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# Bis(μ-4-hydroxybenzoato-$\kappa^2O$:$O'$)bis[triaquabis(4-hydroxybenzoato)-$\kappa O$;$\kappa^2O$,$O'$-terbium(III)] decahydrate

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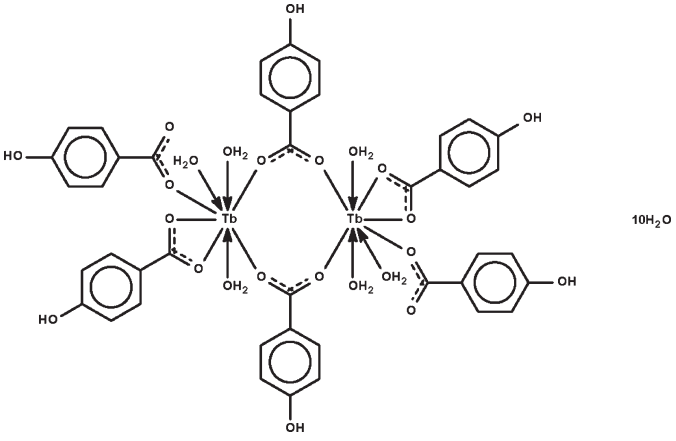
Key indicators: single-crystal X-ray study; $T$ = 173 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.018; $wR$ factor = 0.059; data-to-parameter ratio = 14.1.

The title dinuclear compound, $[Tb_2(C_7H_5O_3)_6(H_2O)_6]\cdot 10H_2O$, lies on a center of inversion and the two $Tb^{III}$ atoms are bridged by two 4-hydroxybenzoate anions; each metal atom is further coordinated by one monodentate anion and chelated by the third anion. The eight-coordinate geometry approximates a square antiprism. Hydrogen bonds of the O—H···O type connect the uncoordinated water molecules to the dinuclear species, forming a three-dimensional network.

## Related literature

For a related structure, $Tb_2(H_2O)_2(DMF)_2(C_7H_5O_3)_6$, see: Zhou *et al.* (2008).




## Experimental

### Crystal data

$[Tb_2(C_7H_5O_3)_6(H_2O)_6]\cdot 10H_2O$
$M_r$ = 1428.76
Triclinic, $P\overline{1}$
$a$ = 10.8308 (5) Å
$b$ = 11.3337 (6) Å
$c$ = 11.5128 (6) Å
$\alpha$ = 90.463 (1)°
$\beta$ = 101.690 (1)°
$\gamma$ = 105.249 (1)°
$V$ = 1332.37 (12) Å$^3$
$Z$ = 1
Mo $K\alpha$ radiation
$\mu$ = 2.73 mm$^{-1}$
$T$ = 173 K
0.47 × 0.30 × 0.19 mm

### Data collection

Bruker SMART area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.360, $T_{max}$ = 0.625
12836 measured reflections
5756 independent reflections
5391 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.018

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.018
$wR(F^2)$ = 0.059
$S$ = 1.09
5756 reflections
409 parameters
27 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.67 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.64 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O3—H3···O7$w^{i}$ | 0.84 (1) | 1.82 (2) | 2.612 (3) | 158 (3) |
| O6—H6···O6$w^{ii}$ | 0.83 (1) | 1.87 (1) | 2.678 (3) | 165 (4) |
| O9—H9···O4$w^{iii}$ | 0.84 (1) | 1.98 (2) | 2.763 (3) | 156 (3) |
| O1$w$—H11···O4$w$ | 0.84 (1) | 2.04 (1) | 2.870 (3) | 171 (3) |
| O1$w$—H12···O9$^{iv}$ | 0.83 (1) | 1.96 (1) | 2.766 (3) | 164 (3) |
| O2$w$—H22···O5$w$ | 0.83 (1) | 1.86 (1) | 2.678 (3) | 167 (3) |
| O2$w$—H21···O5$w^{v}$ | 0.84 (1) | 2.20 (2) | 2.952 (3) | 150 (3) |
| O3$w$—H31···O3$^{ii}$ | 0.84 (1) | 1.95 (1) | 2.777 (3) | 171 (3) |
| O3$w$—H32···O7$^{vi}$ | 0.84 (1) | 2.25 (2) | 2.916 (2) | 137 (3) |
| O4$w$—H41···O8$w^{vii}$ | 0.84 (1) | 1.94 (1) | 2.753 (3) | 163 (3) |
| O4$w$—H42···O5$^{viii}$ | 0.84 (1) | 2.10 (1) | 2.927 (3) | 169 (3) |
| O5$w$—H52···O7$w$ | 0.85 (1) | 2.01 (2) | 2.811 (3) | 157 (3) |
| O5$w$—H51···O8$w^{ix}$ | 0.86 (1) | 1.96 (1) | 2.791 (3) | 163 (3) |
| O6$w$—H61···O2 | 0.85 (1) | 1.90 (1) | 2.743 (2) | 172 (3) |
| O6$w$—H62···O5 | 0.84 (1) | 1.95 (1) | 2.785 (2) | 171 (3) |
| O7$w$—H71···O6$w$ | 0.84 (1) | 1.90 (1) | 2.732 (3) | 172 (3) |
| O7$w$—H72···O5$^{x}$ | 0.83 (1) | 1.89 (1) | 2.725 (3) | 175 (3) |
| O8$w$—H81···O1$^{xi}$ | 0.84 (1) | 2.10 (2) | 2.818 (3) | 144 (3) |
| O8$w$—H82···O6 | 0.84 (1) | 1.90 (1) | 2.713 (3) | 164 (3) |

Symmetry codes: (i) $-x+2, -y+1, -z$; (ii) $x-1, y, z$; (iii) $x+1, y+1, z$; (iv) $-x+2, -y+1, -z+1$; (v) $-x+1, -y+1, -z$; (vi) $-x+1, -y+1, -z+1$; (vii) $-x, -y, -z+1$; (viii) $-x+1, -y, -z+1$; (ix) $-x, -y, -z$; (x) $-x+1, -y, -z$; (xi) $x-1, y-1, z$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2706).

