5) and 6.239 Å, respectively. The layers are further connected by untermolecular O—H···O hydrogen bonding interactions inolving the coordinated water molecules to form a three-dimensional supramolecular network (Table 1, Fig. 2). Within each layer, free water molecules further link the complexes through N-H···O and O-H···O bonding interactions (Table 1).

Experimental

A mixture of Dy_2O_3 (0.375 g; 1 mmol), 2-oxynicotinic acid (0.127 g; 1 mmol), oxalic acid (0.09 g; 1 mmol), water (10 ml) in the presence of HNO_3 (0.024 g; 0.385 mmol) was stirred vigorously for 20 min and then sealed in a Teflon-lined stainless-steel autoclave (20 ml, capacity). The autoclave was heated and maintained at 446 K for 2 days, and then cooled to room temperature at 5 K h^{-1} and obtained the colorless block crystals.

Refinement

Water H atoms were tentatively located in difference Fourier maps and were refined with distance restraints of O–H = 0.85 Å and H···H = 1.39 Å, and with U_{iso}(H) = 1.5 U_{eq}(O), and then were treated as riding mode. H atoms attached to C and N atoms were placed at calculated positions and were treated as riding on their parent atoms with C—H = 0.93 Å, and N-H= 0.86Å with U_{iso}(H) = 1.2 U_{eq}(C,N).

Figures

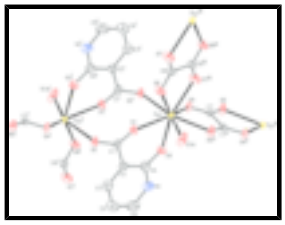


Fig. 1. The molecular structure showing the atomic-numbering scheme. Displacement ellipsoids drawn at the 30% probability level. Symmetry codes: (i)1 - *x*, 1 - *y*, 2 - *z*; (ii)1 - *x*, 2 - *y*, 1 - *z*; (iii)1 - *x*, 2 - *y*, 2 - *z*.

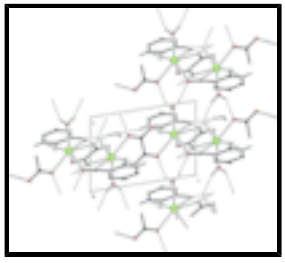


Fig. 2. A view of the three-dimensional supramolecular network. Hydrogen bonds are shown as dashed lines.

Poly[[aqua(μ$_2$-oxalato)(μ$_2$-2-oxidopyridinium-3- carboxylato)dysprosium(III)] monohydrate]

Crystal data

$[Dy(C_6H_4NO_3)(C_2O_4)(H_2O)]\cdot H_2O$	$Z = 2$
$M_r = 424.65$	$F_{000} = 402$
Triclinic, $P\overline{1}$	$D_x = 2.503$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 6.5359$ (15) Å	Cell parameters from 6377 reflections
$b = 9.561$ (2) Å	$\theta = 1.7$–28.0º
$c = 9.734$ (2) Å	$\mu = 6.68$ mm^{-1}
$\alpha = 71.906$ (2)º	$T = 296$ (2) K
$\beta = 78.800$ (3)º	Block, colourless
$\gamma = 80.305$ (2)º	0.17 × 0.16 × 0.14 mm
$V = 563.4$ (2) Å^3	

Data collection

Bruker APEXII area-detector diffractometer	2003 independent reflections
Radiation source: fine-focus sealed tube	1888 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.021$
$T = 296(2)$ K	$\theta_{max} = 25.2$º
φ and ω scans	$\theta_{min} = 2.2$º
Absorption correction: multi-scan (APEX2; Bruker, 2004)	$h = -7\rightarrow7$
$T_{min} = 0.397$, $T_{max} = 0.455$	$k = -11\rightarrow9$
2926 measured reflections	$l = -11\rightarrow7$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.030$	H-atom parameters constrained
$wR(F^2) = 0.077$	$w = 1/[\sigma^2(F_o^2) + (0.0379P)^2 + 2.6561P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.09$	$(\Delta/\sigma)_{max} = 0.001$
2003 reflections	$\Delta\rho_{max} = 2.29$ e Å^{-3}
172 parameters	$\Delta\rho_{min} = -1.55$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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.

