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Poly[diaquatris(μ_4-benzene-1,3-dicarboxylato)diterbium(III)]

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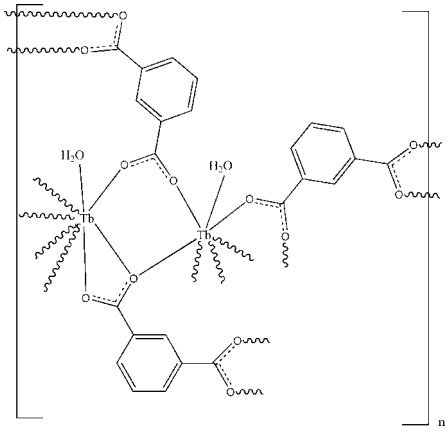
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.009 Å; disorder in main residue; R factor = 0.034; wR factor = 0.073; data-to-parameter ratio = 12.6.

The title terbium coordination polymer, $\{[Tb_2(C_8H_4O_4)_3(H_2O)_2]\}_n$, was obtained by the hydrothermal reaction of $Tb(NO_3)_3$ with benzene-1,3-dicarboxylic acid (1,3-BDC) in alkaline aqueous solution. In the asymmetric unit, there are two crystallographically independent Tb^{III} ions: one seven-coordinate Tb centre is coordinated by six O atoms from six 1,3-BDC ligands and one water molecule, and the other Tb centre is eight-coordinate and surrounded by seven O atoms from six 1,3-BDC ligands and one water molecule. The bridging ligands link the metal centres, forming a three-dimensional network which is further stabilized by hydrogen-bonding interactions. One benzene ring is disordered over two positions with site occupancies of 0.60 (3) and 0.40 (3).

Related literature

De Bettencourt-Dias (2005) and Zhang *et al.* (2003) have described the use of benzene-1,3-dicarboxylic acid (1,3-BDC) in the construction of supramolecular complexes.



Experimental

Crystal data

$[Tb_2(C_8H_4O_4)_3(H_2O)_2]$
M_r = 846.21
Monoclinic, $P2_1/n$
a = 13.2647 (3) Å
b = 14.3741 (3) Å
c = 13.5323 (3) Å
β = 103.944 (1)°
V = 2504.14 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 5.68 mm^{-1}
T = 296 (2) K
0.15 × 0.14 × 0.11 mm

Data collection

Bruker APEX-II area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.445, T_{max} = 0.538
35926 measured reflections
5182 independent reflections
3834 reflections with $I > 2\sigma(I)$
R_{int} = 0.077

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.073
S = 1.02
5182 reflections
410 parameters
66 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 2.21 e Å^{-3}
$\Delta\rho_{min}$ = −1.08 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H1W···O7^{i}	0.818 (10)	2.04 (4)	2.789 (7)	151 (7)
O1W—H2W···O4^{ii}	0.819 (10)	2.29 (4)	3.020 (7)	149 (7)
O2W—H3W···O2^{iii}	0.823 (10)	1.959 (14)	2.780 (7)	176 (9)
C3—H3···O8^{iv}	0.93	2.35	3.257 (8)	164
C11′—H11′···O1^{v}	0.93	2.39	3.284 (10)	161

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); 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, 2004); software used to prepare material for publication: *SHELXTL*.

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: ZL2078).

References

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supplementary materials

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Poly[diaquatris(μ_4-benzene-1,3-dicarboxylato)diterbium(III)]

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Comment

As a building block, benzene-1,3-dicarboxylic acid (1,3-BDC) is an excellent candidate for the construction of supramolecular complexes (De Bettencourt-Dias, 2005; Zhang *et al.*, 2003). Recently, we obtained the title coordination polymer by the reaction of $Tb(NO_3)_3$ with benzene-1,3-dicarboxylic acid in alkaline aqueous solution, and its crystal structure is reported here.

In the asymmetric unit of the title complex, there are two crystallographically independent Tb^{III} ions: one seven-coordinate Tb centre is coordinated by six oxygen atoms from six 1,3-BDC ligands and one water molecule, and the other Tb centre is eight-coordinate and surrounded by seven oxygen atoms from six 1,3-BDC ligands and one water molecules (Fig. 1). The adjacent Tb···Tb separations are 4.690 (4) and 4.698 (5) Å, respectively. The carboxylate groups of 1,3-BDC ligands are distinct, one of which acts as bis(monodentate) bridge, the other acts as chelate and monodentate bridge. They cross-link the metal ions to form a three-dimensional network (Fig. 2). The crystal structure is stabilized by intra/intermolecular O—H···O and C—H···O hydrogen bonding interactions (Table 1).

