

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

Poly[[triqua(μ_3 -4-oxidopyridine-2,6-dicarboxylato)thulium(III)] monohydrate]

 Zhu-Qing Gao,^{a*} Dong-Yu Lv,^b Jin-Zhong Gu^b and Hong-Jin Li^a

^aSchool of Chemistry and Biology Engineering, Taiyuan University of Science and Technology, Taiyuan 030021, People's Republic of China, and ^bKey Laboratory of Nonferrous Metal Chemistry and Resources Utilization of Gansu Province, College of Chemistry and Chemical Engineering, Lanzhou University, Lanzhou 730000, People's Republic of China

Correspondence e-mail: zqgao2008@163.com

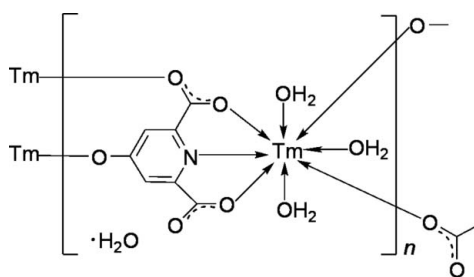
Received 21 February 2011; accepted 28 February 2011

Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.007$ Å; R factor = 0.031; wR factor = 0.082; data-to-parameter ratio = 11.5.

In the title coordination polymer, $[\{\text{Tm}(\text{C}_7\text{H}_2\text{NO}_5)(\text{H}_2\text{O})_3\} \cdot \text{H}_2\text{O}]_n$, the Tm^{III} atom is eight-coordinated by a tridentate 4-oxidopyridine-2,6-dicarboxylate trianion, two monodentate anions and three water molecules, forming a distorted bicapped trigonal-prismatic TmNO_7 coordination geometry. The anions bridge adjacent Tm^{III} ions into double chains. Adjacent chains are further connected into sheets. $\text{O}-\text{H} \cdots \text{O}$ hydrogen bonds involving both coordinated and uncoordinated water molecules generate a three-dimensional supramolecular framework.

Related literature

For the structures and properties of lanthanide coordination compounds including the isotopic Dy and Eu analogues, see: Qin *et al.* (2011); Lv *et al.* (2010); Gao *et al.* (2006). For structures of complexes containing eight-coordinate Tm^{III} , see: Qin *et al.* (2011); Tian *et al.* (2009).



Experimental

Crystal data

 $[\text{Tm}(\text{C}_7\text{H}_2\text{NO}_5)(\text{H}_2\text{O})_3] \cdot \text{H}_2\text{O}$
 $M_r = 421.09$

 Monoclinic, $P2_1/n$
 $a = 9.829$ (3) Å

 $b = 7.559$ (2) Å

 $c = 15.350$ (5) Å

 $\beta = 105.589$ (3)°

 $V = 1098.6$ (6) Å³
 $Z = 4$

 Mo $K\alpha$ radiation

 $\mu = 8.12$ mm⁻¹
 $T = 296$ K

 $0.28 \times 0.26 \times 0.22$ mm

Data collection

Bruker APEXII CCD

diffractometer

Absorption correction: multi-scan

(SADABS; Bruker, 2004)

 $T_{\text{min}} = 0.118$, $T_{\text{max}} = 0.168$

5810 measured reflections

2028 independent reflections

 1721 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.038$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.031$
 $wR(F^2) = 0.082$
 $S = 1.08$

2028 reflections

176 parameters

12 restraints

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\text{max}} = 1.45$ e Å⁻³
 $\Delta\rho_{\text{min}} = -1.83$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H} \cdots A$	$D-\text{H}$	$\text{H} \cdots A$	$D \cdots A$	$D-\text{H} \cdots A$
$\text{O9}-\text{H8W} \cdots \text{O2}^{\text{i}}$	0.88	2.54	3.111 (6)	123
$\text{O9}-\text{H7W} \cdots \text{O5}^{\text{ii}}$	0.83	2.03	2.694 (6)	137
$\text{O8}-\text{H6W} \cdots \text{O9}^{\text{iii}}$	0.95	1.73	2.679 (5)	179
$\text{O8}-\text{H5W} \cdots \text{O9}^{\text{iv}}$	0.90	2.27	3.068 (6)	148
$\text{O7}-\text{H4W} \cdots \text{O5}^{\text{v}}$	0.95	1.79	2.697 (5)	158
$\text{O7}-\text{H3W} \cdots \text{O1}^{\text{i}}$	0.90 (3)	1.85 (3)	2.672 (5)	151 (5)
$\text{O6}-\text{H2W} \cdots \text{O4}^{\text{vi}}$	0.83 (4)	2.11 (5)	2.791 (5)	139 (5)
$\text{O6}-\text{H1W} \cdots \text{O3}^{\text{vii}}$	0.91 (4)	1.84 (4)	2.706 (5)	156 (4)

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINTE* (Bruker, 2004); data reduction: *SAINTE*; 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*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2392).