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Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
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Westrip, S. P. (2009). *publCIF*. In preparation.
Zhou, Y.-X., Shen, X.-Q., Zhang, H.-Y., Du, C.-X. & Hou, H.-W. (2008). *J. Coord. Chem.* **61**, 3981–3992.

# supporting information

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# Bis(μ-4-hydroxybenzoato-$\kappa^2O$:$O'$)bis[triaquabis(4-hydroxybenzoato)-$\kappa O$;$\kappa^2O$,$O'$-terbium(III)] decahydrate

**Yi-Min Zhu, Pei-Pei Feng, Yang-Yi Yang and Seik Weng Ng**

## S1. Experimental

4-Hydroxybenzoic acid (0.3 mmol) and sodium hydroxide (0.3 mmol) were dissolved in water (5 ml) and this was mixed with a solution of terbium chloride (0.1 mmol) dissolved in water (5 ml). The white precipitate that formed was removed by filtration. Colorless crystals were isolated from the filtrated after two weeks.

## S2. Refinement

Carbon-bound hydrogen atoms were generated geometrically and were constrained to ride on their parent atoms [C–H = 0.95, $U_{iso}$(H) = 1.2$U_{eq}$(C)]. The hydroxy and water H-atoms were located in a difference Fourier map, and were refined with distance restraints of O–H 0.84±0.01 Å and H···H 1.37±0.01 Å. Their temperature factors were tied by a factor of 1.5.

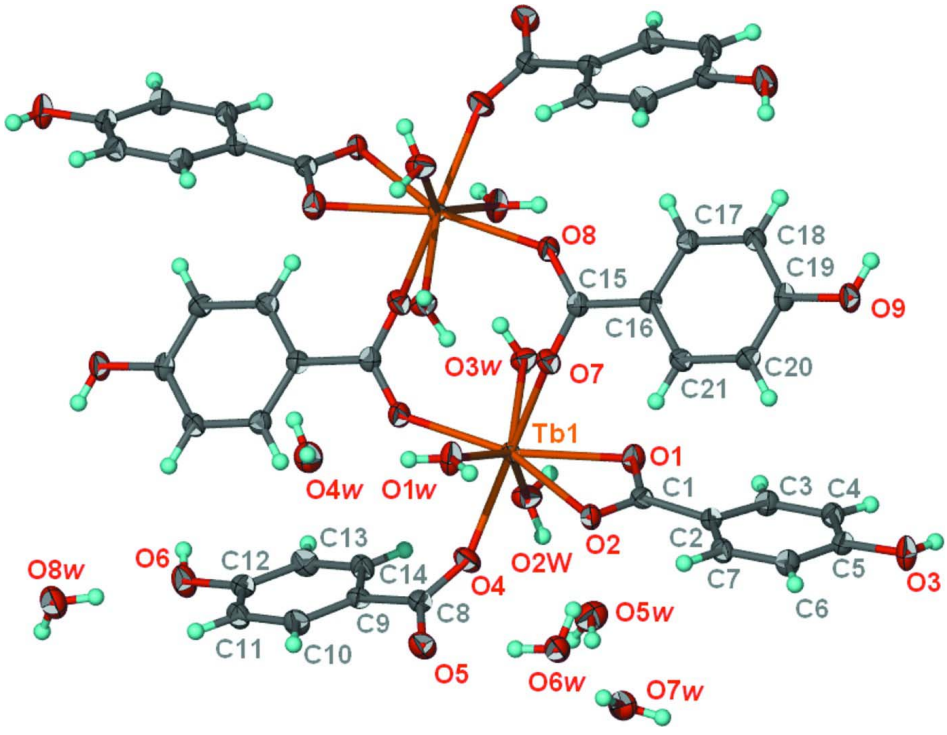


**Figure 1**

Thermal ellipsoid plot (Barbour, 2001) of $Tb_2(H_2O)_6(C_7H_5O_3)_6{\cdot}10H_2O$ at the 70% probability level; hydrogen atoms are drawn as sphere of arbitrary radius.

