

Poly[aqua(μ_2 -oxalato)(4-oxidopyridinium)erbium(II)]

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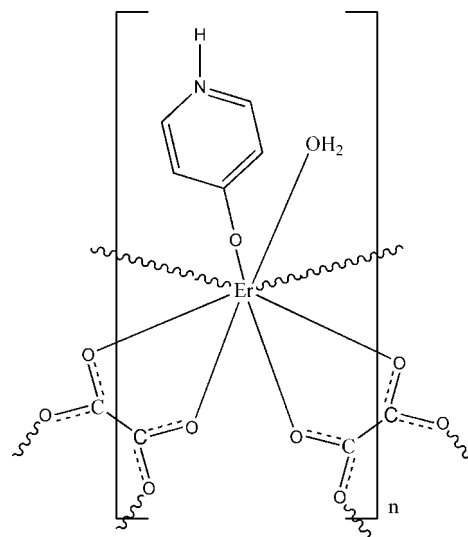
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; disorder in main residue; R factor = 0.015; wR factor = 0.037; data-to-parameter ratio = 13.0.

The title complex, $[\text{Er}(\text{C}_5\text{H}_5\text{NO})(\text{C}_2\text{O}_4)(\text{H}_2\text{O})]_n$, is a new erbium polymer based on oxalate and 4-oxidopyridinium ligands. The Er^{II} center is coordinated by six O atoms from three oxalate ligands, one O atom from a 4-oxidopyridinium ligand and one water molecule, and displays a distorted square-antiprismatic coordination geometry. The oxalate ligands are both chelating and bridging, and link Er^{II} ions, forming Er -oxalate layers in which the attached water and 4-oxidopyridinium units point alternately up and down. A mirror plane passes through the Er atom, one C, the oxide O and two oxalate O atoms. The layers are assembled into a three-dimensional supramolecular network *via* intermolecular hydrogen bonding and π - π stacking interactions [centroid-centroid distances of 3.587 (2) Å between parallel pyridinium rings]. Both the water molecule and the 4-oxidopyridinium ligand are disordered over two sites in a 1:1 ratio.

Related literature

For related literature, see: Yaghi *et al.* (1998, 2003); Serre *et al.* (2004); James (2003).



Experimental

Crystal data

$[\text{Er}(\text{C}_5\text{H}_5\text{NO})(\text{C}_2\text{O}_4)(\text{H}_2\text{O})]$
 $M_r = 412.41$
Monoclinic, $C2/m$
 $a = 16.8649$ (2) Å
 $b = 11.1863$ (2) Å
 $c = 6.5152$ (1) Å
 $\beta = 112.213$ (1)°

$V = 1137.91$ (3) Å³
 $Z = 4$
Mo $K\alpha$ radiation
 $\mu = 7.41$ mm⁻¹
 $T = 296$ (2) K
 $0.21 \times 0.19 \times 0.13$ mm

Data collection

Bruker APEXII area-detector diffractometer
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\text{min}} = 0.241$, $T_{\text{max}} = 0.392$

7274 measured reflections
1365 independent reflections
1341 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.022$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.014$
 $wR(F^2) = 0.036$
 $S = 1.16$
1365 reflections
105 parameters
39 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.53$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.88$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O1W}-\text{H2W}\cdots\text{O1}^i$	0.818 (10)	2.03 (4)	2.769 (4)	149 (8)
$\text{O1W}-\text{H1W}\cdots\text{O2}^i$	0.818 (10)	2.18 (6)	2.729 (4)	124 (6)
$\text{N1}-\text{H6}\cdots\text{O4}^{\text{ii}}$	0.86	2.41	3.041 (3)	130
$\text{N1}-\text{H6}\cdots\text{O4}^{\text{iii}}$	0.86	2.02	2.794 (3)	150

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

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: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL.

The authors thank Guang Dong Ocean University for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZL2097).