References

- Bruker (2004). *APEX2*, *SAINTE* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Gao, H. L., Yi, L., Zhao, B., Zhao, X. Q., Cheng, P., Liao, D. Z. & Yan, S. P. (2006). *Inorg. Chem.* **45**, 5980–5988.
- Lv, D.-Y., Gao, Z.-Q. & Gu, J.-Z. (2010). *Acta Cryst.* **E66**, m1694–m1695.
- Qin, J. S., Du, D. Y., Chen, L., Sun, X. Y., Lan, Y. Q. & Su, Z. M. (2011). *J. Solid State Chem.* **184**, 373–378.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Tian, Y. P., Li, L., Zhou, Y. H., Wang, P., Zhou, H. P., Wu, J. Y., Hu, Z. J., Yang, Y. X., Kong, L., Xu, G. B., Tao, X. T. & Jiang, M. H. (2009). *Cryst. Growth Des.* **9**, 1499–1504.

supporting information

Acta Cryst. (2011). E67, m409 [doi:10.1107/S1600536811007628]

Poly[[triaqua(μ_3 -4-oxidopyridine-2,6-dicarboxylato)thulium(III)] monohydrate]**Zhu-Qing Gao, Dong-Yu Lv, Jin-Zhong Gu and Hong-Jin Li****S1. Comment**

The lanthanide coordination polymers have shown not only versatile architectures but also desirable properties, *e.g.*, luminescent, magnetic, catalytic, and gas absorption and separation properties (Qin *et al.*, 2011; Lv *et al.*, 2010). In order to extend our investigations in this field, we have designed and synthesized a novel lanthanide coordination polymer, $\{[\text{Tm}(\text{C}_7\text{H}_2\text{NO}_5)(\text{H}_2\text{O})_3]\cdot\text{H}_2\text{O}\}_n$, by choosing 4-oxidopyridine-2,6-dicarboxylic acid as a functional ligand, and report its crystal structure in this paper.

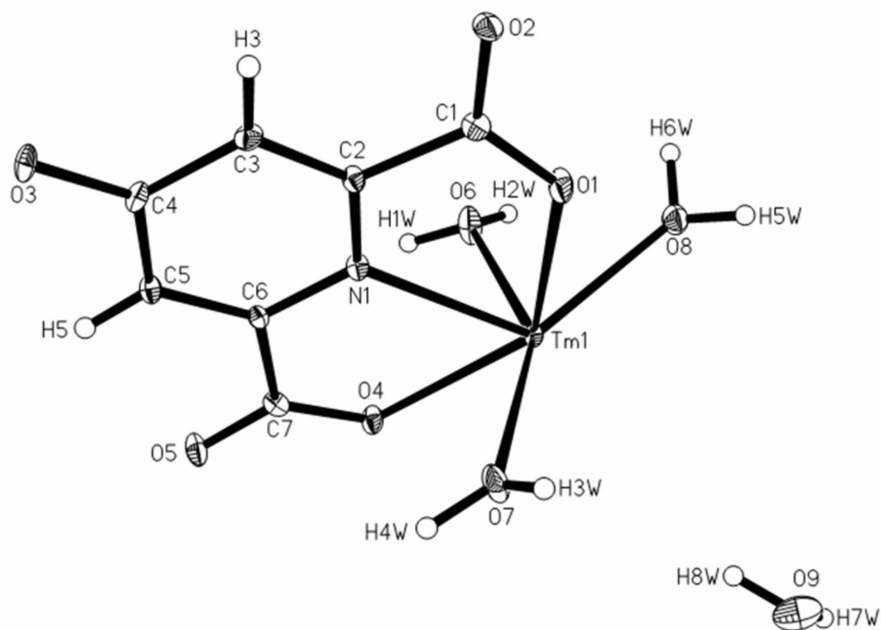
The title compound is isotopic with its Dy (Gao *et al.*, 2006) and Eu (Lv *et al.*, 2010) analogues. The asymmetrical unit of the title complex contains a Tm(III) ion, a 4-oxidopyridine-2,6-dicarboxylate anion, three coordinated water molecules, and a molecule of water of crystallization (Fig. 1). The Tm atom is eight-coordinated by seven oxygen atoms from three anions and three coordinated water molecules and by a nitrogen atom from a tridentate anion (the other two anions are monodentate), forming a distorted bicapped trigonal-prismatic coordination environment. The Tm–O bond lengths [2.263 (3) - 2.385 (3) Å] are shorter than the Tm–N bond length [2.430 (4) Å], which is in agreement with the bond lengths observed in other Tm(III) complexes (Qin *et al.*, 2011; Tian *et al.*, 2009). The anion adopts a μ_3 -pentadentate coordination mode. The anions bridge the adjacent Tm^{III} ions to form infinite double chains (Fig. 2). Adjacent chains are further connected by the coordination of the anions and Tm(III) ions into two-dimensional sheets (Fig. 3), which are further extended into a three-dimensional supramolecular framework through O–H \cdots O hydrogen-bonding interactions including both coordinated and uncoordinated water molecules (Table 1).

