

Bis{6,6'-dimethoxy-2,2'-[ethane-1,2-diyl-bis(iminomethylene)]diphenolato(1.5-)- κ^4O,N,N',O' }terbium(III)

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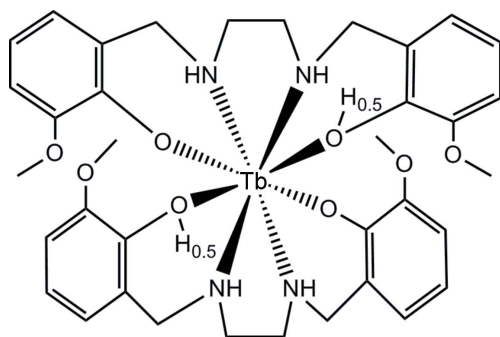
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.020$ Å; R factor = 0.044; wR factor = 0.126; data-to-parameter ratio = 12.7.

The title compound, $[\text{Tb}(\text{C}_{18}\text{H}_{22.5}\text{N}_2\text{O}_4)_2]$, is isotypic with its Pr and Tb analogues. All interatomic distances, angles and the hydrogen bond geometry are very similar for the three structures.

Related literature

For related structures, see: Liu *et al.*, (2007), Xia *et al.* (2006).
For isotypic structures, see: Xia *et al.* (2009a,b).



Experimental

Crystal data

$[\text{Tb}(\text{C}_{18}\text{H}_{22.5}\text{N}_2\text{O}_4)_2]$
 $M_r = 820.68$

Orthorhombic, $Iba2$
 $a = 21.885$ (2) Å

$b = 11.1407$ (10) Å
 $c = 14.0928$ (14) Å
 $V = 3436.0$ (6) Å³
 $Z = 4$

Mo $K\alpha$ radiation
 $\mu = 2.12$ mm⁻¹
 $T = 298$ (2) K
 $0.34 \times 0.19 \times 0.11$ mm

Data collection

Siemens SMART 1000 CCD area-detector diffractometer
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.533$, $T_{\max} = 0.801$

7735 measured reflections
2820 independent reflections
1893 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.046$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.126$
 $S = 1.08$
2820 reflections
222 parameters
1 restraint

H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 1.09$ e Å⁻³
 $\Delta\rho_{\text{min}} = -1.60$ e Å⁻³
Absolute structure: Flack (1983),
1230 Friedel pairs
Flack parameter: 0.07 (4)

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O3}-\text{H3C}\cdots\text{O4}$	0.85	2.10	2.640 (10)	121
$\text{N1}-\text{H1}\cdots\text{O4}^i$	0.91	2.34	3.226 (12)	166
$\text{N2}-\text{H2}\cdots\text{O2}^i$	0.91	2.58	3.459 (13)	162

Symmetry code: (i) $-x + 1, -y + 1, z$.

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); 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.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2682).

References

- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
Liu, Y.-F., Xia, H.-T., Wang, D.-Q. & Yang, S.-P. (2007). *Acta Cryst.* **E63**, m484–m486.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
Siemens. (1996). *SMART* and *SAINTE*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Xia, H.-T., Liu, Y.-F., Yang, S.-P. & Wang, D.-Q. (2006). *Acta Cryst.* **E62**, o5864–o5865.
Xia, H.-T., Liu, Y.-F., Yang, S.-P. & Wang, D.-Q. (2009a). *Acta Cryst.* **E65**, m200.
Xia, H.-T., Rong, D.-F., Zhang, Y.-Y., Yang, S.-P. & Wang, D.-Q. (2009b). *Acta Cryst.* **E65**, m198–m199.

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Acta Cryst. (2009). E65, m201 [doi:10.1107/S1600536809001494]

Bis{6,6'-dimethoxy-2,2'-[ethane-1,2-diylbis(iminomethylene)]diphenolato(1.5-) κ^4 O,N,N',O'}\}terbium(III)

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Comment

Diamine derivatives are potentially multidentate ligands. we have recently reported the crystal structure (C₁₈H₂₄O₂N₄) (II) (Xia *et al.*, 2006) which is the ligand of the title compound and two complexes [Ce(C₁₈H₂₂N₂O₄)₂] (III) (Liu *et al.*, 2007), [Er(C₁₈H_{22.5}N₂O₄)₂] (IV) (Xia *et al.*, 2009). We report here the crystal structure of new rare earth complex (I).