Experimental

A mixture of $Tb(NO_3)_3$ (0.5 mmol, 0.172 g), benzenedicarboxylic acid (0.75 mmol, 0.125 g), NaOH (1.5 mmol; 0.06 g) and H_2O (12 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 dryed in air.

Refinement

One of the phenyl rings was found to be split into two positions and the occupancy ratio refined to 0.60 (3) to 0.40 (3). Due to the significant overlap of the disordered atoms the following restraints were applied: The phenyl ring C10 C11 C12 C13 C14 C15 and its disordered counterpart were each restrained to be flat and their equivalent bond distances were restrained to be the same within a standard deviation of 0.01 Å. All water H atoms were tentatively located in difference density Fourier maps and were refined with O–H distance restraints of 0.82 (1) Å and with U_{iso}(H) = 1.5 U_{eq}(O). Carbon-bound H atoms were placed at calculated positions and were treated as riding on the parent C atoms with C—H = 0.93 Å, and with U_{iso}(H) = 1.2 U_{eq}(C).

Figures

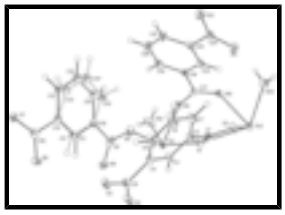


Fig. 1. The molecular structure showing the atomic-numbering scheme and displacement ellipsoids drawn at the 30% probability level.

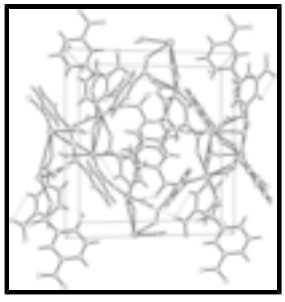


Fig. 2. The molecular packing showing the intermolecular hydrogen bonding interactions as broken lines. The minor moieties of the disordered phenyl rings were omitted for clarity.

Poly[diaquatris(m$_4$-benzene-1,3-dicarboxylato)diterbium(III)]

Crystal data

$[Tb_2(C_8H_4O_4)_3(H_2O)_2]$	F_{000} = 1608
M_r = 846.21	D_x = 2.245 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 4289 reflections
a = 13.2647 (3) Å	θ = 1.7–28.0º
b = 14.3741 (3) Å	μ = 5.68 mm^{-1}
c = 13.5323 (3) Å	T = 296 (2) K
β = 103.9440 (10)º	Block, colorless
V = 2504.14 (9) Å^3	0.15 × 0.14 × 0.11 mm
Z = 4	

Data collection

Bruker APEX-II area-detector diffractometer	5182 independent reflections
Radiation source: fine-focus sealed tube	3834 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.077
T = 296(2) K	θ_{max} = 26.5º
φ and ω scan	θ_{min} = 1.9º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −16→16
T_{min} = 0.445, T_{max} = 0.538	k = −17→18
35926 measured reflections	l = −16→16

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.034$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.073$	$w = 1/[\sigma^2(F_o^2) + (0.0245P)^2 + 8.8675P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} = 0.001$
5182 reflections	$\Delta\rho_{max} = 2.21$ e Å^{-3}
410 parameters	$\Delta\rho_{min} = -1.08$ e Å^{-3}
66 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.8731 (5)	0.1394 (4)	0.1428 (4)	0.0231 (14)	
C2	0.9120 (5)	0.2379 (4)	0.1428 (5)	0.0273 (15)	
C3	0.8840 (5)	0.3000 (4)	0.2099 (5)	0.0292 (15)	
H3	0.8430	0.2801	0.2528	0.035*	
C4	0.9166 (5)	0.3912 (4)	0.2132 (5)	0.0285 (15)	
C5	0.9762 (6)	0.4214 (5)	0.1484 (6)	0.0418 (19)	
H5	0.9974	0.4832	0.1498	0.050*	
C6	1.0044 (7)	0.3588 (5)	0.0808 (6)	0.051 (2)	
H6	1.0443	0.3788	0.0370	0.061*	
C7	0.9728 (6)	0.2667 (5)	0.0792 (6)	0.0382 (18)	
H7	0.9925	0.2245	0.0353	0.046*	
C8	0.8853 (5)	0.4571 (4)	0.2847 (5)	0.0272 (15)	
C9	0.6369 (6)	0.4092 (5)	0.1246 (5)	0.0339 (17)	
C10	0.6583 (6)	0.4838 (5)	0.0566 (5)	0.0377 (18)	
C11	0.6834 (13)	0.5728 (7)	0.0950 (9)	0.021 (7)	0.40 (3)
H11	0.6739	0.5890	0.1587	0.025*	0.40 (3)