S2. Experimental

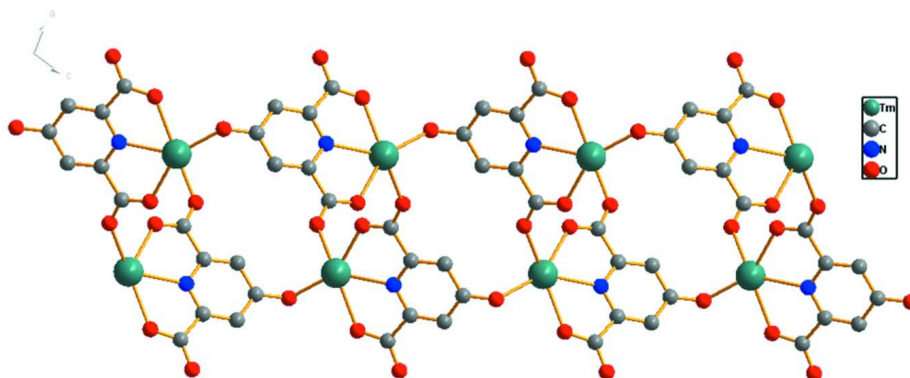
To a solution of thulium(III)nitrate hexahydrate (0.139 g, 0.3 mmol) in water (5 ml) was added an aqueous solution (5 ml) of the ligand (0.060 g, 0.3 mmol) and a drop of triethylamine. The reactants were sealed in a 25-ml Teflon-lined, stainless-steel Parr bomb. The bomb was heated at 433 K for 3 days. On cooling the solution, single crystals (*ca* 70% yield) were obtained which were suitable for single crystal X-ray diffraction studies.

S3. Refinement

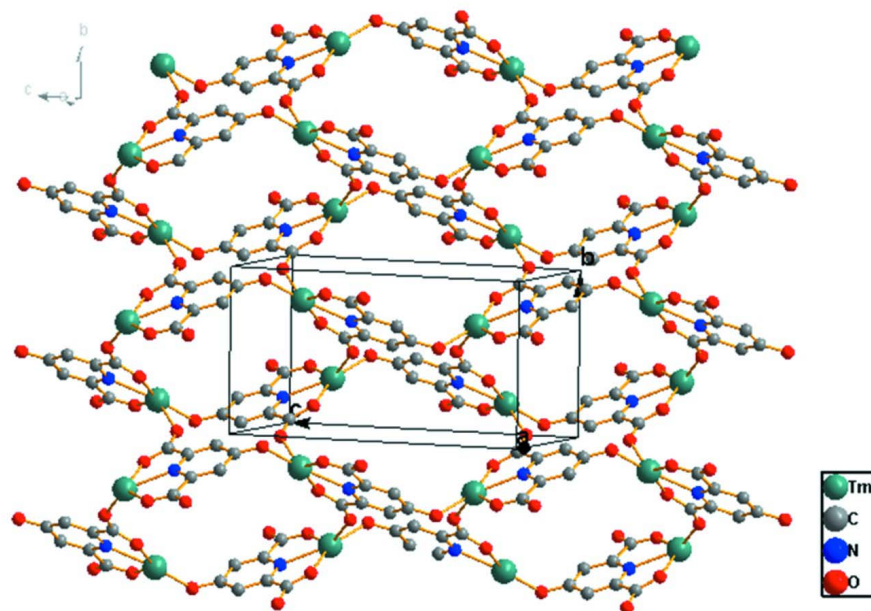
The coordinated water H atoms were located in a different Fourier map and were refined with distance constraints of O–H = 0.83 (3) Å. The free water H atoms were placed at calculated positions and refined with a riding model, considering the position of oxygen atoms and the quantity of H atoms. The carbon-bound H atoms were placed in geometrically idealized positions, with C–H = 0.93 Å and constrained to ride on their respective parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$. The final difference map was essentially featureless with residual electron density within 1.0 Å of the Th atom.