In the title complex (I), the coordination environment of the Tb atom and coordination modes of (I) ligands to Tb^{III} ion is in agreement with the complexes reported above (Fig. 1). The average bond lengths of between the terbium center oxygen atoms are 2.205 (7)Å and nitrogen atom are 2.616 (9) Å, longer than the 2.199 (4)Å and shorter than the 2.624 (4)Å of complexes (III), respectively, longer than those 2.203 (6)Å and longer than the 2.612 (8)Å of complexes (IV), respectively. The dihedral angles between phenyl ring (C4—C9 ring) and another phenyl ring are 41.85 (31)°(C12—C17 ring), 47.59 (30)°(C4A—C9A ring) and 15.27 (48)°(C12A—C17A ring) [symmetry codes: (A) 1 - x, 1 - y, z].

In (I), the Tb atom is eight-coordinated by four O atoms and four N atoms from two 6,6'-dimethoxy-2,2'-(ethane-1,2-diyl-diiminodimethylene)diphenol. The molecules are connected by van der Waals forces, resulting in a three-dimensional network.

Experimental

A solution of 6,6'-dimethoxy-2,2'-(ethane-1,2-diyl-diiminodimethylene) diphenol (0.328 g, 2 mmol) in ethanol (20 ml), and then a solution of Tb(NO₃)₃.6H₂O (0.454 g, 1 mmol) in ethanol (10 ml) was added. The reaction mixture was stirred for 3 h in the air and then filtered. X-ray quality crystals of (I) were obtained by evaporation of an ethanol solution.

Refinement

The space group was uniquely assigned from the systematic absences. All H atoms were located in difference Fourier maps. H atoms bonded to C, O and N atoms were treated as riding atoms, with C—H distances of 0.93 Å (aryl), 0.96 Å (methyl), 0.97Å (methylene) and N—H distances of 0.90 Å (amino), $U_{iso}(H) = 1.2U_{eq}(aryl, \text{methylene}, NH)$ or $1.5U_{eq}(C)$ (methyl or OH). The H3C bonded to O3 is disordered and were refined with the occupancies ties to 0.5.

Figures

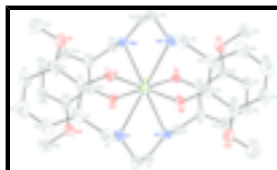


Fig. 1. The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are at the 30% probability level. For clarity, H atoms have been omitted. [Symmetry codes: (A) 1 - x, 1 - y, z].

supplementary materials

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Crystal data

[Tb(C ₁₈ H _{22.5} N ₂ O ₄) ₂]	$F_{000} = 1672$
$M_r = 820.68$	$D_x = 1.586 \text{ Mg m}^{-3}$
Orthorhombic, <i>Iba</i> 2	Mo $K\alpha$ radiation
Hall symbol: I 2 -2c	$\lambda = 0.71073 \text{ \AA}$
$a = 21.885 (2) \text{ \AA}$	Cell parameters from 3398 reflections
$b = 11.1407 (10) \text{ \AA}$	$\theta = 2.9\text{--}25.8^\circ$
$c = 14.0928 (14) \text{ \AA}$	$\mu = 2.12 \text{ mm}^{-1}$
$V = 3436.0 (6) \text{ \AA}^3$	$T = 298 (2) \text{ K}$
$Z = 4$	Block, brown
	$0.34 \times 0.19 \times 0.11 \text{ mm}$