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}
Dy1	0.59123 (5)	0.79001 (3)	0.80042 (3)	0.01511 (12)
O4	0.3353 (7)	0.8866 (5)	0.6354 (5)	0.0200 (10)
O1	0.7142 (8)	0.5573 (5)	0.9516 (5)	0.0228 (10)
O7	0.5714 (9)	0.9762 (6)	1.1684 (5)	0.0332 (13)
C6	0.7318 (12)	0.4932 (8)	0.6786 (8)	0.0255 (15)
O3	0.6553 (9)	0.6282 (5)	0.6621 (5)	0.0276 (11)
C8	0.5595 (11)	0.9416 (7)	1.0579 (7)	0.0205 (14)
O6	0.6333 (8)	0.8228 (5)	1.0278 (5)	0.0278 (11)
C7	0.3857 (10)	0.9806 (7)	0.5168 (7)	0.0162 (13)
O5	0.2701 (8)	1.0470 (5)	0.4234 (5)	0.0243 (11)
C1	0.7310 (10)	0.4252 (8)	0.9513 (7)	0.0204 (14)
N1	0.7737 (11)	0.4446 (7)	0.5575 (7)	0.0350 (15)
H1	0.7413	0.5052	0.4768	0.042*
C2	0.7787 (11)	0.3863 (7)	0.8109 (7)	0.0222 (14)
C3	0.8671 (14)	0.2456 (9)	0.8087 (9)	0.0353 (18)
H3	0.8970	0.1760	0.8954	0.042*
C5	0.8633 (15)	0.3065 (10)	0.5567 (10)	0.044 (2)
H5	0.8909	0.2816	0.4693	0.053*

C4	0.9127 (16)	0.2052 (10)	0.6788 (10)	0.051 (3)
H4	0.9756	0.1107	0.6775	0.061*
O2	0.7138 (8)	0.3183 (5)	1.0690 (5)	0.0232 (10)
O1W	0.9535 (9)	0.7979 (7)	0.7825 (6)	0.0398 (14)
H1W	1.0327	0.7410	0.8417	0.060*
H2W	1.0234	0.8615	0.7177	0.060*
O2W	0.7275 (12)	0.5642 (8)	0.2640 (7)	0.0591 (19)
H3W	0.6854	0.6442	0.2877	0.089*
H4W	0.7313	0.5812	0.1725	0.089*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Dy1	0.01446 (19)	0.01602 (18)	0.01286 (17)	−0.00079 (12)	−0.00214 (11)	−0.00175 (12)
O4	0.018 (3)	0.023 (2)	0.015 (2)	−0.007 (2)	−0.0030 (18)	0.0031 (18)
O1	0.029 (3)	0.020 (2)	0.017 (2)	−0.001 (2)	−0.0044 (19)	−0.0022 (19)
O7	0.046 (4)	0.029 (3)	0.027 (3)	0.009 (2)	−0.018 (2)	−0.011 (2)
C6	0.027 (4)	0.026 (4)	0.024 (4)	−0.006 (3)	0.000 (3)	−0.009 (3)
O3	0.038 (3)	0.025 (3)	0.019 (2)	0.003 (2)	−0.007 (2)	−0.008 (2)
C8	0.018 (4)	0.019 (3)	0.022 (3)	0.000 (3)	−0.002 (3)	−0.004 (3)
O6	0.035 (3)	0.023 (3)	0.026 (3)	0.008 (2)	−0.013 (2)	−0.009 (2)
C7	0.012 (4)	0.016 (3)	0.018 (3)	0.002 (3)	0.000 (2)	−0.004 (3)
O5	0.019 (3)	0.027 (3)	0.021 (2)	−0.006 (2)	−0.0065 (19)	0.005 (2)
C1	0.008 (3)	0.027 (4)	0.022 (3)	−0.006 (3)	−0.001 (2)	0.000 (3)
N1	0.041 (4)	0.037 (4)	0.028 (3)	−0.004 (3)	0.001 (3)	−0.015 (3)
C2	0.016 (4)	0.022 (3)	0.027 (4)	−0.003 (3)	0.001 (3)	−0.007 (3)
C3	0.036 (5)	0.029 (4)	0.038 (4)	−0.006 (4)	0.003 (4)	−0.009 (3)
C5	0.048 (6)	0.049 (5)	0.041 (5)	−0.006 (4)	0.006 (4)	−0.029 (4)
C4	0.063 (7)	0.034 (5)	0.053 (6)	−0.001 (5)	0.009 (5)	−0.021 (4)
O2	0.019 (3)	0.021 (2)	0.022 (2)	−0.001 (2)	−0.0043 (19)	0.0049 (19)
O1W	0.022 (3)	0.054 (4)	0.029 (3)	−0.010 (3)	−0.009 (2)	0.015 (3)
O2W	0.067 (5)	0.070 (5)	0.039 (4)	−0.019 (4)	−0.015 (3)	−0.004 (3)