**Figure 1**

Drawing of the asymmetric unit of the title complex, showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level.

**Figure 2**

A view of the title structure along the *b* axis, showing the double chain.

**Figure 3**

A view of the unit cell along the a axis, showing the sheet structure of the title complex; H-atoms have been excluded for clarity.

Poly[[triaqua(μ_3 -4-oxidopyridine-2,6-dicarboxylato)thulium(III)] monohydrate]

Crystal data

[Tm(C₇H₂NO₃)(H₂O)₃]·H₂O

$M_r = 421.09$

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 9.829$ (3) Å

$b = 7.559$ (2) Å

$c = 15.350$ (5) Å

$\beta = 105.589$ (3)°

$V = 1098.6$ (6) Å³

$Z = 4$

$F(000) = 800$

$D_x = 2.546$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 3206 reflections

$\theta = 2.2$ – 28.1 °

$\mu = 8.12$ mm⁻¹

$T = 296$ K

Block, colorless

$0.28 \times 0.26 \times 0.22$ mm

Data collection

Bruker APEXII CCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2004)

$T_{\min} = 0.118$, $T_{\max} = 0.168$

5810 measured reflections

2028 independent reflections

1721 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.038$

$\theta_{\max} = 25.5$ °, $\theta_{\min} = 2.2$ °

$h = -11 \rightarrow 11$

$k = -9 \rightarrow 9$

$l = -18 \rightarrow 11$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.031$
 $wR(F^2) = 0.082$
 $S = 1.08$
 2028 reflections
 176 parameters
 12 restraints
 Primary atom site location: structure-invariant
 direct methods
 Secondary atom site location: difference Fourier
 map

Hydrogen site location: inferred from
 neighbouring sites
 H atoms treated by a mixture of independent
 and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.039P)^2 + 1.1506P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.002$
 $\Delta\rho_{\max} = 1.45 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -1.83 \text{ e } \text{\AA}^{-3}$
 Extinction correction: *SHELXL97* (Sheldrick,
 2008), $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.0158 (6)

Special details

Experimental. Anal. Calcd for $C_7H_{10}TbNO_9$: C, 19.97; H, 2.39; N, 3.33. Found: C, 20.31; H, 2.47; N, 3.12.

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	$U_{\text{iso}}^*/U_{\text{eq}}$
Tm1	0.498130 (19)	0.67912 (3)	0.252028 (12)	0.00947 (16)
C1	0.2175 (5)	0.9137 (7)	0.1631 (3)	0.0156 (11)
C2	0.2755 (5)	0.8881 (6)	0.0829 (3)	0.0127 (10)
C3	0.2046 (5)	0.9447 (6)	-0.0015 (3)	0.0155 (11)
H3	0.1204	1.0072	-0.0105	0.019*
C4	0.2614 (5)	0.9069 (6)	-0.0753 (3)	0.0121 (10)
C5	0.3907 (5)	0.8160 (6)	-0.0544 (3)	0.0132 (11)
H5	0.4336	0.7888	-0.0999	0.016*
C6	0.4537 (5)	0.7675 (6)	0.0335 (3)	0.0123 (10)
C7	0.5906 (5)	0.6682 (6)	0.0630 (4)	0.0132 (11)
N1	0.3961 (5)	0.8002 (5)	0.1020 (3)	0.0125 (9)
O1	0.2806 (4)	0.8294 (5)	0.2333 (3)	0.0242 (10)
O2	0.1121 (4)	1.0091 (5)	0.1562 (2)	0.0190 (8)
O3	0.1933 (4)	0.9514 (5)	-0.1587 (2)	0.0173 (8)
O4	0.6311 (4)	0.6267 (5)	0.1454 (2)	0.0168 (8)
O5	0.6587 (4)	0.6359 (5)	0.0068 (2)	0.0212 (9)
O6	0.6355 (4)	0.9322 (5)	0.2582 (3)	0.0251 (9)
O7	0.3975 (5)	0.4312 (5)	0.1720 (3)	0.0269 (10)
H4W	0.3923	0.3823	0.1140	0.032*
O8	0.5064 (5)	0.8348 (4)	0.3875 (3)	0.0232 (9)
H5W	0.4593	0.8051	0.4279	0.028*
H6W	0.5247	0.9565	0.4025	0.028*