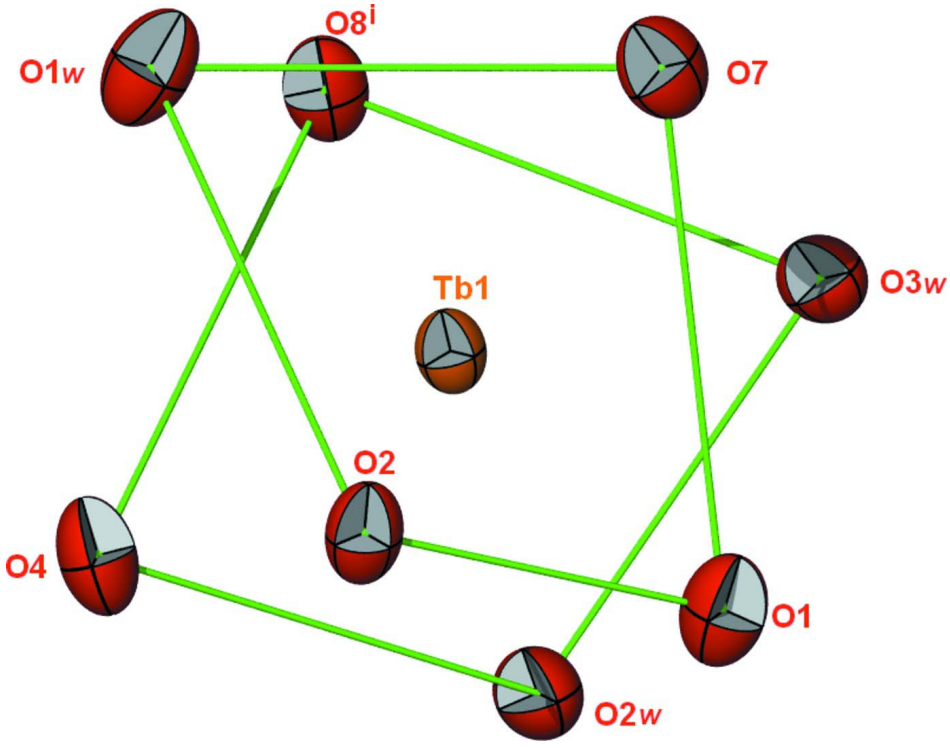


**Figure 2**

Detail of the coordination environment of the terbium(III) atom.

**Bis(*μ*-4-hydroxybenzoato-$\kappa^2O$:$O'$)bis[triaquabis(4- hydroxybenzoato)-$\kappa O$;$\kappa^2O,O'$-terbium(III)] decahydrate**

*Crystal data*

$[Tb_2(C_7H_5O_3)_6(H_2O)_6]\cdot 10H_2O$
$M_r = 1428.76$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 10.8308\ (5)$ Å
$b = 11.3337\ (6)$ Å
$c = 11.5128\ (6)$ Å
$\alpha = 90.463\ (1)°$
$\beta = 101.690\ (1)°$
$\gamma = 105.249\ (1)°$
$V = 1332.37\ (12)$ Å$^3$
$Z = 1$
$F(000) = 716$
$D_x = 1.781$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5392 reflections
$\theta = 2.3–27.0°$
$\mu = 2.73$ mm$^{-1}$
$T = 173$ K
Block, colorless
0.47 × 0.30 × 0.19 mm

*Data collection*

Bruker SMART area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.360$, $T_{max} = 0.625$
12836 measured reflections
5756 independent reflections
5391 reflections with $I > 2\sigma(I)$
$R_{int} = 0.018$
$\theta_{max} = 27.0°$, $\theta_{min} = 1.8°$
$h = -13\rightarrow13$
$k = -13\rightarrow14$
$l = -14\rightarrow14$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.018$
$wR(F^2) = 0.059$
$S = 1.09$
5756 reflections
409 parameters
27 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
$w = 1/[\sigma^2(F_o^2) + (0.0407P)^2 + 0.0925P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.67$ e Å$^{-3}$
$\Delta\rho_{min} = -0.64$ e Å$^{-3}$

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Tb1 | 0.521099 (9) | 0.392335 (9) | 0.316629 (8) | 0.01393 (5) |
| O1 | 0.68247 (16) | 0.52500 (16) | 0.22113 (15) | 0.0219 (4) |
| O2 | 0.71938 (16) | 0.35118 (15) | 0.28025 (15) | 0.0189 (3) |
| O3 | 1.25530 (17) | 0.60503 (18) | 0.13235 (17) | 0.0243 (4) |
| H3 | 1.262 (3) | 0.6687 (19) | 0.095 (3) | 0.036* |
| O4 | 0.44616 (17) | 0.20553 (16) | 0.21445 (16) | 0.0233 (4) |
| O5 | 0.44536 (17) | 0.00991 (16) | 0.21119 (16) | 0.0236 (4) |
| O6 | −0.15974 (18) | −0.03603 (17) | 0.16884 (18) | 0.0280 (4) |
| H6 | −0.192 (3) | 0.023 (2) | 0.171 (3) | 0.042* |
| O7 | 0.66267 (16) | 0.51402 (16) | 0.47709 (14) | 0.0207 (3) |
| O8 | 0.65933 (16) | 0.66801 (16) | 0.59555 (15) | 0.0209 (4) |
| O9 | 1.23480 (17) | 0.85877 (17) | 0.50180 (16) | 0.0220 (4) |
| H9 | 1.262 (3) | 0.9320 (13) | 0.529 (3) | 0.033* |
| O1w | 0.56576 (17) | 0.25542 (17) | 0.47360 (16) | 0.0241 (4) |
| H11 | 0.509 (2) | 0.206 (2) | 0.502 (3) | 0.036* |
| H12 | 0.6353 (16) | 0.234 (3) | 0.487 (3) | 0.036* |
| O2w | 0.40382 (18) | 0.41807 (17) | 0.12181 (15) | 0.0233 (4) |
| H21 | 0.405 (3) | 0.4895 (13) | 0.102 (2) | 0.035* |
| H22 | 0.426 (3) | 0.380 (2) | 0.071 (2) | 0.035* |
| O3w | 0.44922 (17) | 0.57723 (16) | 0.32029 (15) | 0.0201 (3) |
| H31 | 0.397 (2) | 0.593 (3) | 0.2625 (14) | 0.030* |
| H32 | 0.428 (3) | 0.593 (3) | 0.3838 (13) | 0.030* |
| O4w | 0.39635 (19) | 0.08545 (18) | 0.59456 (16) | 0.0267 (4) |
| H41 | 0.358 (3) | 0.130 (3) | 0.624 (2) | 0.040* |
| H42 | 0.450 (3) | 0.066 (3) | 0.6490 (19) | 0.040* |
| O5w | 0.4872 (2) | 0.3305 (2) | −0.05591 (17) | 0.0309 (4) |
| H51 | 0.421 (2) | 0.294 (3) | −0.111 (2) | 0.046* |
| H52 | 0.530 (3) | 0.278 (2) | −0.037 (3) | 0.046* |
| O6w | 0.69870 (17) | 0.12258 (17) | 0.18465 (16) | 0.0235 (4) |
| H61 | 0.711 (3) | 0.1957 (12) | 0.211 (3) | 0.035* |
| H62 | 0.6246 (17) | 0.082 (2) | 0.194 (3) | 0.035* |
| O7w | 0.6606 (2) | 0.18314 (17) | −0.04660 (16) | 0.0286 (4) |
| H71 | 0.676 (3) | 0.160 (3) | 0.0230 (11) | 0.043* |