Geometric parameters (Å, °)

Dy1—O3	2.289 (5)	C7—O5	1.244 (8)
Dy1—O1W	2.352 (5)	C7—C7^{ii}	1.549 (12)
Dy1—O2^{i}	2.357 (5)	C1—O2	1.277 (8)
Dy1—O1	2.364 (4)	C1—C2	1.488 (9)
Dy1—O5^{ii}	2.366 (4)	N1—C5	1.353 (11)
Dy1—O7^{iii}	2.391 (5)	N1—H1	0.8600
Dy1—O6	2.402 (5)	C2—C3	1.377 (11)
Dy1—O4	2.415 (4)	C3—C4	1.398 (12)
O4—C7	1.247 (8)	C3—H3	0.9300
O1—C1	1.250 (8)	C5—C4	1.336 (13)
O7—C8	1.239 (8)	C5—H5	0.9300
C6—O3	1.275 (9)	C4—H4	0.9300
C6—N1	1.361 (9)	O1W—H1W	0.8500

C6—C2	1.424 (10)	O1W—H2W	0.8490
C8—O6	1.255 (8)	O2W—H3W	0.8534
C8—C8^{iii}	1.547 (13)	O2W—H4W	0.8503
O3—Dy1—O1W	90.9 (2)	N1—C6—C2	115.8 (7)
O3—Dy1—O2^{i}	90.55 (18)	C6—O3—Dy1	136.4 (4)
O1W—Dy1—O2^{i}	148.35 (17)	O7—C8—O6	127.9 (6)
O3—Dy1—O1	73.20 (16)	O7—C8—C8^{iii}	116.5 (7)
O1W—Dy1—O1	75.35 (18)	O6—C8—C8^{iii}	115.6 (7)
O2^{i}—Dy1—O1	74.84 (16)	C8—O6—Dy1	120.2 (4)
O3—Dy1—O5^{ii}	81.96 (17)	O5—C7—O4	126.4 (6)
O1W—Dy1—O5^{ii}	67.74 (17)	O5—C7—C7^{ii}	116.8 (7)
O2^{i}—Dy1—O5^{ii}	143.60 (16)	O4—C7—C7^{ii}	116.8 (7)
O1—Dy1—O5^{ii}	134.77 (17)	C7—O5—Dy1^{ii}	120.1 (4)
O3—Dy1—O7^{iii}	148.30 (17)	O1—C1—O2	122.5 (6)
O1W—Dy1—O7^{iii}	105.1 (2)	O1—C1—C2	120.4 (6)