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supplementary materials

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Poly[aqua(μ_2 -oxalato)(4-oxidopyridinium)erbium(II)]

C.-S. Gu, X.-M. Hao, W.-D. Song, H.-S. Lin and D.-Y. Ma

Comment

The use of multifunctional organic linker molecules to polymerize metal centers into open-framework materials has led to the development of a rich field of chemistry (Yaghi *et al.*, 1998, 2003; Serre *et al.*, 2004; James, 2003) owing to the potential applications of these materials in catalysis, separation, gas storage and molecular recognition. Among such novel open-framework materials, lanthanide oxalates are particularly noteworthy. The wide variety of coordination modes of the oxalate anion permits the use of metal-oxalate units as excellent building blocks to construct a great diversity of frameworks ranging from discrete oligomeric entities to one-, two- and three-dimensional networks. Recently, we obtained the title erbium polymer (I), and its crystal structure is reported here.

The Er^{II} centre in the title compound exhibits a distorted square-antiprismatic coordination geometry, defined by six O atoms from three oxalate ligands, one O atom from the 4-oxidopyridinium ligand and one water molecule (Fig. 1). The oxalate ligands exhibit bidentate O atoms and link to the Er^{II} ions in a bridging mode to adjacent metal centres with Er—Er distances of 6.153 (2) Å and 6.112 (3) Å, respectively, thus forming Er-oxalate layers with the attached water and the 4-oxidopyridinium units that are alternately pointing up and down (Fig. 2). The layers are assembled into a three-dimensional supramolecular network *via* intermolecular O—H \cdots O and N—H \cdots O hydrogen bonding interactions (Table 1) involving the coordinated water molecules, N-protonated 4-hydroxypyridine, the hydroxy O atoms and the oxalate O atoms. They are also stabilized by π - π stacking interactions with centroid to centroid distances of 3.587 (2) Å between parallel pyridinium rings of neighboring complexes (at $-x, y, 1-z$). The coordinated water molecule and the 4-oxidopyridinium ligands are located close to a mirror plane perpendicular to the b-axis of the unit cell and are disordered across this plane in a one to one ratio. As stated above the water molecules are engaged in hydrogen bonding to the hydroxide O atom O1 and to oxalate atom O2 (Table 1), and the orientation of the hydrogen O—H \cdots O bond is equivalent but opposite for the two different disordered moieties, thus causing the disorder observed for the water molecule. The hydrogen bond formed by the 4-oxidopyridinium moiety is directed to either of the two symmetry equivalent oxalate oxygen atoms O4 (Table 1), and formation of either of the two H bonds is again responsible for the presence of the disorder observed.

Experimental

A mixture of Er₂O₃ (0.5 mmol), oxalic acid (1 mmol), 4-hydroxypyridine (1 mmol) and H₂O (10 ml) was placed in a 23 ml Teflon reactor, which was heated to 433 K for three days and then cooled to room temperature at a rate of 10 K h⁻¹. The crystals obtained were washed with water and dried in air.

Refinement

In the initial refinement with disorder not taken into account both the water molecule and the 4-oxidopyridinium moiety showed significantly elongated thermal ellipsoids indicating disorder, and they were thus refined as being disordered over two positions across a crystallographic mirror plane perpendicular to the b-axis. The ADPs of the disordered atoms were

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restrained to be close to isotropic and those of equivalent atoms were set to be identical. Carbon-bound H atoms were placed in calculated positions and were treated as riding on the parent C atoms with C—H = 0.93 Å, N—H = 0.86 Å and with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C}, \text{N})$; 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, each within a standard deviation of 0.01 Å, and with $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{O})$.

Figures

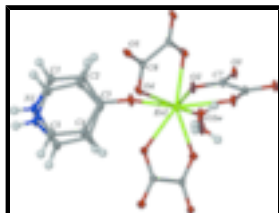


Fig. 1. The structure of the title compound, showing the atom-numbering scheme and displacement ellipsoids drawn at the 30% probability level.

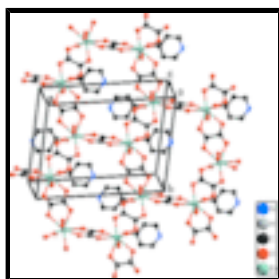


Fig. 2. A layer view of (I). Hydrogen bonds are depicted as broken lines.