O9	0.5599 (5)	0.1768 (5)	0.4284 (3)	0.0386 (12)
H7W	0.6306	0.1698	0.4728	0.046*
H8W	0.5610	0.2542	0.3853	0.046*
H1W	0.689 (5)	0.940 (7)	0.218 (3)	0.018 (14)*
H2W	0.689 (5)	0.964 (7)	0.307 (3)	0.017 (16)*
H3W	0.318 (5)	0.393 (6)	0.184 (3)	0.012 (13)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Tm1	0.0065 (2)	0.0139 (2)	0.0060 (2)	0.00015 (7)	−0.00175 (13)	0.00069 (7)
C1	0.010 (3)	0.020 (3)	0.014 (3)	0.002 (2)	−0.002 (2)	−0.001 (2)
C2	0.010 (3)	0.018 (2)	0.010 (3)	0.001 (2)	0.001 (2)	−0.001 (2)
C3	0.011 (3)	0.018 (3)	0.016 (3)	0.004 (2)	0.001 (2)	0.003 (2)
C4	0.008 (3)	0.016 (2)	0.011 (3)	−0.0047 (19)	−0.001 (2)	0.003 (2)
C5	0.010 (3)	0.019 (3)	0.010 (3)	−0.0025 (19)	0.001 (2)	−0.0009 (19)
C6	0.014 (3)	0.013 (2)	0.010 (3)	0.000 (2)	0.003 (2)	−0.001 (2)
C7	0.007 (3)	0.015 (2)	0.016 (3)	−0.0042 (19)	0.001 (2)	−0.004 (2)
N1	0.007 (2)	0.019 (2)	0.011 (2)	0.0006 (17)	0.0003 (19)	0.0006 (16)
O1	0.019 (2)	0.041 (2)	0.012 (2)	0.0165 (16)	0.0038 (18)	0.0091 (16)
O2	0.017 (2)	0.0239 (19)	0.015 (2)	0.0084 (16)	0.0027 (16)	0.0000 (15)
O3	0.015 (2)	0.025 (2)	0.008 (2)	−0.0046 (15)	−0.0036 (16)	0.0028 (14)
O4	0.013 (2)	0.0250 (19)	0.011 (2)	0.0046 (16)	0.0015 (15)	0.0032 (15)
O5	0.017 (2)	0.035 (2)	0.011 (2)	0.0074 (17)	0.0034 (16)	0.0002 (16)
O6	0.028 (3)	0.032 (2)	0.015 (2)	−0.0193 (18)	0.006 (2)	−0.0063 (18)
O7	0.037 (3)	0.030 (2)	0.018 (2)	−0.0158 (19)	0.013 (2)	−0.0105 (17)
O8	0.031 (3)	0.022 (2)	0.018 (2)	−0.0030 (16)	0.008 (2)	−0.0017 (15)
O9	0.027 (3)	0.027 (2)	0.055 (3)	−0.0055 (17)	−0.001 (2)	−0.0030 (19)

Geometric parameters (Å, °)

Tm1—O3 ⁱ	2.263 (3)	C5—C6	1.374 (7)
Tm1—O7	2.312 (4)	C5—H5	0.9300
Tm1—O6	2.329 (4)	C6—N1	1.345 (6)
Tm1—O1	2.369 (4)	C6—C7	1.501 (7)
Tm1—O8	2.372 (4)	C7—O5	1.250 (6)
Tm1—O2 ⁱⁱ	2.372 (3)	C7—O4	1.260 (6)
Tm1—O4	2.385 (3)	O2—Tm1 ⁱⁱⁱ	2.372 (3)
Tm1—N1	2.430 (4)	O3—Tm1 ^{iv}	2.263 (3)
C1—O2	1.243 (6)	O6—H1W	0.91 (4)
C1—O1	1.262 (6)	O6—H2W	0.83 (4)
C1—C2	1.502 (7)	O7—H4W	0.9517
C2—N1	1.321 (6)	O7—H3W	0.90 (3)
C2—C3	1.364 (7)	O8—H5W	0.8963
C3—C4	1.421 (7)	O8—H6W	0.9533
C3—H3	0.9300	O9—H7W	0.8345
C4—O3	1.318 (6)	O9—H8W	0.8850
C4—C5	1.404 (7)		