| | | | | |
|---|---|---|---|---|
| H72 | 0.627 (3) | 0.1215 (18) | −0.0936 (19) | 0.043* |
| O8w | −0.30680 (19) | −0.22701 (18) | 0.26414 (18) | 0.0301 (4) |
| H81 | −0.280 (3) | −0.2897 (17) | 0.276 (3) | 0.045* |
| H82 | −0.249 (2) | −0.172 (2) | 0.242 (3) | 0.045* |
| C1 | 0.7539 (2) | 0.4523 (2) | 0.2315 (2) | 0.0169 (5) |
| C2 | 0.8802 (2) | 0.4855 (2) | 0.19285 (19) | 0.0163 (4) |
| C3 | 0.9139 (2) | 0.5908 (2) | 0.1316 (2) | 0.0190 (5) |
| H3A | 0.8513 | 0.6352 | 0.1057 | 0.023* |
| C4 | 1.0383 (2) | 0.6307 (2) | 0.1084 (2) | 0.0186 (5) |
| H4 | 1.0609 | 0.7020 | 0.0660 | 0.022* |
| C5 | 1.1296 (2) | 0.5662 (2) | 0.1474 (2) | 0.0177 (5) |
| C6 | 1.0956 (2) | 0.4574 (2) | 0.2042 (2) | 0.0188 (5) |
| H6A | 1.1572 | 0.4112 | 0.2268 | 0.023* |
| C7 | 0.9713 (2) | 0.4181 (2) | 0.22698 (19) | 0.0178 (5) |
| H7 | 0.9475 | 0.3446 | 0.2661 | 0.021* |
| C8 | 0.3871 (2) | 0.0935 (2) | 0.2068 (2) | 0.0184 (5) |
| C9 | 0.2416 (2) | 0.0589 (2) | 0.1919 (2) | 0.0168 (4) |
| C10 | 0.1763 (2) | −0.0536 (2) | 0.2258 (2) | 0.0206 (5) |
| H10 | 0.2237 | −0.1109 | 0.2542 | 0.025* |
| C11 | 0.0414 (2) | −0.0831 (2) | 0.2184 (2) | 0.0224 (5) |
| H11A | −0.0027 | −0.1592 | 0.2440 | 0.027* |
| C12 | −0.0276 (2) | −0.0015 (2) | 0.1740 (2) | 0.0201 (5) |
| C13 | 0.0352 (2) | 0.1103 (2) | 0.1364 (2) | 0.0214 (5) |
| H13 | −0.0135 | 0.1651 | 0.1034 | 0.026* |
| C14 | 0.1704 (2) | 0.1410 (2) | 0.1477 (2) | 0.0207 (5) |
| H14 | 0.2146 | 0.2185 | 0.1251 | 0.025* |
| C15 | 0.7152 (2) | 0.6180 (2) | 0.53115 (19) | 0.0159 (4) |
| C16 | 0.8513 (2) | 0.6851 (2) | 0.52099 (19) | 0.0153 (4) |
| C17 | 0.9058 (2) | 0.8072 (2) | 0.56448 (19) | 0.0167 (4) |
| H17 | 0.8550 | 0.8494 | 0.5982 | 0.020* |
| C18 | 1.0337 (2) | 0.8673 (2) | 0.5587 (2) | 0.0187 (5) |
| H18 | 1.0704 | 0.9503 | 0.5883 | 0.022* |
| C19 | 1.1080 (2) | 0.8052 (2) | 0.5093 (2) | 0.0177 (5) |
| C20 | 1.0543 (2) | 0.6841 (2) | 0.4635 (2) | 0.0187 (5) |
| H20 | 1.1045 | 0.6427 | 0.4282 | 0.022* |
| C21 | 0.9260 (2) | 0.6245 (2) | 0.4702 (2) | 0.0176 (5) |
| H21A | 0.8890 | 0.5418 | 0.4398 | 0.021* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Tb1 | 0.01190 (7) | 0.01180 (7) | 0.01807 (7) | 0.00220 (4) | 0.00446 (4) | 0.00005 (4) |
| O1 | 0.0188 (8) | 0.0190 (9) | 0.0318 (9) | 0.0082 (7) | 0.0104 (7) | 0.0067 (7) |
| O2 | 0.0174 (8) | 0.0138 (8) | 0.0276 (8) | 0.0042 (6) | 0.0098 (6) | 0.0035 (6) |
| O3 | 0.0152 (8) | 0.0248 (10) | 0.0348 (10) | 0.0051 (7) | 0.0099 (7) | 0.0120 (8) |
| O4 | 0.0198 (8) | 0.0152 (9) | 0.0338 (9) | 0.0032 (7) | 0.0051 (7) | −0.0029 (7) |
| O5 | 0.0186 (8) | 0.0183 (9) | 0.0343 (9) | 0.0062 (7) | 0.0049 (7) | −0.0008 (7) |
| O6 | 0.0173 (9) | 0.0201 (10) | 0.0484 (11) | 0.0055 (7) | 0.0105 (8) | 0.0042 (8) |