Data collection

Siemens SMART 1000 CCD area-detector diffractometer	2820 independent reflections
Radiation source: fine-focus sealed tube	1893 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.046$
$T = 298(2) \text{ K}$	$\theta_{\text{max}} = 25.0^\circ$
φ and ω scans	$\theta_{\text{min}} = 1.9^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -26 \rightarrow 24$
$T_{\text{min}} = 0.533$, $T_{\text{max}} = 0.801$	$k = -13 \rightarrow 9$
7735 measured reflections	$l = -16 \rightarrow 13$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.044$	$w = 1/[\sigma^2(F_o^2) + (0.0351P)^2 + 45.2805P]$
$wR(F^2) = 0.126$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.08$	$(\Delta/\sigma)_{\text{max}} = 0.001$
2820 reflections	$\Delta\rho_{\text{max}} = 1.09 \text{ e \AA}^{-3}$
222 parameters	$\Delta\rho_{\text{min}} = -1.60 \text{ e \AA}^{-3}$
1 restraint	Extinction correction: none
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 1230 Freidel pairs
Secondary atom site location: difference Fourier map	Flack parameter: 0.07 (4)

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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Tb1	0.5000	0.5000	0.34645 (16)	0.03630 (19)	
N1	0.6048 (4)	0.5729 (9)	0.4107 (7)	0.044 (2)	
H1	0.6336	0.5343	0.3755	0.053*	
N2	0.5341 (4)	0.7104 (8)	0.2793 (7)	0.042 (2)	
H2	0.5156	0.7651	0.3177	0.051*	
O1	0.5348 (3)	0.3643 (7)	0.4462 (5)	0.0430 (19)	
O2	0.5618 (4)	0.1315 (8)	0.4292 (7)	0.063 (3)	
O3	0.4318 (3)	0.5592 (6)	0.2408 (5)	0.0363 (17)	
H3C	0.4011	0.5141	0.2510	0.054*	0.50
O4	0.3133 (3)	0.5867 (7)	0.2682 (6)	0.047 (2)	
C1	0.6151 (6)	0.7008 (13)	0.3952 (12)	0.052 (4)	
H1A	0.6575	0.7205	0.4073	0.063*	
H1B	0.5898	0.7474	0.4381	0.063*	
C2	0.5989 (6)	0.7296 (13)	0.2937 (12)	0.054 (4)	
H2A	0.6092	0.8125	0.2800	0.064*	
H2B	0.6221	0.6786	0.2512	0.064*	
C3	0.6163 (6)	0.5383 (13)	0.5089 (9)	0.059 (4)	
H3A	0.5802	0.5532	0.5471	0.071*	
H3B	0.6497	0.5853	0.5346	0.071*	
C4	0.6326 (6)	0.4039 (14)	0.5116 (9)	0.055 (3)	
C5	0.5898 (6)	0.3267 (14)	0.4759 (9)	0.045 (4)	
C6	0.6039 (6)	0.2012 (13)	0.4677 (9)	0.056 (4)	
C7	0.6629 (7)	0.1649 (16)	0.5002 (11)	0.061 (5)	
H7	0.6744	0.0846	0.4973	0.073*	
C8	0.7010 (7)	0.2462 (17)	0.5346 (11)	0.069 (4)	
H8	0.7395	0.2204	0.5538	0.082*	
C9	0.6884 (6)	0.3600 (16)	0.5434 (10)	0.065 (4)	
H9	0.7167	0.4117	0.5708	0.078*	
C10	0.5795 (8)	0.0145 (14)	0.4051 (13)	0.087 (5)	
H10A	0.6170	0.0172	0.3698	0.130*	
H10B	0.5482	-0.0220	0.3670	0.130*	
H10C	0.5853	-0.0317	0.4618	0.130*	
C11	0.5116 (6)	0.7377 (12)	0.1847 (9)	0.050 (3)	