O3 ⁱ —Tm1—O7	98.07 (13)	C3—C2—C1	121.9 (5)
O3 ⁱ —Tm1—O6	86.88 (14)	C2—C3—C4	119.0 (5)
O7—Tm1—O6	147.98 (14)	C2—C3—H3	120.5
O3 ⁱ —Tm1—O1	150.82 (12)	C4—C3—H3	120.5
O7—Tm1—O1	94.66 (15)	O3—C4—C5	122.6 (4)
O6—Tm1—O1	96.05 (14)	O3—C4—C3	121.2 (5)
O3 ⁱ —Tm1—O8	82.05 (13)	C5—C4—C3	116.2 (4)
O7—Tm1—O8	141.00 (13)	C6—C5—C4	119.8 (5)
O6—Tm1—O8	70.96 (13)	C6—C5—H5	120.1
O1—Tm1—O8	71.66 (14)	C4—C5—H5	120.1
O3 ⁱ —Tm1—O2 ⁱⁱ	81.52 (13)	N1—C6—C5	123.1 (5)
O7—Tm1—O2 ⁱⁱ	71.23 (12)	N1—C6—C7	112.8 (4)
O6—Tm1—O2 ⁱⁱ	140.63 (13)	C5—C6—C7	124.1 (4)
O1—Tm1—O2 ⁱⁱ	77.90 (13)	O5—C7—O4	124.2 (5)
O8—Tm1—O2 ⁱⁱ	70.24 (12)	O5—C7—C6	119.5 (5)
O3 ⁱ —Tm1—O4	79.12 (12)	O4—C7—C6	116.3 (4)
O7—Tm1—O4	74.67 (13)	C2—N1—C6	117.4 (4)
O6—Tm1—O4	75.29 (13)	C2—N1—Tm1	121.1 (3)
O1—Tm1—O4	129.75 (12)	C6—N1—Tm1	121.3 (3)
O8—Tm1—O4	141.98 (13)	C1—O1—Tm1	124.5 (3)
O2 ⁱⁱ —Tm1—O4	137.63 (11)	C1—O2—Tm1 ⁱⁱⁱ	140.1 (3)
O3 ⁱ —Tm1—N1	143.61 (13)	C4—O3—Tm1 ^{iv}	127.1 (3)
O7—Tm1—N1	78.04 (14)	C7—O4—Tm1	124.4 (3)
O6—Tm1—N1	79.41 (14)	Tm1—O6—H1W	117 (3)
O1—Tm1—N1	64.89 (13)	Tm1—O6—H2W	120 (4)
O8—Tm1—N1	123.52 (13)	H1W—O6—H2W	104 (4)
O2 ⁱⁱ —Tm1—N1	128.94 (14)	Tm1—O7—H4W	136.0
O4—Tm1—N1	64.86 (13)	Tm1—O7—H3W	115 (3)
O2—C1—O1	124.9 (5)	H4W—O7—H3W	103.9
O2—C1—C2	119.8 (4)	Tm1—O8—H5W	125.4
O1—C1—C2	115.3 (4)	Tm1—O8—H6W	130.5
N1—C2—C3	124.6 (5)	H5W—O8—H6W	100.0
N1—C2—C1	113.5 (4)	H7W—O9—H8W	118.5
O2—C1—C2—N1	172.9 (5)	O4—Tm1—N1—C2	180.0 (4)
O1—C1—C2—N1	-8.8 (6)	O3 ⁱ —Tm1—N1—C6	14.6 (5)
O2—C1—C2—C3	-9.9 (7)	O7—Tm1—N1—C6	-72.8 (4)
O1—C1—C2—C3	168.4 (5)	O6—Tm1—N1—C6	84.3 (4)
N1—C2—C3—C4	0.6 (7)	O1—Tm1—N1—C6	-173.8 (4)
C1—C2—C3—C4	-176.2 (4)	O8—Tm1—N1—C6	142.8 (3)
C2—C3—C4—O3	176.7 (4)	O2 ⁱⁱ —Tm1—N1—C6	-126.4 (4)
C2—C3—C4—C5	-1.6 (7)	O4—Tm1—N1—C6	5.7 (3)
O3—C4—C5—C6	-177.6 (4)	O2—C1—O1—Tm1	-171.5 (4)
C3—C4—C5—C6	0.7 (7)	C2—C1—O1—Tm1	10.3 (6)
C4—C5—C6—N1	1.2 (7)	O3 ⁱ —Tm1—O1—C1	163.5 (4)
C4—C5—C6—C7	179.2 (4)	O7—Tm1—O1—C1	-80.7 (4)
N1—C6—C7—O5	-176.9 (4)	O6—Tm1—O1—C1	69.1 (4)