| | | | | | | |
|---|---|---|---|---|---|---|
| O7 | 0.0160 (8) | 0.0188 (9) | 0.0244 (8) | 0.0005 (7) | 0.0032 (6) | −0.0025 (7) |
| O8 | 0.0174 (8) | 0.0170 (9) | 0.0302 (9) | 0.0039 (7) | 0.0105 (7) | −0.0001 (7) |
| O9 | 0.0134 (8) | 0.0204 (9) | 0.0321 (9) | 0.0023 (7) | 0.0079 (7) | 0.0027 (7) |
| O1w | 0.0184 (8) | 0.0241 (10) | 0.0337 (9) | 0.0082 (7) | 0.0108 (7) | 0.0114 (7) |
| O2w | 0.0316 (10) | 0.0196 (9) | 0.0203 (8) | 0.0097 (8) | 0.0058 (7) | 0.0005 (7) |
| O3w | 0.0237 (9) | 0.0198 (9) | 0.0195 (8) | 0.0093 (7) | 0.0065 (7) | 0.0013 (7) |
| O4w | 0.0262 (10) | 0.0251 (10) | 0.0301 (9) | 0.0082 (8) | 0.0067 (7) | 0.0042 (8) |
| O5w | 0.0298 (10) | 0.0359 (12) | 0.0292 (10) | 0.0122 (9) | 0.0072 (8) | −0.0007 (8) |
| O6w | 0.0200 (9) | 0.0194 (9) | 0.0330 (10) | 0.0057 (7) | 0.0092 (7) | 0.0001 (7) |
| O7w | 0.0404 (11) | 0.0183 (9) | 0.0272 (9) | 0.0055 (8) | 0.0109 (8) | 0.0021 (7) |
| O8w | 0.0293 (10) | 0.0204 (10) | 0.0427 (11) | 0.0062 (8) | 0.0128 (8) | 0.0068 (8) |
| C1 | 0.0153 (11) | 0.0168 (11) | 0.0172 (10) | 0.0023 (9) | 0.0030 (8) | −0.0022 (9) |
| C2 | 0.0156 (10) | 0.0159 (11) | 0.0170 (10) | 0.0029 (9) | 0.0047 (8) | 0.0000 (8) |
| C3 | 0.0185 (11) | 0.0181 (12) | 0.0224 (11) | 0.0074 (9) | 0.0057 (9) | 0.0039 (9) |
| C4 | 0.0207 (11) | 0.0155 (11) | 0.0206 (11) | 0.0047 (9) | 0.0066 (9) | 0.0040 (9) |
| C5 | 0.0162 (11) | 0.0183 (12) | 0.0185 (11) | 0.0028 (9) | 0.0057 (8) | −0.0004 (9) |
| C6 | 0.0180 (11) | 0.0180 (12) | 0.0222 (11) | 0.0066 (9) | 0.0055 (9) | 0.0046 (9) |
| C7 | 0.0190 (11) | 0.0155 (11) | 0.0182 (10) | 0.0028 (9) | 0.0046 (8) | 0.0029 (9) |
| C8 | 0.0181 (11) | 0.0172 (12) | 0.0189 (10) | 0.0026 (9) | 0.0044 (8) | −0.0012 (9) |
| C9 | 0.0185 (11) | 0.0136 (11) | 0.0185 (10) | 0.0046 (9) | 0.0039 (8) | −0.0004 (8) |
| C10 | 0.0203 (11) | 0.0158 (12) | 0.0276 (12) | 0.0078 (9) | 0.0054 (9) | 0.0029 (9) |
| C11 | 0.0222 (12) | 0.0147 (12) | 0.0318 (13) | 0.0040 (10) | 0.0102 (10) | 0.0027 (10) |
| C12 | 0.0186 (11) | 0.0184 (12) | 0.0230 (11) | 0.0036 (9) | 0.0055 (9) | −0.0021 (9) |
| C13 | 0.0215 (12) | 0.0185 (12) | 0.0247 (12) | 0.0082 (10) | 0.0023 (9) | 0.0034 (9) |
| C14 | 0.0216 (12) | 0.0156 (12) | 0.0245 (11) | 0.0032 (10) | 0.0064 (9) | 0.0022 (9) |
| C15 | 0.0141 (10) | 0.0166 (11) | 0.0176 (10) | 0.0052 (9) | 0.0031 (8) | 0.0036 (8) |
| C16 | 0.0147 (10) | 0.0166 (11) | 0.0142 (10) | 0.0043 (9) | 0.0021 (8) | 0.0016 (8) |
| C17 | 0.0163 (11) | 0.0163 (11) | 0.0189 (10) | 0.0055 (9) | 0.0051 (8) | 0.0024 (9) |
| C18 | 0.0188 (11) | 0.0148 (11) | 0.0221 (11) | 0.0030 (9) | 0.0053 (9) | 0.0008 (9) |
| C19 | 0.0121 (10) | 0.0209 (12) | 0.0200 (11) | 0.0036 (9) | 0.0040 (8) | 0.0051 (9) |
| C20 | 0.0175 (11) | 0.0190 (12) | 0.0217 (11) | 0.0070 (9) | 0.0064 (9) | 0.0001 (9) |
| C21 | 0.0163 (11) | 0.0161 (11) | 0.0189 (10) | 0.0026 (9) | 0.0028 (8) | −0.0008 (9) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Tb1—O4 | 2.2791 (17) | O8w—H81 | 0.836 (10) |
| Tb1—O7 | 2.3162 (16) | O8w—H82 | 0.840 (10) |
| Tb1—O8$^{i}$ | 2.3276 (16) | C1—C2 | 1.480 (3) |
| Tb1—O2w | 2.4042 (17) | C2—C3 | 1.394 (3) |
| Tb1—O3w | 2.4227 (18) | C2—C7 | 1.398 (3) |
| Tb1—O2 | 2.4276 (16) | C3—C4 | 1.385 (3) |
| Tb1—O1 | 2.4388 (17) | C3—H3A | 0.9500 |
| Tb1—O1w | 2.4482 (17) | C4—C5 | 1.386 (3) |
| O1—C1 | 1.262 (3) | C4—H4 | 0.9500 |
| O2—C1 | 1.280 (3) | C5—C6 | 1.398 (3) |
| O3—C5 | 1.364 (3) | C6—C7 | 1.383 (3) |
| O3—H3 | 0.837 (10) | C6—H6A | 0.9500 |
| O4—C8 | 1.256 (3) | C7—H7 | 0.9500 |