C13	0.738 (2)	0.6150 (13)	−0.0580 (11)	0.026 (5)	0.40 (3)
H13	0.7678	0.6582	−0.0932	0.032*	0.40 (3)
C14	0.708 (3)	0.5288 (13)	−0.1007 (15)	0.041 (6)	0.40 (3)
H14	0.7083	0.5155	−0.1677	0.049*	0.40 (3)
C15	0.677 (2)	0.4638 (14)	−0.0398 (12)	0.039 (6)	0.40 (3)
H15	0.6670	0.4029	−0.0634	0.047*	0.40 (3)
C11'	0.7033 (11)	0.5661 (6)	0.1000 (7)	0.022 (4)	0.60 (3)
H11'	0.7203	0.5736	0.1703	0.026*	0.60 (3)
C13'	0.6938 (16)	0.6260 (9)	−0.0677 (7)	0.033 (4)	0.60 (3)
H13'	0.7037	0.6746	−0.1097	0.040*	0.60 (3)
C14'	0.6506 (17)	0.5431 (9)	−0.1100 (10)	0.039 (4)	0.60 (3)
H14'	0.6362	0.5353	−0.1802	0.047*	0.60 (3)
C15'	0.6286 (15)	0.4723 (9)	−0.0504 (7)	0.033 (4)	0.60 (3)
H15'	0.5952	0.4185	−0.0795	0.040*	0.60 (3)
C12	0.7224 (5)	0.6369 (4)	0.0379 (5)	0.0312 (16)	
C16	0.7631 (5)	0.7280 (4)	0.0848 (5)	0.0226 (14)	
C17	0.6837 (5)	0.0910 (4)	−0.0429 (4)	0.0225 (14)	
C18	0.7180 (5)	0.1586 (4)	−0.1142 (5)	0.0258 (15)	
C19	0.6553 (6)	0.2320 (5)	−0.1574 (5)	0.0332 (16)	
H19	0.5897	0.2385	−0.1450	0.040*	
C20	0.6886 (6)	0.2954 (5)	−0.2183 (5)	0.0378 (18)	
H20	0.6445	0.3427	−0.2497	0.045*	
C21	0.7881 (5)	0.2886 (5)	−0.2329 (5)	0.0341 (17)	
H21	0.8119	0.3332	−0.2716	0.041*	
C22	0.8525 (5)	0.2159 (4)	−0.1901 (5)	0.0270 (15)	
C23	0.8166 (5)	0.1504 (5)	−0.1316 (4)	0.0256 (15)	
H23	0.8589	0.1006	−0.1038	0.031*	
C24	0.9607 (5)	0.2067 (5)	−0.2044 (5)	0.0279 (15)	
O1	0.7873 (3)	0.1236 (3)	0.1631 (3)	0.0279 (10)	
O2	0.9288 (4)	0.0743 (3)	0.1211 (4)	0.0335 (11)	
O3	0.9080 (4)	0.5415 (3)	0.2778 (4)	0.0390 (12)	
O4	0.8359 (4)	0.4262 (3)	0.3473 (4)	0.0364 (12)	
O5	0.6263 (4)	0.3265 (3)	0.0916 (4)	0.0436 (13)	
O6	0.6325 (4)	0.4317 (3)	0.2125 (4)	0.0423 (13)	
O7	0.7630 (3)	0.7967 (3)	0.0277 (3)	0.0290 (11)	
O8	0.7966 (4)	0.7328 (3)	0.1800 (3)	0.0311 (11)	
O9	0.6139 (3)	0.1186 (3)	−0.0012 (3)	0.0301 (11)	
O10	0.7277 (3)	0.0131 (3)	−0.0287 (3)	0.0275 (10)	
O11	1.0184 (4)	0.1465 (3)	−0.1523 (3)	0.0329 (11)	
O12	0.9859 (4)	0.2601 (3)	−0.2675 (4)	0.0360 (12)	
O1W	0.4451 (4)	0.1564 (5)	0.0500 (4)	0.0564 (17)	
H1W	0.387 (3)	0.178 (6)	0.048 (6)	0.085*	
H2W	0.436 (6)	0.121 (5)	0.002 (5)	0.085*	
O2W	0.8967 (4)	−0.0717 (4)	−0.0413 (4)	0.0432 (13)	
H3W	0.948 (3)	−0.075 (6)	−0.066 (5)	0.065*	
H4W	0.849 (3)	−0.059 (6)	−0.091 (3)	0.065*	
Tb1	0.61128 (2)	0.18274 (2)	0.15619 (2)	0.02036 (8)	
Tb2	0.82716 (2)	−0.06625 (2)	0.11562 (2)	0.02429 (9)	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.022 (4)	0.025 (3)	0.022 (3)	0.002 (3)	0.004 (3)	−0.006 (3)
C2	0.026 (4)	0.023 (4)	0.035 (4)	−0.004 (3)	0.011 (3)	−0.002 (3)
C3	0.029 (4)	0.025 (4)	0.036 (4)	−0.004 (3)	0.013 (3)	−0.010 (3)
C4	0.027 (4)	0.021 (3)	0.041 (4)	0.001 (3)	0.016 (3)	−0.004 (3)