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H11A	0.5211	0.6718	0.1422	0.060*
H11B	0.5316	0.8093	0.1609	0.060*
C12	0.4438 (6)	0.7571 (11)	0.1875 (8)	0.044 (3)
C13	0.4076 (6)	0.6638 (12)	0.2215 (9)	0.039 (3)
C14	0.3444 (5)	0.6833 (11)	0.2345 (8)	0.044 (3)
C15	0.3193 (7)	0.7932 (13)	0.2107 (10)	0.052 (4)
H15	0.2775	0.8066	0.2167	0.063*
C16	0.3576 (7)	0.8827 (13)	0.1777 (11)	0.059 (4)
H16	0.3410	0.9577	0.1643	0.070*
C17	0.4159 (7)	0.8665 (12)	0.1647 (9)	0.057 (3)
H17	0.4394	0.9285	0.1398	0.068*
C18	0.2533 (6)	0.6067 (14)	0.2980 (10)	0.067 (4)
H18A	0.2516	0.6790	0.3350	0.101*
H18B	0.2398	0.5402	0.3360	0.101*
H18C	0.2272	0.6146	0.2436	0.101*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Tb1	0.0277 (3)	0.0393 (3)	0.0419 (3)	0.0000 (3)	0.000	0.000
N1	0.032 (5)	0.052 (6)	0.049 (6)	0.003 (5)	0.002 (4)	-0.017 (5)
N2	0.035 (5)	0.041 (6)	0.051 (6)	-0.005 (4)	0.008 (4)	-0.001 (5)
O1	0.029 (4)	0.058 (5)	0.042 (4)	0.012 (4)	-0.002 (3)	0.008 (4)
O2	0.056 (6)	0.057 (6)	0.075 (7)	0.017 (5)	0.012 (5)	0.016 (5)
O3	0.034 (4)	0.033 (4)	0.041 (4)	0.007 (4)	-0.010 (4)	-0.004 (4)
O4	0.035 (4)	0.055 (5)	0.052 (5)	0.006 (4)	-0.004 (4)	0.002 (4)
C1	0.032 (7)	0.057 (9)	0.068 (10)	-0.009 (6)	0.007 (6)	-0.019 (8)
C2	0.042 (8)	0.047 (8)	0.072 (10)	-0.011 (6)	0.014 (7)	-0.011 (7)
C3	0.047 (7)	0.076 (10)	0.055 (8)	0.000 (7)	-0.001 (6)	-0.014 (7)
C4	0.041 (7)	0.078 (10)	0.047 (7)	0.012 (7)	-0.003 (6)	-0.002 (7)
C5	0.037 (8)	0.063 (10)	0.033 (8)	0.013 (8)	0.004 (6)	0.003 (7)
C6	0.046 (8)	0.071 (10)	0.052 (8)	0.021 (7)	0.007 (6)	0.012 (7)
C7	0.049 (10)	0.075 (12)	0.060 (10)	0.021 (9)	0.007 (7)	0.012 (8)
C8	0.054 (9)	0.092 (13)	0.060 (9)	0.018 (9)	-0.001 (7)	0.007 (9)
C9	0.050 (8)	0.091 (12)	0.055 (8)	0.009 (8)	-0.005 (7)	-0.002 (8)
C10	0.084 (11)	0.069 (11)	0.107 (12)	0.008 (9)	0.011 (9)	0.015 (10)
C11	0.053 (9)	0.044 (7)	0.053 (7)	-0.004 (6)	0.011 (6)	0.000 (6)
C12	0.048 (7)	0.042 (7)	0.044 (7)	0.005 (6)	0.003 (6)	0.001 (6)
C13	0.040 (8)	0.043 (8)	0.034 (8)	0.007 (7)	-0.006 (6)	-0.005 (6)
C14	0.045 (7)	0.047 (7)	0.040 (6)	0.009 (6)	-0.005 (6)	-0.002 (6)
C15	0.047 (8)	0.055 (8)	0.055 (9)	0.014 (7)	-0.005 (6)	0.000 (7)
C16	0.065 (10)	0.051 (9)	0.060 (9)	0.014 (7)	-0.006 (7)	0.001 (7)
C17	0.063 (9)	0.047 (8)	0.060 (8)	0.001 (7)	0.005 (7)	0.004 (7)
C18	0.045 (8)	0.080 (10)	0.077 (9)	-0.001 (7)	-0.001 (7)	0.007 (8)