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|---|---|---|---|
| O5—C8 | 1.265 (3) | C8—C9 | 1.494 (3) |
| O6—C12 | 1.370 (3) | C9—C10 | 1.387 (3) |
| O6—H6 | 0.833 (10) | C9—C14 | 1.395 (4) |
| O7—C15 | 1.262 (3) | C10—C11 | 1.395 (3) |
| O8—C15 | 1.262 (3) | C10—H10 | 0.9500 |
| O8—Tb1$^{i}$ | 2.3276 (16) | C11—C12 | 1.375 (4) |
| O9—C19 | 1.368 (3) | C11—H11A | 0.9500 |
| O9—H9 | 0.841 (10) | C12—C13 | 1.389 (4) |
| O1w—H11 | 0.839 (10) | C13—C14 | 1.392 (3) |
| O1w—H12 | 0.834 (10) | C13—H13 | 0.9500 |
| O2w—H21 | 0.838 (10) | C14—H14 | 0.9500 |
| O2w—H22 | 0.832 (10) | C15—C16 | 1.499 (3) |
| O3w—H31 | 0.835 (10) | C16—C21 | 1.395 (3) |
| O3w—H32 | 0.840 (10) | C16—C17 | 1.397 (3) |
| O4w—H41 | 0.837 (10) | C17—C18 | 1.389 (3) |
| O4w—H42 | 0.840 (10) | C17—H17 | 0.9500 |
| O5w—H51 | 0.861 (10) | C18—C19 | 1.394 (3) |
| O5w—H52 | 0.852 (10) | C18—H18 | 0.9500 |
| O6w—H61 | 0.846 (10) | C19—C20 | 1.394 (3) |
| O6w—H62 | 0.841 (10) | C20—C21 | 1.394 (3) |
| O7w—H71 | 0.842 (10) | C20—H20 | 0.9500 |
| O7w—H72 | 0.833 (10) | C21—H21A | 0.9500 |

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|---|---|---|---|
| O4—Tb1—O7 | 147.98 (6) | C4—C3—H3A | 119.9 |
| O4—Tb1—O8$^{i}$ | 86.52 (6) | C2—C3—H3A | 119.9 |
| O7—Tb1—O8$^{i}$ | 97.15 (6) | C3—C4—C5 | 119.7 (2) |
| O4—Tb1—O2w | 71.93 (6) | C3—C4—H4 | 120.2 |
| O7—Tb1—O2w | 138.23 (6) | C5—C4—H4 | 120.2 |
| O8$^{i}$—Tb1—O2w | 97.25 (6) | O3—C5—C4 | 122.0 (2) |
| O4—Tb1—O3w | 136.40 (6) | O3—C5—C6 | 117.2 (2) |
| O7—Tb1—O3w | 74.94 (6) | C4—C5—C6 | 120.8 (2) |
| O8$^{i}$—Tb1—O3w | 77.43 (6) | C7—C6—C5 | 119.1 (2) |
| O2w—Tb1—O3w | 70.33 (6) | C7—C6—H6A | 120.4 |
| O4—Tb1—O2 | 76.79 (6) | C5—C6—H6A | 120.4 |
| O7—Tb1—O2 | 83.64 (6) | C6—C7—C2 | 120.5 (2) |
| O8$^{i}$—Tb1—O2 | 147.37 (6) | C6—C7—H7 | 119.8 |
| O2w—Tb1—O2 | 103.72 (6) | C2—C7—H7 | 119.8 |
| O3w—Tb1—O2 | 133.16 (6) | O4—C8—O5 | 122.9 (2) |
| O4—Tb1—O1 | 108.71 (6) | O4—C8—C9 | 117.9 (2) |
| O7—Tb1—O1 | 78.16 (6) | O5—C8—C9 | 119.2 (2) |
| O8$^{i}$—Tb1—O1 | 158.46 (6) | C10—C9—C14 | 119.2 (2) |
| O2w—Tb1—O1 | 74.23 (6) | C10—C9—C8 | 120.3 (2) |
| O3w—Tb1—O1 | 81.06 (6) | C14—C9—C8 | 120.4 (2) |
| O2—Tb1—O1 | 53.69 (6) | C9—C10—C11 | 120.4 (2) |
| O4—Tb1—O1w | 78.28 (6) | C9—C10—H10 | 119.8 |
| O7—Tb1—O1w | 73.07 (6) | C11—C10—H10 | 119.8 |
| O8$^{i}$—Tb1—O1w | 70.82 (6) | C12—C11—C10 | 119.7 (2) |
| O2w—Tb1—O1w | 148.59 (6) | C12—C11—H11A | 120.2 |