C5	0.058 (5)	0.017 (4)	0.058 (5)	−0.009 (4)	0.031 (4)	−0.006 (3)
C6	0.066 (6)	0.034 (5)	0.069 (6)	−0.012 (4)	0.051 (5)	−0.008 (4)
C7	0.046 (5)	0.029 (4)	0.048 (4)	−0.001 (3)	0.028 (4)	−0.008 (3)
C8	0.021 (4)	0.024 (4)	0.039 (4)	0.000 (3)	0.012 (3)	−0.007 (3)
C9	0.039 (5)	0.030 (4)	0.031 (4)	−0.005 (3)	0.003 (3)	0.008 (3)
C10	0.053 (5)	0.024 (4)	0.034 (4)	−0.010 (3)	0.007 (4)	0.005 (3)
C11	0.013 (8)	0.018 (9)	0.026 (10)	0.006 (7)	−0.006 (6)	−0.001 (7)
C13	0.028 (10)	0.020 (8)	0.031 (8)	0.002 (7)	0.008 (7)	−0.002 (6)
C14	0.055 (11)	0.040 (9)	0.030 (8)	0.003 (8)	0.016 (8)	−0.009 (7)
C15	0.040 (11)	0.033 (9)	0.046 (9)	−0.007 (8)	0.012 (7)	−0.006 (7)
C11'	0.028 (7)	0.019 (7)	0.019 (6)	0.001 (5)	0.006 (5)	0.004 (5)
C13'	0.039 (8)	0.025 (6)	0.032 (6)	−0.001 (6)	0.001 (5)	0.004 (5)
C14'	0.050 (9)	0.033 (7)	0.032 (6)	−0.005 (6)	0.006 (6)	0.001 (5)
C15'	0.034 (8)	0.027 (6)	0.038 (6)	−0.009 (6)	0.006 (5)	−0.003 (5)
C12	0.042 (4)	0.022 (4)	0.029 (4)	−0.004 (3)	0.007 (3)	0.001 (3)
C16	0.016 (3)	0.024 (4)	0.028 (3)	−0.002 (3)	0.005 (3)	−0.002 (3)
C17	0.015 (3)	0.030 (4)	0.022 (3)	−0.003 (3)	0.003 (3)	−0.001 (3)
C18	0.024 (4)	0.026 (4)	0.030 (3)	−0.003 (3)	0.012 (3)	−0.002 (3)
C19	0.024 (4)	0.038 (4)	0.038 (4)	0.007 (3)	0.010 (3)	0.001 (3)
C20	0.031 (4)	0.034 (4)	0.049 (4)	0.011 (3)	0.010 (3)	0.010 (3)
C21	0.032 (4)	0.030 (4)	0.042 (4)	0.006 (3)	0.014 (3)	0.007 (3)
C22	0.026 (4)	0.025 (4)	0.032 (4)	0.005 (3)	0.013 (3)	0.003 (3)
C23	0.025 (4)	0.031 (4)	0.023 (3)	0.000 (3)	0.009 (3)	0.002 (3)
C24	0.027 (4)	0.030 (4)	0.028 (3)	0.004 (3)	0.009 (3)	0.007 (3)
O1	0.021 (3)	0.031 (3)	0.033 (3)	−0.006 (2)	0.010 (2)	−0.006 (2)
O2	0.029 (3)	0.022 (3)	0.052 (3)	−0.002 (2)	0.015 (2)	−0.009 (2)
O3	0.051 (3)	0.021 (3)	0.050 (3)	−0.003 (2)	0.023 (3)	−0.009 (2)
O4	0.040 (3)	0.026 (3)	0.049 (3)	−0.004 (2)	0.023 (2)	−0.009 (2)
O5	0.063 (4)	0.020 (3)	0.048 (3)	−0.010 (2)	0.014 (3)	0.005 (2)
O6	0.063 (4)	0.028 (3)	0.036 (3)	−0.016 (3)	0.011 (2)	0.006 (2)
O7	0.036 (3)	0.017 (2)	0.033 (2)	−0.004 (2)	0.008 (2)	0.0023 (19)
O8	0.030 (3)	0.035 (3)	0.028 (2)	−0.004 (2)	0.008 (2)	−0.004 (2)
O9	0.028 (3)	0.037 (3)	0.029 (2)	0.002 (2)	0.013 (2)	−0.001 (2)
O10	0.029 (3)	0.025 (3)	0.034 (3)	0.004 (2)	0.017 (2)	0.003 (2)
O11	0.028 (3)	0.034 (3)	0.041 (3)	0.002 (2)	0.018 (2)	0.009 (2)
O12	0.029 (3)	0.041 (3)	0.043 (3)	0.003 (2)	0.019 (2)	0.016 (2)
O1W	0.023 (3)	0.088 (5)	0.055 (4)	0.007 (3)	0.003 (3)	−0.038 (3)
O2W	0.043 (3)	0.049 (3)	0.045 (3)	0.006 (3)	0.025 (2)	0.003 (3)
Tb1	0.02051 (17)	0.01598 (16)	0.02636 (16)	−0.00013 (13)	0.00914 (12)	−0.00032 (12)
Tb2	0.02325 (18)	0.01728 (16)	0.03264 (18)	−0.00032 (13)	0.00735 (13)	0.00021 (13)