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

Tb1—O1	2.200 (7)	C3—H3B	0.9700
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Tb1—O1 ⁱ	2.200 (7)	C4—C5	1.37 (2)
Tb1—O3 ⁱ	2.210 (7)	C4—C9	1.390 (18)
Tb1—O3	2.210 (7)	C5—C6	1.436 (19)
Tb1—N1	2.596 (9)	C6—C7	1.427 (19)
Tb1—N1 ⁱ	2.596 (9)	C7—C8	1.32 (2)
Tb1—N2 ⁱ	2.636 (9)	C7—H7	0.9300
Tb1—N2	2.636 (9)	C8—C9	1.30 (2)
Tb1—H3C	2.5530	C8—H8	0.9300
N1—C3	1.458 (16)	C9—H9	0.9300
N1—C1	1.458 (17)	C10—H10A	0.9600
N1—H1	0.9100	C10—H10B	0.9600
N2—C2	1.449 (16)	C10—H10C	0.9600
N2—C11	1.453 (15)	C11—C12	1.500 (17)
N2—H2	0.9100	C11—H11A	0.9700
O1—C5	1.342 (15)	C11—H11B	0.9700
O2—C6	1.323 (16)	C12—C13	1.392 (18)
O2—C10	1.402 (17)	C12—C17	1.400 (17)
O3—C13	1.308 (14)	C13—C14	1.412 (17)
O3—H3C	0.8499	C14—C15	1.383 (18)
O4—C14	1.360 (14)	C15—C16	1.38 (2)
O4—C18	1.395 (15)	C15—H15	0.9300
C1—C2	1.508 (17)	C16—C17	1.30 (2)
C1—H1A	0.9700	C16—H16	0.9300
C1—H1B	0.9700	C17—H17	0.9300
C2—H2A	0.9700	C18—H18A	0.9600
C2—H2B	0.9700	C18—H18B	0.9600
C3—C4	1.54 (2)	C18—H18C	0.9600
C3—H3A	0.9700		
O1—Tb1—O1 ⁱ	100.6 (4)	N2—C2—H2B	109.8
O1—Tb1—O3 ⁱ	89.5 (3)	C1—C2—H2B	109.8
O1 ⁱ —Tb1—O3 ⁱ	150.4 (3)	H2A—C2—H2B	108.3
O1—Tb1—O3	150.4 (3)	N1—C3—C4	108.7 (10)
O1 ⁱ —Tb1—O3	89.5 (3)	N1—C3—H3A	109.9
O3 ⁱ —Tb1—O3	95.3 (4)	C4—C3—H3A	109.9
O1—Tb1—N1	71.7 (3)	N1—C3—H3B	109.9
O1 ⁱ —Tb1—N1	82.4 (3)	C4—C3—H3B	109.9
O3 ⁱ —Tb1—N1	74.4 (3)	H3A—C3—H3B	108.3
O3—Tb1—N1	137.6 (3)	C5—C4—C9	119.9 (14)
O1—Tb1—N1 ⁱ	82.4 (3)	C5—C4—C3	116.4 (11)
O1 ⁱ —Tb1—N1 ⁱ	71.7 (3)	C9—C4—C3	123.6 (13)
O3 ⁱ —Tb1—N1 ⁱ	137.6 (3)	O1—C5—C4	122.2 (13)
O3—Tb1—N1 ⁱ	74.4 (3)	O1—C5—C6	118.2 (13)
N1—Tb1—N1 ⁱ	139.2 (4)	C4—C5—C6	119.6 (13)
O1—Tb1—N2 ⁱ	73.5 (3)	O2—C6—C7	126.6 (14)
O1 ⁱ —Tb1—N2 ⁱ	137.9 (3)	O2—C6—C5	117.0 (12)