C5—C6—C7—O5	5.0 (7)	O8—Tm1—O1—C1	136.7 (4)
N1—C6—C7—O4	1.3 (6)	O2 ⁱⁱ —Tm1—O1—C1	-150.3 (4)
C5—C6—C7—O4	-176.9 (4)	O4—Tm1—O1—C1	-6.8 (5)
C3—C2—N1—C6	1.3 (7)	N1—Tm1—O1—C1	-6.2 (4)
C1—C2—N1—C6	178.4 (4)	O1—C1—O2—Tm1 ⁱⁱⁱ	25.6 (9)
C3—C2—N1—Tm1	-173.2 (4)	C2—C1—O2—Tm1 ⁱⁱⁱ	-156.3 (4)
C1—C2—N1—Tm1	3.9 (6)	C5—C4—O3—Tm1 ^{iv}	104.5 (5)
C5—C6—N1—C2	-2.2 (7)	C3—C4—O3—Tm1 ^{iv}	-73.7 (5)
C7—C6—N1—C2	179.6 (4)	O5—C7—O4—Tm1	-177.8 (3)
C5—C6—N1—Tm1	172.3 (4)	C6—C7—O4—Tm1	4.1 (6)
C7—C6—N1—Tm1	-5.9 (5)	O3 ⁱ —Tm1—O4—C7	-179.8 (4)
O3 ⁱ —Tm1—N1—C2	-171.1 (3)	O7—Tm1—O4—C7	78.6 (4)
O7—Tm1—N1—C2	101.5 (4)	O6—Tm1—O4—C7	-90.2 (4)
O6—Tm1—N1—C2	-101.4 (4)	O1—Tm1—O4—C7	-4.6 (4)
O1—Tm1—N1—C2	0.5 (4)	O8—Tm1—O4—C7	-118.1 (4)
O8—Tm1—N1—C2	-42.9 (4)	O2 ⁱⁱ —Tm1—O4—C7	115.9 (4)
O2 ⁱⁱ —Tm1—N1—C2	47.9 (4)	N1—Tm1—O4—C7	-5.1 (3)

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

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

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O9—H8 <i>W</i> ...O2 ⁱⁱ	0.88	2.54	3.111 (6)	123
O9—H7 <i>W</i> ...O5 ^v	0.83	2.03	2.694 (6)	137
O8—H6 <i>W</i> ...O9 ^{vi}	0.95	1.73	2.679 (5)	179
O8—H5 <i>W</i> ...O9 ^{vii}	0.90	2.27	3.068 (6)	148
O7—H4 <i>W</i> ...O5 ^{viii}	0.95	1.79	2.697 (5)	158
O7—H3 <i>W</i> ...O1 ⁱⁱ	0.90 (3)	1.85 (3)	2.672 (5)	151 (5)
O6—H2 <i>W</i> ...O4 ^{ix}	0.83 (4)	2.11 (5)	2.791 (5)	139 (5)
O6—H1 <i>W</i> ...O3 ^x	0.91 (4)	1.84 (4)	2.706 (5)	156 (4)

Symmetry codes: (ii) $-x+1/2, y-1/2, -z+1/2$; (v) $-x+3/2, y-1/2, -z+1/2$; (vi) $x, y+1, z$; (vii) $-x+1, -y+1, -z+1$; (viii) $-x+1, -y+1, -z$; (ix) $-x+3/2, y+1/2, -z+1/2$; (x) $-x+1, -y+2, -z$.