Geometric parameters (Å, °)

C1—O1	1.255 (7)	C17—O10	1.256 (7)
C1—O2	1.269 (7)	C17—O9	1.259 (7)
C1—C2	1.507 (9)	C17—C18	1.515 (8)
C2—C7	1.377 (9)	C18—C19	1.383 (9)
C2—C3	1.387 (9)	C18—C23	1.388 (9)
C3—C4	1.378 (9)	C19—C20	1.371 (10)
C3—H3	0.9300	C19—H19	0.9300
C4—C5	1.385 (9)	C20—C21	1.384 (10)
C4—C8	1.483 (9)	C20—H20	0.9300
C5—C6	1.397 (10)	C21—C22	1.385 (9)
C5—H5	0.9300	C21—H21	0.9300
C6—C7	1.388 (10)	C22—C23	1.386 (9)
C6—H6	0.9300	C22—C24	1.500 (9)
C7—H7	0.9300	C23—H23	0.9300
C8—O3	1.259 (7)	C24—O12	1.251 (7)
C8—O4	1.269 (8)	C24—O11	1.254 (7)
C9—O6	1.248 (8)	O1—Tb1	2.465 (4)
C9—O5	1.265 (8)	O1—Tb2	2.882 (4)
C9—C10	1.484 (9)	O2—Tb2	2.420 (4)
C10—C11'	1.390 (10)	O3—Tb1^{i}	2.258 (4)
C10—C11	1.390 (11)	O4—Tb2^{i}	2.338 (5)
C10—C15	1.413 (13)	O5—Tb1	2.271 (4)
C10—C15'	1.417 (11)	O6—Tb2^{i}	2.258 (5)
C11—C12	1.382 (11)	O7—Tb2^{ii}	2.352 (4)
C11—H11	0.9300	O8—Tb1^{i}	2.370 (4)
C13—C14	1.384 (13)	O9—Tb1	2.329 (4)
C13—C12	1.396 (12)	O10—Tb2	2.369 (4)
C13—H13	0.9300	O11—Tb2^{iii}	2.299 (5)
C14—C15	1.372 (14)	O12—Tb1^{iv}	2.310 (4)
C14—H14	0.9300	O1W—Tb1	2.354 (5)
C15—H15	0.9300	O1W—H1W	0.818 (10)
C11'—C12	1.381 (10)	O1W—H2W	0.819 (10)
C11'—H11'	0.9300	O2W—Tb2	2.513 (5)
C13'—C14'	1.385 (12)	O2W—H3W	0.823 (10)
C13'—C12	1.395 (11)	O2W—H4W	0.822 (10)
C13'—H13'	0.9300	Tb1—O3^{v}	2.258 (4)
C14'—C15'	1.373 (13)	Tb1—O12^{vi}	2.310 (4)
C14'—H14'	0.9300	Tb1—O8^{v}	2.370 (4)
C15'—H15'	0.9300	Tb2—O6^{v}	2.258 (5)
C12—C16	1.499 (9)	Tb2—O11^{iii}	2.299 (5)
C16—O7	1.253 (7)	Tb2—O4^{v}	2.338 (5)
C16—O8	1.260 (7)	Tb2—O7^{vii}	2.352 (4)
O1—C1—O2	121.8 (6)	C20—C21—C22	120.4 (7)