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Crystal data

[Er(C₅H₅NO)(C₂O₄)(H₂O)]

$M_r = 412.41$

Monoclinic, $C2/m$

Hall symbol: $-C 2y$

$a = 16.8649 (2) \text{ \AA}$

$b = 11.1863 (2) \text{ \AA}$

$c = 6.51520 (10) \text{ \AA}$

$\beta = 112.2130 (10)^\circ$

$V = 1137.91 (3) \text{ \AA}^3$

$Z = 4$

$F_{000} = 776$

$D_x = 2.407 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 8000 reflections

$\theta = 1.7\text{--}26.0^\circ$

$\mu = 7.41 \text{ mm}^{-1}$

$T = 296 (2) \text{ K}$

Block, white

$0.21 \times 0.19 \times 0.13 \text{ mm}$

Data collection

Bruker APEXII area-detector
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 296(2) \text{ K}$

φ and ω scans

1365 independent reflections

1341 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.022$

$\theta_{\text{max}} = 27.5^\circ$

$\theta_{\text{min}} = 2.2^\circ$

Absorption correction: multi-scan
(SADABS; Sheldrick, 1996) $h = -21 \rightarrow 18$
 $T_{\min} = 0.241$, $T_{\max} = 0.392$ $k = -14 \rightarrow 14$
 7274 measured reflections $l = -8 \rightarrow 8$

Refinement

Refinement on F^2 Secondary atom site location: difference Fourier map
 Least-squares matrix: full Hydrogen site location: inferred from neighbouring sites
 $R[F^2 > 2\sigma(F^2)] = 0.014$ H atoms treated by a mixture of independent and constrained refinement
 $wR(F^2) = 0.036$ $w = 1/[\sigma^2(F_o^2) + (0.019P)^2 + 1.5595P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $S = 1.16$ $(\Delta/\sigma)_{\max} = 0.001$
 1365 reflections $\Delta\rho_{\max} = 0.53 \text{ e } \text{\AA}^{-3}$
 105 parameters $\Delta\rho_{\min} = -0.87 \text{ e } \text{\AA}^{-3}$
 39 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
N1	-0.02721 (14)	0.4755 (4)	0.2392 (5)	0.0325 (18)	0.50
H6	-0.0815	0.4701	0.2056	0.039*	0.50
C1	0.0218 (3)	0.3724 (4)	0.2667 (9)	0.0358 (13)	0.50
H1	-0.0041	0.2977	0.2487	0.043*	0.50
C2	0.1096 (3)	0.3811 (8)	0.3210 (9)	0.0381 (10)	0.50
H2	0.1425	0.3121	0.3394	0.046*	0.50
C3	0.14844 (16)	0.4927 (10)	0.3478 (5)	0.0326 (13)	0.50
C4	0.0994 (4)	0.5958 (8)	0.3203 (9)	0.0381 (10)	0.50
H4	0.1254	0.6705	0.3383	0.046*	0.50
C5	0.0116 (4)	0.5871 (4)	0.2660 (9)	0.0358 (13)	0.50
H5	-0.0212	0.6561	0.2476	0.043*	0.50
O1W	0.3241 (2)	0.4753 (8)	-0.1619 (6)	0.040 (3)	0.50

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H1W	0.358 (4)	0.438 (6)	-0.200 (10)	0.060*	0.50
H2W	0.291 (4)	0.506 (8)	-0.276 (7)	0.060*	0.50
C6	0.20541 (15)	0.2749 (2)	-0.0746 (4)	0.0252 (5)	
C7	0.4856 (2)	0.5000	0.5987 (5)	0.0212 (6)	
Er1	0.311393 (8)	0.5000	0.17883 (2)	0.01752 (6)	
O1	0.23010 (16)	0.5000	0.3883 (4)	0.0347 (6)	
O2	0.40635 (16)	0.5000	0.5514 (4)	0.0313 (6)	
O3	0.54305 (16)	0.5000	0.7871 (4)	0.0324 (6)	
O4	0.19303 (11)	0.38280 (16)	-0.0507 (3)	0.0313 (4)	
O5	0.15399 (12)	0.20337 (17)	-0.2053 (3)	0.0338 (4)	