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| O3w—Tb1—O1w | 130.93 (6) | C10—C11—H11A | 120.2 |
| O2—Tb1—O1w | 78.37 (6) | O6—C12—C11 | 116.8 (2) |
| O1—Tb1—O1w | 126.13 (6) | O6—C12—C13 | 122.2 (2) |
| C1—O1—Tb1 | 93.28 (14) | C11—C12—C13 | 121.0 (2) |
| C1—O2—Tb1 | 93.32 (14) | C14—C13—C12 | 119.1 (2) |
| C5—O3—H3 | 107 (2) | C14—C13—H13 | 120.5 |
| C8—O4—Tb1 | 150.98 (16) | C12—C13—H13 | 120.5 |
| C12—O6—H6 | 114 (2) | C13—C14—C9 | 120.6 (2) |
| C15—O7—Tb1 | 150.17 (16) | C13—C14—H14 | 119.7 |
| C15—O8—Tb1$^{i}$ | 136.88 (15) | C9—C14—H14 | 119.7 |
| C19—O9—H9 | 113 (2) | O8—C15—O7 | 123.1 (2) |
| Tb1—O1w—H11 | 125.7 (19) | O8—C15—C16 | 117.9 (2) |
| Tb1—O1w—H12 | 121.8 (19) | O7—C15—C16 | 119.0 (2) |
| H11—O1w—H12 | 109.2 (16) | C21—C16—C17 | 119.4 (2) |
| Tb1—O2w—H21 | 118 (2) | C21—C16—C15 | 119.8 (2) |
| Tb1—O2w—H22 | 110 (2) | C17—C16—C15 | 120.8 (2) |
| H21—O2w—H22 | 110.7 (16) | C18—C17—C16 | 120.3 (2) |
| Tb1—O3w—H31 | 121 (2) | C18—C17—H17 | 119.8 |
| Tb1—O3w—H32 | 114.7 (19) | C16—C17—H17 | 119.8 |
| H31—O3w—H32 | 109.5 (16) | C17—C18—C19 | 119.7 (2) |
| H41—O4w—H42 | 108.8 (16) | C17—C18—H18 | 120.1 |
| H51—O5w—H52 | 105.4 (15) | C19—C18—H18 | 120.1 |
| H61—O6w—H62 | 108.3 (16) | O9—C19—C18 | 122.6 (2) |
| H71—O7w—H72 | 108.9 (16) | O9—C19—C20 | 116.8 (2) |
| H81—O8w—H82 | 109.5 (16) | C18—C19—C20 | 120.6 (2) |
| O1—C1—O2 | 119.7 (2) | C21—C20—C19 | 119.2 (2) |
| O1—C1—C2 | 120.5 (2) | C21—C20—H20 | 120.4 |
| O2—C1—C2 | 119.7 (2) | C19—C20—H20 | 120.4 |
| C3—C2—C7 | 119.6 (2) | C20—C21—C16 | 120.7 (2) |
| C3—C2—C1 | 120.0 (2) | C20—C21—H21A | 119.7 |
| C7—C2—C1 | 120.2 (2) | C16—C21—H21A | 119.7 |
| C4—C3—C2 | 120.2 (2) | | |

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| O4—Tb1—O1—C1 | −56.96 (14) | C3—C4—C5—C6 | 3.5 (3) |
| O7—Tb1—O1—C1 | 90.56 (14) | O3—C5—C6—C7 | 176.6 (2) |
| O8$^{i}$—Tb1—O1—C1 | 169.90 (15) | C4—C5—C6—C7 | −3.4 (3) |
| O2w—Tb1—O1—C1 | −121.11 (14) | C5—C6—C7—C2 | 0.5 (3) |
| O3w—Tb1—O1—C1 | 166.92 (14) | C3—C2—C7—C6 | 2.3 (3) |
| O2—Tb1—O1—C1 | −0.22 (12) | C1—C2—C7—C6 | −171.8 (2) |
| O1w—Tb1—O1—C1 | 31.99 (16) | Tb1—O4—C8—O5 | −108.1 (3) |
| O4—Tb1—O2—C1 | 125.77 (14) | Tb1—O4—C8—C9 | 72.0 (4) |
| O7—Tb1—O2—C1 | −79.74 (13) | O4—C8—C9—C10 | −156.2 (2) |
| O8$^{i}$—Tb1—O2—C1 | −173.07 (12) | O5—C8—C9—C10 | 23.8 (3) |
| O2w—Tb1—O2—C1 | 58.44 (14) | O4—C8—C9—C14 | 21.5 (3) |
| O3w—Tb1—O2—C1 | −17.32 (16) | O5—C8—C9—C14 | −158.5 (2) |
| O1—Tb1—O2—C1 | 0.22 (12) | C14—C9—C10—C11 | −1.5 (4) |
| O1w—Tb1—O2—C1 | −153.71 (14) | C8—C9—C10—C11 | 176.2 (2) |
| O7—Tb1—O4—C8 | 66.9 (4) | C9—C10—C11—C12 | 1.9 (4) |