O1—C1—C2	119.9 (6)	C20—C21—H21	119.8
O2—C1—C2	118.3 (6)	C22—C21—H21	119.8
C7—C2—C3	120.4 (6)	C21—C22—C23	119.4 (6)
C7—C2—C1	122.3 (6)	C21—C22—C24	121.6 (6)
C3—C2—C1	117.3 (6)	C23—C22—C24	119.0 (6)
C4—C3—C2	120.1 (6)	C22—C23—C18	120.4 (6)
C4—C3—H3	119.9	C22—C23—H23	119.8
C2—C3—H3	119.9	C18—C23—H23	119.8
C3—C4—C5	120.0 (6)	O12—C24—O11	125.5 (6)
C3—C4—C8	119.5 (6)	O12—C24—C22	117.1 (6)
C5—C4—C8	120.4 (6)	O11—C24—C22	117.4 (6)
C4—C5—C6	119.8 (6)	C1—O1—Tb1	146.1 (4)
C4—C5—H5	120.1	C1—O1—Tb2	84.1 (4)
C6—C5—H5	120.1	Tb1—O1—Tb2	122.75 (16)
C7—C6—C5	119.8 (7)	C1—O2—Tb2	105.8 (4)
C7—C6—H6	120.1	C8—O3—Tb1^{i}	141.7 (5)
C5—C6—H6	120.1	C8—O4—Tb2^{i}	141.2 (4)
C2—C7—C6	119.8 (7)	C9—O5—Tb1	137.0 (5)
C2—C7—H7	120.1	C9—O6—Tb2^{i}	158.7 (5)
C6—C7—H7	120.1	C16—O7—Tb2^{ii}	113.7 (4)
O3—C8—O4	123.9 (6)	C16—O8—Tb1^{i}	156.2 (4)
O3—C8—C4	117.2 (6)	C17—O9—Tb1	135.2 (4)
O4—C8—C4	118.9 (6)	C17—O10—Tb2	134.5 (4)
O6—C9—O5	123.6 (6)	C24—O11—Tb2^{iii}	149.2 (4)
O6—C9—C10	117.6 (6)	C24—O12—Tb1^{iv}	149.6 (4)
O5—C9—C10	118.8 (6)	Tb1—O1W—H1W	134 (5)
C11'—C10—C15	114.3 (11)	Tb1—O1W—H2W	122 (5)
C11—C10—C15	117.0 (11)	H1W—O1W—H2W	104.0 (17)
C11'—C10—C15'	121.1 (8)	Tb2—O2W—H3W	148 (5)
C11—C10—C15'	118.2 (8)	Tb2—O2W—H4W	108 (5)
C11'—C10—C9	118.7 (7)	H3W—O2W—H4W	103.3 (17)
C11—C10—C9	119.9 (7)	O3^{v}—Tb1—O5	178.18 (19)
C15—C10—C9	121.7 (10)	O3^{v}—Tb1—O12^{vi}	88.87 (17)
C15'—C10—C9	120.1 (7)	O5—Tb1—O12^{vi}	89.33 (18)
C12—C11—C10	119.5 (9)	O3^{v}—Tb1—O9	91.95 (17)
C12—C11—H11	120.3	O5—Tb1—O9	89.20 (17)
C10—C11—H11	120.3	O12^{vi}—Tb1—O9	136.12 (16)
C14—C13—C12	119.9 (15)	O3^{v}—Tb1—O1W	85.3 (2)
C14—C13—H13	120.1	O5—Tb1—O1W	93.9 (2)
C12—C13—H13	120.1	O12^{vi}—Tb1—O1W	70.16 (17)
C15—C14—C13	117.2 (18)	O9—Tb1—O1W	66.20 (17)
C15—C14—H14	121.4	O3^{v}—Tb1—O8^{v}	89.16 (16)
C13—C14—H14	121.4	O5—Tb1—O8^{v}	90.58 (16)
C14—C15—C10	124.0 (17)	O12^{vi}—Tb1—O8^{v}	74.82 (16)
C14—C15—H15	118.0	O9—Tb1—O8^{v}	149.05 (16)