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0174 (18)	0.056 (6)	0.0245 (17)	-0.0017 (19)	0.0082 (14)	-0.0025 (18)
C1	0.0264 (18)	0.052 (4)	0.0305 (15)	-0.0077 (17)	0.0121 (13)	-0.0001 (17)
C2	0.0235 (17)	0.059 (3)	0.0322 (15)	-0.011 (2)	0.0107 (13)	0.005 (2)
C3	0.0204 (19)	0.062 (4)	0.0147 (15)	0.008 (5)	0.0055 (14)	-0.008 (5)
C4	0.0235 (17)	0.059 (3)	0.0322 (15)	-0.011 (2)	0.0107 (13)	0.005 (2)
C5	0.0264 (18)	0.052 (4)	0.0305 (15)	-0.0077 (17)	0.0121 (13)	-0.0001 (17)
O1W	0.0155 (15)	0.089 (9)	0.0155 (13)	0.001 (2)	0.0049 (11)	0.002 (2)
C6	0.0150 (12)	0.0301 (11)	0.0257 (12)	0.0024 (9)	0.0021 (10)	-0.0033 (9)
C7	0.0120 (15)	0.0322 (16)	0.0177 (14)	0.000	0.0036 (12)	0.000
Er1	0.00977 (9)	0.02597 (9)	0.01457 (8)	0.000	0.00206 (6)	0.000
O1	0.0134 (12)	0.0683 (19)	0.0206 (12)	0.000	0.0044 (10)	0.000
O2	0.0111 (11)	0.0639 (18)	0.0177 (11)	0.000	0.0041 (9)	0.000
O3	0.0122 (12)	0.0645 (18)	0.0186 (12)	0.000	0.0038 (10)	0.000
O4	0.0161 (9)	0.0284 (8)	0.0392 (10)	0.0039 (7)	-0.0011 (8)	-0.0073 (7)
O5	0.0187 (9)	0.0307 (9)	0.0374 (10)	0.0035 (8)	-0.0058 (7)	-0.0064 (8)

Geometric parameters (\AA , $^\circ$)

N1—C1	1.3900	C6—O5	1.249 (3)
N1—C5	1.3900	C6—C6 ⁱ	1.553 (5)
N1—H6	0.8600	C7—O3	1.243 (4)
C1—C2	1.3900	C7—O2	1.253 (4)
C1—H1	0.9300	C7—C7 ⁱⁱ	1.537 (6)
C2—C3	1.3900	Er1—O1	2.271 (3)
C2—H2	0.9300	Er1—O1W ⁱⁱⁱ	2.326 (3)
C3—O1	1.303 (3)	Er1—O5 ⁱ	2.3388 (19)
C3—C4	1.3900	Er1—O5 ^{iv}	2.3388 (19)
C4—C5	1.3900	Er1—O2	2.349 (2)
C4—H4	0.9300	Er1—O3 ⁱⁱ	2.380 (2)
C5—H5	0.9300	Er1—O4	2.3839 (17)
O1W—Er1	2.326 (3)	Er1—O4 ⁱⁱⁱ	2.3839 (17)
O1W—H1W	0.818 (10)	O1—C3 ⁱⁱⁱ	1.303 (4)
O1W—H2W	0.818 (10)	O3—Er1 ⁱⁱ	2.380 (2)