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O3 ⁱ —Tb1—N2 ⁱ	71.6 (3)	C7—C6—C5	116.4 (15)
O3—Tb1—N2 ⁱ	80.3 (3)	C8—C7—C6	119.5 (16)
N1—Tb1—N2 ⁱ	130.8 (3)	C8—C7—H7	120.2
N1 ⁱ —Tb1—N2 ⁱ	66.2 (3)	C6—C7—H7	120.2
O1—Tb1—N2	137.9 (3)	C9—C8—C7	124.5 (16)
O1 ⁱ —Tb1—N2	73.5 (3)	C9—C8—H8	117.7
O3 ⁱ —Tb1—N2	80.3 (3)	C7—C8—H8	117.7
O3—Tb1—N2	71.6 (3)	C8—C9—C4	119.8 (15)
N1—Tb1—N2	66.2 (3)	C8—C9—H9	120.1
N1 ⁱ —Tb1—N2	130.8 (3)	C4—C9—H9	120.1
N2 ⁱ —Tb1—N2	137.9 (4)	O2—C10—H10A	109.5
O1—Tb1—H3C	132.2	O2—C10—H10B	109.5
O1 ⁱ —Tb1—H3C	90.1	H10A—C10—H10B	109.5
O3 ⁱ —Tb1—H3C	103.7	O2—C10—H10C	109.5
O3—Tb1—H3C	18.8	H10A—C10—H10C	109.5
N1—Tb1—H3C	156.0	H10B—C10—H10C	109.5
N1 ⁱ —Tb1—H3C	56.9	N2—C11—C12	110.0 (9)
N2 ⁱ —Tb1—H3C	68.0	N2—C11—H11A	109.7
N2—Tb1—H3C	89.8	C12—C11—H11A	109.7
C3—N1—C1	112.0 (11)	N2—C11—H11B	109.7
C3—N1—Tb1	113.6 (8)	C12—C11—H11B	109.7
C1—N1—Tb1	112.9 (7)	H11A—C11—H11B	108.2
C3—N1—H1	105.9	C13—C12—C17	118.7 (12)
C1—N1—H1	105.9	C13—C12—C11	117.7 (11)
Tb1—N1—H1	105.9	C17—C12—C11	123.4 (11)
C2—N2—C11	115.4 (10)	O3—C13—C12	120.5 (12)
C2—N2—Tb1	111.0 (8)	O3—C13—C14	120.4 (12)
C11—N2—Tb1	114.8 (7)	C12—C13—C14	119.2 (12)
C2—N2—H2	104.8	O4—C14—C15	125.8 (11)
C11—N2—H2	104.8	O4—C14—C13	114.5 (11)
Tb1—N2—H2	104.8	C15—C14—C13	119.6 (12)
C5—O1—Tb1	136.4 (8)	C14—C15—C16	118.5 (14)
C6—O2—C10	116.9 (12)	C14—C15—H15	120.7
C13—O3—Tb1	132.8 (7)	C16—C15—H15	120.7
C13—O3—H3C	104.1	C17—C16—C15	122.8 (14)
Tb1—O3—H3C	104.1	C17—C16—H16	118.6
C14—O4—C18	116.8 (10)	C15—C16—H16	118.6
N1—C1—C2	108.3 (12)	C16—C17—C12	121.1 (14)
N1—C1—H1A	110.0	C16—C17—H17	119.5
C2—C1—H1A	110.0	C12—C17—H17	119.5
N1—C1—H1B	110.0	O4—C18—H18A	109.5
C2—C1—H1B	110.0	O4—C18—H18B	109.5
H1A—C1—H1B	108.4	H18A—C18—H18B	109.5
N2—C2—C1	109.3 (12)	O4—C18—H18C	109.5
N2—C2—H2A	109.8	H18A—C18—H18C	109.5
C1—C2—H2A	109.8	H18B—C18—H18C	109.5