C10—C15—H15	118.0	O1W—Tb1—O8^{v}	144.62 (18)
C12—C11'—C10	119.6 (8)	O3^{v}—Tb1—O1	82.62 (16)
C12—C11'—H11'	120.2	O5—Tb1—O1	99.09 (17)
C10—C11'—H11'	120.2	O12^{vi}—Tb1—O1	152.00 (15)
C14'—C13'—C12	120.2 (11)	O9—Tb1—O1	71.07 (15)
C14'—C13'—H13'	119.9	O1W—Tb1—O1	135.00 (16)
C12—C13'—H13'	119.9	O8^{v}—Tb1—O1	78.42 (15)
C15'—C14'—C13'	121.4 (12)	O6^{v}—Tb2—O11^{iii}	78.07 (17)
C15'—C14'—H14'	119.3	O6^{v}—Tb2—O4^{v}	77.30 (18)
C13'—C14'—H14'	119.3	O11^{iii}—Tb2—O4^{v}	138.85 (15)
C14'—C15'—C10	117.9 (11)	O6^{v}—Tb2—O7^{vii}	118.50 (16)
C14'—C15'—H15'	121.1	O11^{iii}—Tb2—O7^{vii}	83.35 (16)
C10—C15'—H15'	121.1	O4^{v}—Tb2—O7^{vii}	80.15 (16)
C11'—C12—C13'	119.7 (8)	O6^{v}—Tb2—O10	143.33 (16)
C11—C12—C13'	116.9 (8)	O11^{iii}—Tb2—O10	135.23 (15)
C11'—C12—C13	119.2 (10)	O4^{v}—Tb2—O10	80.73 (15)
C11—C12—C13	121.8 (10)	O7^{vii}—Tb2—O10	85.66 (15)
C11'—C12—C16	119.1 (6)	O6^{v}—Tb2—O2	89.27 (17)
C11—C12—C16	119.4 (7)	O11^{iii}—Tb2—O2	87.20 (15)
C13'—C12—C16	120.9 (7)	O4^{v}—Tb2—O2	124.72 (16)
C13—C12—C16	118.3 (9)	O7^{vii}—Tb2—O2	147.76 (15)
O7—C16—O8	122.3 (6)	O10—Tb2—O2	79.58 (15)
O7—C16—C12	118.7 (5)	O6^{v}—Tb2—O2W	145.73 (19)
O8—C16—C12	118.9 (6)	O11^{iii}—Tb2—O2W	71.18 (17)
O10—C17—O9	125.3 (6)	O4^{v}—Tb2—O2W	136.65 (17)
O10—C17—C18	118.0 (6)	O7^{vii}—Tb2—O2W	73.04 (17)
O9—C17—C18	116.7 (6)	O10—Tb2—O2W	64.10 (16)
C19—C18—C23	119.2 (6)	O2—Tb2—O2W	74.73 (17)
C19—C18—C17	121.1 (6)	O6^{v}—Tb2—O1	78.07 (14)
C23—C18—C17	119.6 (6)	O11^{iii}—Tb2—O1	128.96 (14)
C20—C19—C18	120.9 (7)	O4^{v}—Tb2—O1	76.53 (14)
C20—C19—H19	119.6	O7^{vii}—Tb2—O1	147.41 (14)
C18—C19—H19	119.6	O10—Tb2—O1	68.43 (13)
C19—C20—C21	119.7 (7)	O2—Tb2—O1	48.19 (14)
C19—C20—H20	120.2	O2W—Tb2—O1	110.02 (16)
C21—C20—H20	120.2		