C6—O4	1.245 (3)	O5—Er1 ⁱ	2.3388 (19)
C1—N1—C5	120.0	O1W—Er1—O5 ^{iv}	94.3 (2)
C1—N1—H6	120.0	O5 ⁱ —Er1—O5 ^{iv}	153.17 (9)
C5—N1—H6	120.0	O1—Er1—O2	73.12 (9)
C2—C1—N1	120.0	O1W ⁱⁱⁱ —Er1—O2	135.51 (11)
C2—C1—H1	120.0	O1W—Er1—O2	135.51 (11)
N1—C1—H1	120.0	O5 ⁱ —Er1—O2	82.54 (5)
C1—C2—C3	120.0	O5 ^{iv} —Er1—O2	82.54 (5)
C1—C2—H2	120.0	O1—Er1—O3 ⁱⁱ	141.24 (9)
C3—C2—H2	120.0	O1W ⁱⁱⁱ —Er1—O3 ⁱⁱ	67.96 (11)
O1—C3—C4	120.4 (7)	O1W—Er1—O3 ⁱⁱ	67.96 (11)
O1—C3—C2	119.5 (7)	O5 ⁱ —Er1—O3 ⁱⁱ	76.89 (5)
C4—C3—C2	120.0	O5 ^{iv} —Er1—O3 ⁱⁱ	76.89 (5)
C5—C4—C3	120.0	O2—Er1—O3 ⁱⁱ	68.12 (8)
C5—C4—H4	120.0	O1—Er1—O4	79.89 (7)
C3—C4—H4	120.0	O1W ⁱⁱⁱ —Er1—O4	79.87 (16)
C4—C5—N1	120.0	O1W—Er1—O4	72.16 (15)
C4—C5—H5	120.0	O5 ⁱ —Er1—O4	68.77 (6)
N1—C5—H5	120.0	O5 ^{iv} —Er1—O4	135.04 (6)
Er1—O1W—H1W	133 (5)	O2—Er1—O4	136.92 (6)
Er1—O1W—H2W	123 (4)	O3 ⁱⁱ —Er1—O4	130.40 (6)
H1W—O1W—H2W	104.2 (17)	O1—Er1—O4 ⁱⁱⁱ	79.89 (7)
O4—C6—O5	126.9 (2)	O1W ⁱⁱⁱ —Er1—O4 ⁱⁱⁱ	72.16 (15)
O4—C6—C6 ⁱ	116.0 (3)	O1W—Er1—O4 ⁱⁱⁱ	79.87 (16)
O5—C6—C6 ⁱ	117.1 (3)	O5 ⁱ —Er1—O4 ⁱⁱⁱ	135.04 (6)
O3—C7—O2	127.1 (3)	O5 ^{iv} —Er1—O4 ⁱⁱⁱ	68.77 (6)
O3—C7—C7 ⁱⁱ	116.8 (4)	O2—Er1—O4 ⁱⁱⁱ	136.92 (6)
O2—C7—C7 ⁱⁱ	116.0 (4)	O3 ⁱⁱ —Er1—O4 ⁱⁱⁱ	130.40 (6)
O1—Er1—O1W ⁱⁱⁱ	150.24 (12)	O4—Er1—O4 ⁱⁱⁱ	66.72 (8)
O1—Er1—O1W	150.24 (12)	C3—O1—Er1	135.2 (2)
O1W ⁱⁱⁱ —Er1—O1W	13.6 (5)	C3 ⁱⁱⁱ —O1—Er1	135.2 (2)
O1—Er1—O5 ⁱ	98.41 (5)	C7—O2—Er1	120.1 (2)
O1W ⁱⁱⁱ —Er1—O5 ⁱ	94.3 (2)	C7—O3—Er1 ⁱⁱ	118.9 (2)
O1W—Er1—O5 ⁱ	81.0 (2)	C6—O4—Er1	118.12 (15)
O1—Er1—O5 ^{iv}	98.41 (5)	C6—O5—Er1 ⁱ	118.91 (16)
O1W ⁱⁱⁱ —Er1—O5 ^{iv}	81.0 (2)		