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| O8$^{i}$—Tb1—O4—C8 | −31.0 (3) | C10—C11—C12—O6 | 179.8 (2) |
| O2w—Tb1—O4—C8 | −129.8 (3) | C10—C11—C12—C13 | −0.1 (4) |
| O3w—Tb1—O4—C8 | −98.7 (3) | O6—C12—C13—C14 | 178.0 (2) |
| O2—Tb1—O4—C8 | 120.8 (3) | C11—C12—C13—C14 | −2.1 (4) |
| O1—Tb1—O4—C8 | 164.6 (3) | C12—C13—C14—C9 | 2.4 (4) |
| O1w—Tb1—O4—C8 | 40.1 (3) | C10—C9—C14—C13 | −0.7 (4) |
| O4—Tb1—O7—C15 | 169.6 (3) | C8—C9—C14—C13 | −178.4 (2) |
| O8$^{i}$—Tb1—O7—C15 | −95.6 (3) | Tb1$^{i}$—O8—C15—O7 | 14.8 (4) |
| O2w—Tb1—O7—C15 | 13.8 (3) | Tb1$^{i}$—O8—C15—C16 | −163.99 (16) |
| O3w—Tb1—O7—C15 | −20.7 (3) | Tb1—O7—C15—O8 | 81.1 (4) |
| O2—Tb1—O7—C15 | 117.3 (3) | Tb1—O7—C15—C16 | −100.2 (3) |
| O1—Tb1—O7—C15 | 63.1 (3) | O8—C15—C16—C21 | 167.8 (2) |
| O1w—Tb1—O7—C15 | −163.0 (3) | O7—C15—C16—C21 | −11.1 (3) |
| Tb1—O1—C1—O2 | 0.4 (2) | O8—C15—C16—C17 | −10.6 (3) |
| Tb1—O1—C1—C2 | −176.78 (18) | O7—C15—C16—C17 | 170.5 (2) |
| Tb1—O2—C1—O1 | −0.4 (2) | C21—C16—C17—C18 | −0.9 (3) |
| Tb1—O2—C1—C2 | 176.80 (18) | C15—C16—C17—C18 | 177.6 (2) |
| O1—C1—C2—C3 | −7.7 (3) | C16—C17—C18—C19 | 0.0 (3) |
| O2—C1—C2—C3 | 175.1 (2) | C17—C18—C19—O9 | −179.5 (2) |
| O1—C1—C2—C7 | 166.3 (2) | C17—C18—C19—C20 | 1.2 (4) |
| O2—C1—C2—C7 | −10.8 (3) | O9—C19—C20—C21 | 179.2 (2) |
| C7—C2—C3—C4 | −2.3 (3) | C18—C19—C20—C21 | −1.5 (4) |
| C1—C2—C3—C4 | 171.8 (2) | C19—C20—C21—C16 | 0.5 (3) |
| C2—C3—C4—C5 | −0.6 (4) | C17—C16—C21—C20 | 0.6 (3) |
| C3—C4—C5—O3 | −176.5 (2) | C15—C16—C21—C20 | −177.8 (2) |

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+1.

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O3—H3···O7*w*$^{ii}$ | 0.84 (1) | 1.82 (2) | 2.612 (3) | 158 (3) |
| O6—H6···O6*w*$^{iii}$ | 0.83 (1) | 1.87 (1) | 2.678 (3) | 165 (4) |
| O9—H9···O4*w*$^{iv}$ | 0.84 (1) | 1.98 (2) | 2.763 (3) | 156 (3) |
| O1*w*—H11···O4*w* | 0.84 (1) | 2.04 (1) | 2.870 (3) | 171 (3) |
| O1*w*—H12···O9$^{v}$ | 0.83 (1) | 1.96 (1) | 2.766 (3) | 164 (3) |
| O2*w*—H22···O5*w* | 0.83 (1) | 1.86 (1) | 2.678 (3) | 167 (3) |
| O2*w*—H21···O5*w*$^{vi}$ | 0.84 (1) | 2.20 (2) | 2.952 (3) | 150 (3) |
| O3*w*—H31···O3$^{iii}$ | 0.84 (1) | 1.95 (1) | 2.777 (3) | 171 (3) |
| O3*w*—H32···O7$^{i}$ | 0.84 (1) | 2.25 (2) | 2.916 (2) | 137 (3) |
| O4*w*—H41···O8*w*$^{vii}$ | 0.84 (1) | 1.94 (1) | 2.753 (3) | 163 (3) |
| O4*w*—H42···O5$^{viii}$ | 0.84 (1) | 2.10 (1) | 2.927 (3) | 169 (3) |
| O5*w*—H52···O7*w* | 0.85 (1) | 2.01 (2) | 2.811 (3) | 157 (3) |
| O5*w*—H51···O8*w*$^{ix}$ | 0.86 (1) | 1.96 (1) | 2.791 (3) | 163 (3) |
| O6*w*—H61···O2 | 0.85 (1) | 1.90 (1) | 2.743 (2) | 172 (3) |
| O6*w*—H62···O5 | 0.84 (1) | 1.95 (1) | 2.785 (2) | 171 (3) |
| O7*w*—H71···O6*w* | 0.84 (1) | 1.90 (1) | 2.732 (3) | 172 (3) |
| O7*w*—H72···O5$^{x}$ | 0.83 (1) | 1.89 (1) | 2.725 (3) | 175 (3) |

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|---|---|---|---|---|
| O8*w*—H81···O1$^{xi}$ | 0.84 (1) | 2.10 (2) | 2.818 (3) | 144 (3) |
| O8*w*—H82···O6 | 0.84 (1) | 1.90 (1) | 2.713 (3) | 164 (3) |

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