O2^{i}—Dy1—O7^{iii}	89.70 (19)	O2—C1—C2	117.1 (6)
O1—Dy1—O7^{iii}	136.90 (16)	C5—N1—C6	123.9 (7)
O5^{ii}—Dy1—O7^{iii}	79.21 (18)	C5—N1—H1	118.0
O3—Dy1—O6	144.68 (17)	C6—N1—H1	118.0
O1W—Dy1—O6	75.02 (19)	C3—C2—C6	119.6 (7)
O2^{i}—Dy1—O6	85.84 (17)	C3—C2—C1	119.9 (7)
O1—Dy1—O6	71.95 (16)	C6—C2—C1	120.5 (6)
O5^{ii}—Dy1—O6	119.90 (17)	C2—C3—C4	121.4 (8)
O7^{iii}—Dy1—O6	66.91 (16)	C2—C3—H3	119.3
O3—Dy1—O4	77.17 (17)	C4—C3—H3	119.3
O1W—Dy1—O4	135.23 (16)	C4—C5—N1	121.4 (8)
O2^{i}—Dy1—O4	75.68 (15)	C4—C5—H5	119.3
O1—Dy1—O4	137.50 (15)	N1—C5—H5	119.3
O5^{ii}—Dy1—O4	67.92 (15)	C5—C4—C3	117.9 (8)
O7^{iii}—Dy1—O4	72.20 (16)	C5—C4—H4	121.0
O6—Dy1—O4	134.95 (16)	C3—C4—H4	121.0
C7—O4—Dy1	118.2 (4)	C1—O2—Dy1^{i}	128.1 (4)
C1—O1—Dy1	136.4 (4)	Dy1—O1W—H1W	125.7
C8—O7—Dy1^{iii}	120.8 (4)	Dy1—O1W—H2W	124.6
O3—C6—N1	117.2 (6)	H1W—O1W—H2W	109.7
O3—C6—C2	127.0 (6)	H3W—O2W—H4W	109.6

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

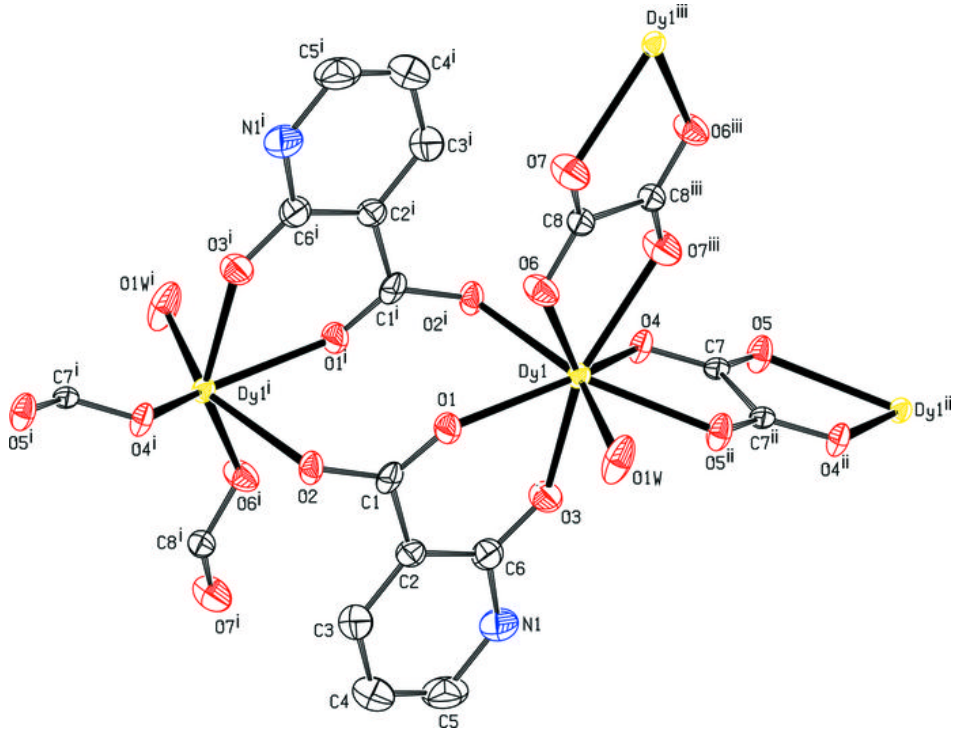
Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2W	0.86	1.99	2.785 (9)	154
O1W—H1W···O2^{iv}	0.85	1.94	2.732 (7)	155
O1W—H2W···O4^{v}	0.85	2.07	2.751 (7)	137