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

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

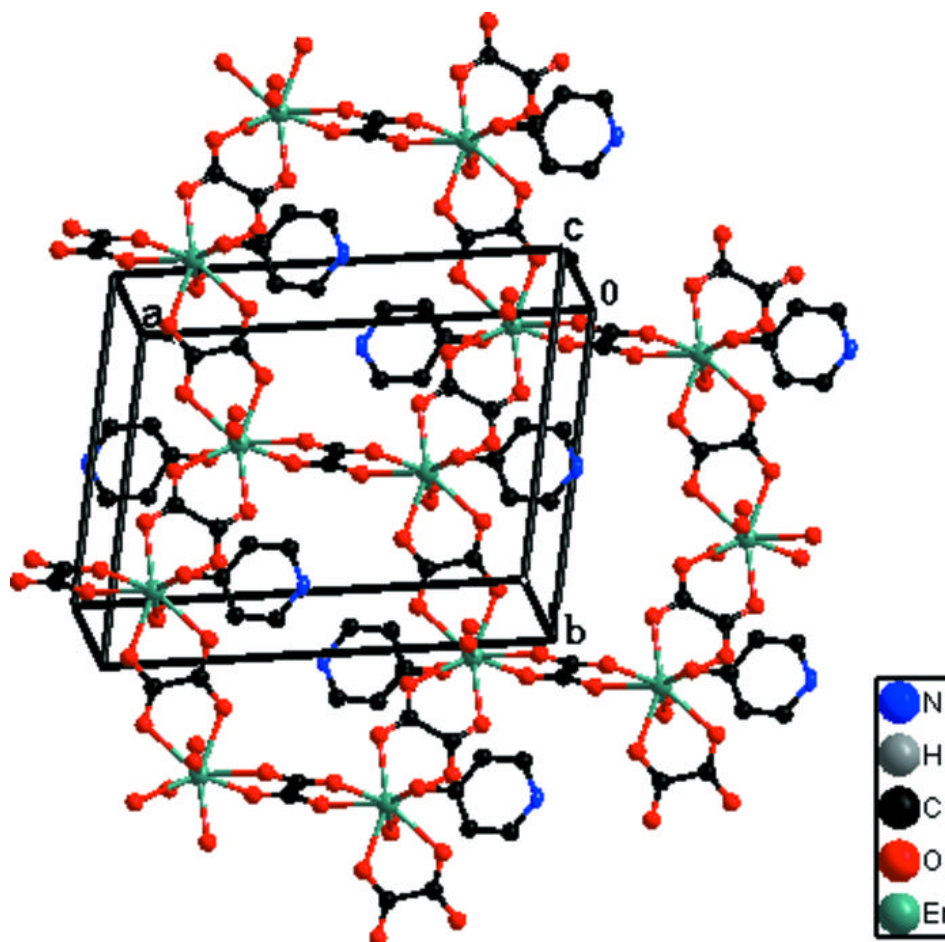
<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O1W—H2W \cdots O1 ^v	0.818 (10)	2.03 (4)	2.769 (4)	149 (8)

supplementary materials

O1W—H1W...O2 ^v	0.818 (10)	2.18 (6)	2.729 (4)	124 (6)
N1—H6...O4 ^{vi}	0.86	2.41	3.041 (3)	130
N1—H6...O4 ^{vii}	0.86	2.02	2.794 (3)	150

Symmetry codes: (v) $x, y, z-1$; (vi) $-x, -y+1, -z$; (vii) $-x, y, -z$.

Fig. 2



Poly[aqua(μ_2 -oxalato)(4-oxidopyridinium)erbium(II)]. Corrigendum

Chang-Sheng Gu,^a Xiao-Min Hao,^a Wen-Dong Song,^{b*}
Hai-Sheng Lin^a and De-Yun Ma^c

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The chemical name in the title, the formula and the scheme of the paper by Gu, Hao, Song, Lin & Ma [*Acta Cryst.* (2008), E64, m649–m650] are corrected.

In the paper by Gu, Hao, Song, Lin & Ma [*Acta Cryst.* (2008), E64, m649–m650], the chemical name in the title, the formula and the scheme are incorrect. The correct title should be 'Poly-[aquaesqui(μ_2 -oxalato)(4-oxidopyridinium)erbium(III)]', $[\text{Er}(\text{C}_2\text{O}_4)_{1.5}(\text{C}_5\text{H}_5\text{NO})(\text{H}_2\text{O})]_n$, and the correct scheme is shown below. Note the revised oxidation state for erbium(III) which is given as erbium(II) twice in the original *Abstract*.