Symmetry codes: (i) −*x*+3/2, *y*+1/2, −*z*+1/2; (ii) *x*, *y*+1, *z*; (iii) −*x*+2, −*y*, −*z*; (iv) *x*+1/2, −*y*+1/2, *z*−1/2; (v) −*x*+3/2, *y*−1/2, −*z*+1/2; (vi) *x*−1/2, −*y*+1/2, *z*+1/2; (vii) *x*, *y*−1, *z*.

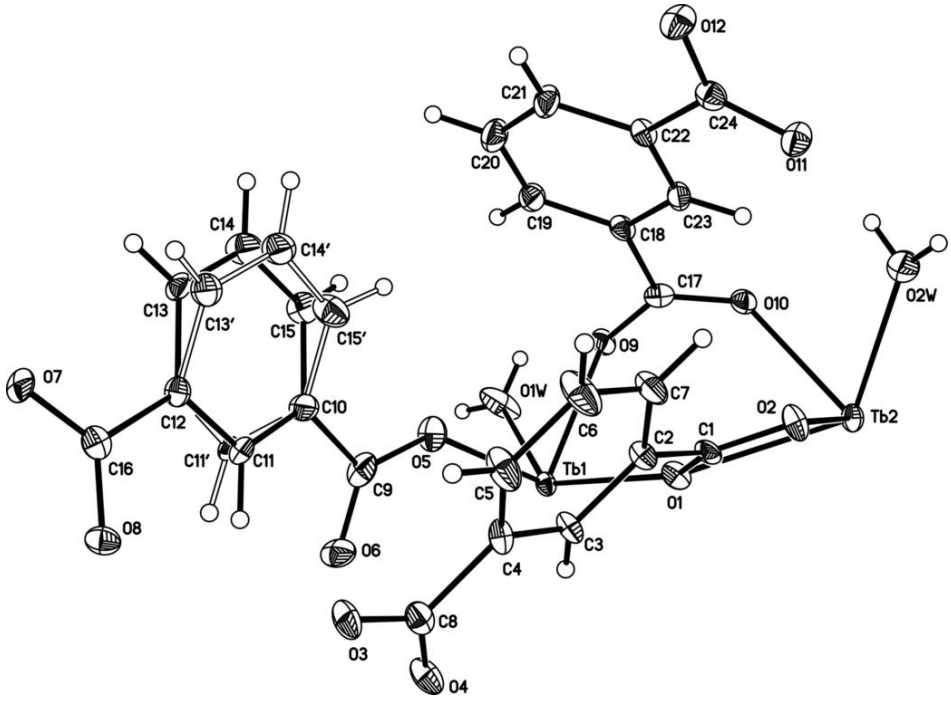
Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1W—H1W···O7^{viii}	0.818 (10)	2.04 (4)	2.789 (7)	151 (7)
O1W—H2W···O4^{ix}	0.819 (10)	2.29 (4)	3.020 (7)	149 (7)

O2W—H3W···O2^{iii}	0.823 (10)	1.959 (14)	2.780 (7)	176 (9)
C3—H3···O8^{v}	0.93	2.35	3.257 (8)	164
C11'—H11'···O1^{i}	0.93	2.39	3.284 (10)	161

Symmetry codes: (viii) −*x*+1, −*y*+1, −*z*; (ix) *x*−1/2, −*y*+1/2, *z*−1/2; (iii) −*x*+2, −*y*, −*z*; (v) −*x*+3/2, *y*−1/2, −*z*+1/2; (i) −*x*+3/2, *y*+1/2, −*z*+1/2.

Fig. 1


O12
C21
C24
C22
O11
C20
C19
C18
C23
C14
C14′
C17
O10
O2W
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C15
C15′
O9
O7
O1W
C7
C10
C6
C2
C1
O2
Tb2
C12
C16
C11′
C11
O5
C9
C5
Tb1
O1
O8
O6
C4
C3
C8
O3
O4

Fig. 2