O2W—H4W···O1^{vi}	0.85	2.26	3.080 (8)	163
O2W—H4W···O6^{vi}	0.85	2.36	2.878 (8)	120

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

Fig. 1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C4i
N1i
C3i
O6iii
O7
C8iii
C2i
C8
O3i
C6i
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O1Wi
O6
C1i
O2i
O4
C7
O5
O1i
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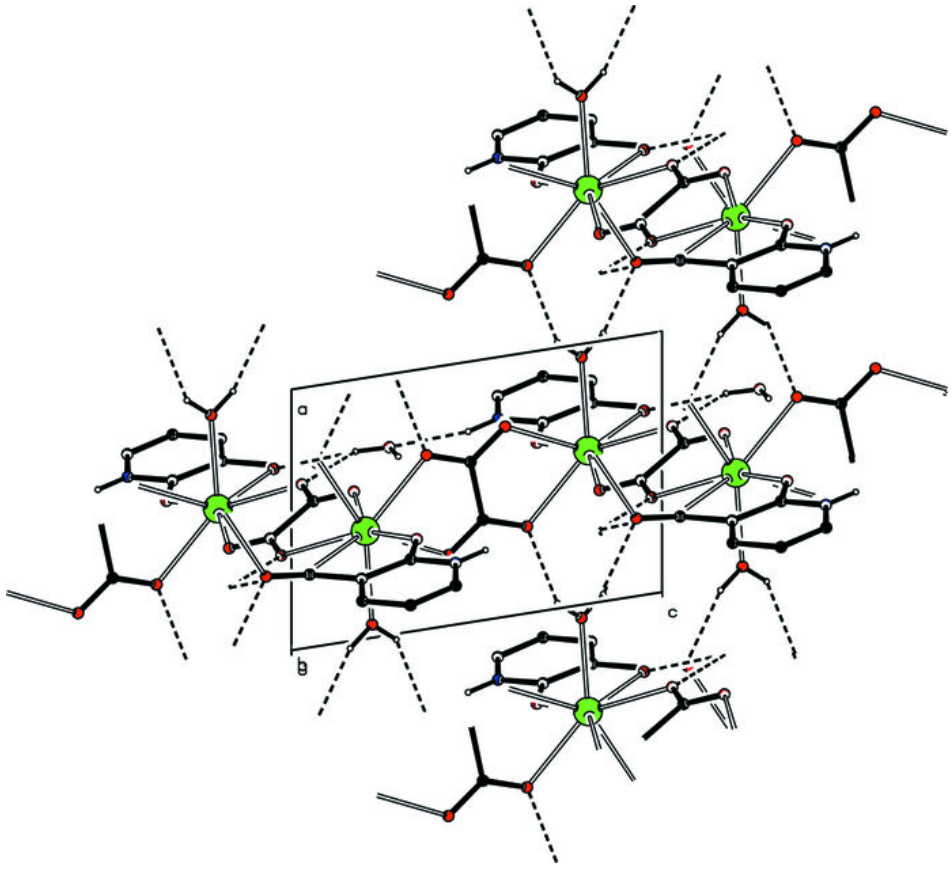
Fig. 2