O1—Tb1—N1—C3	-33.9 (8)	N1—C1—C2—N2	65.1 (13)
O1 ⁱ —Tb1—N1—C3	69.9 (8)	C1—N1—C3—C4	-155.3 (10)
O3 ⁱ —Tb1—N1—C3	-128.7 (8)	Tb1—N1—C3—C4	75.3 (11)
O3—Tb1—N1—C3	150.8 (7)	N1—C3—C4—C5	-58.5 (15)
N1 ⁱ —Tb1—N1—C3	19.6 (7)	N1—C3—C4—C9	118.1 (13)
N2 ⁱ —Tb1—N1—C3	-81.0 (9)	Tb1—O1—C5—C4	53.7 (18)
N2—Tb1—N1—C3	145.2 (9)	Tb1—O1—C5—C6	-125.1 (12)
O1—Tb1—N1—C1	-162.8 (9)	C9—C4—C5—O1	178.6 (11)
O1 ⁱ —Tb1—N1—C1	-59.0 (8)	C3—C4—C5—O1	-4.6 (19)
O3 ⁱ —Tb1—N1—C1	102.4 (9)	C9—C4—C5—C6	-3(2)
O3—Tb1—N1—C1	21.9 (10)	C3—C4—C5—C6	174.1 (11)
N1 ⁱ —Tb1—N1—C1	-109.3 (8)	C10—O2—C6—C7	-10 (2)
N2 ⁱ —Tb1—N1—C1	150.1 (8)	C10—O2—C6—C5	168.5 (12)
N2—Tb1—N1—C1	16.3 (8)	O1—C5—C6—O2	1.4 (18)
O1—Tb1—N2—C2	18.6 (10)	C4—C5—C6—O2	-177.4 (12)
O1 ⁱ —Tb1—N2—C2	106.2 (9)	O1—C5—C6—C7	-179.9 (12)
O3 ⁱ —Tb1—N2—C2	-59.8 (8)	C4—C5—C6—C7	1.3 (19)
O3—Tb1—N2—C2	-158.7 (9)	O2—C6—C7—C8	177.8 (14)
N1—Tb1—N2—C2	17.3 (8)	C5—C6—C7—C8	-1(2)
N1 ⁱ —Tb1—N2—C2	152.7 (8)	C6—C7—C8—C9	2(2)
N2 ⁱ —Tb1—N2—C2	-108.0 (8)	C7—C8—C9—C4	-3(2)
O1—Tb1—N2—C11	151.7 (7)	C5—C4—C9—C8	3(2)
O1 ⁱ —Tb1—N2—C11	-120.7 (8)	C3—C4—C9—C8	-173.1 (13)
O3 ⁱ —Tb1—N2—C11	73.3 (7)	C2—N2—C11—C12	-159.9 (10)
O3—Tb1—N2—C11	-25.6 (7)	Tb1—N2—C11—C12	69.1 (11)
N1—Tb1—N2—C11	150.5 (8)	N2—C11—C12—C13	-58.3 (15)
N1 ⁱ —Tb1—N2—C11	-74.1 (8)	N2—C11—C12—C17	116.7 (13)
N2 ⁱ —Tb1—N2—C11	25.2 (7)	Tb1—O3—C13—C12	64.3 (16)
O1 ⁱ —Tb1—O1—C5	-106.5 (12)	Tb1—O3—C13—C14	-115.9 (11)
O3 ⁱ —Tb1—O1—C5	45.5 (12)	C17—C12—C13—O3	177.9 (11)
O3—Tb1—O1—C5	145.3 (11)	C11—C12—C13—O3	-6.9 (18)
N1—Tb1—O1—C5	-28.3 (11)	C17—C12—C13—C14	-1.9 (18)
N1 ⁱ —Tb1—O1—C5	-176.3 (12)	C11—C12—C13—C14	173.4 (11)
N2 ⁱ —Tb1—O1—C5	116.4 (12)	C18—O4—C14—C15	-12.7 (17)
N2—Tb1—O1—C5	-29.5 (13)	C18—O4—C14—C13	169.1 (11)
O1—Tb1—O3—C13	144.3 (10)	O3—C13—C14—O4	0.5 (17)
O1 ⁱ —Tb1—O3—C13	33.4 (11)	C12—C13—C14—O4	-179.8 (11)
O3 ⁱ —Tb1—O3—C13	-117.4 (11)	O3—C13—C14—C15	-177.8 (12)
N1—Tb1—O3—C13	-44.8 (12)	C12—C13—C14—C15	1.9 (19)
N1 ⁱ —Tb1—O3—C13	104.5 (11)	O4—C14—C15—C16	179.6 (12)
N2 ⁱ —Tb1—O3—C13	172.3 (11)	C13—C14—C15—C16	-2(2)
N2—Tb1—O3—C13	-39.4 (11)	C14—C15—C16—C17	3(2)
C3—N1—C1—C2	-177.0 (11)	C15—C16—C17—C12	-3(2)
Tb1—N1—C1—C2	-47.3 (12)	C13—C12—C17—C16	2(2)

supplementary materials

C11—N2—C2—C1	178.6 (11)	C11—C12—C17—C16	-172.7 (13)
Tb1—N2—C2—C1	-48.6 (12)		

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

Hydrogen-bond geometry (Å, °)

<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>
O3—H3C \cdots O4	0.85	2.10	2.640 (10)	121
N1—H1 \cdots O4 ⁱ	0.91	2.34	3.226 (12)	166
N2—H2 \cdots O2 ⁱ	0.91	2.58	3.459 (13)	162

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

Fig. 1

