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ISSN 1600-5368

## Crystal structure of poly[ $\mu_{6}$-adipato-di-aquadi- $\mu_{2}$-oxalato-didysprosium(III)]

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Received 5 November 2014; accepted 8 November 2014

Edited by M. Gdaniec, Adam Mickiewicz University, Poland

In the title coordination polymer, $\left[\mathrm{Dy}_{2}\left(\mathrm{C}_{6} \mathrm{H}_{8} \mathrm{O}_{4}\right)\left(\mathrm{C}_{2} \mathrm{O}_{4}\right)_{2}\right.$ $\left.\left(\mathrm{H}_{2} \mathrm{O}\right)_{2}\right]_{n}$, the asymmetric unit consists of one $\mathrm{Dy}^{3+}$ cation, one half of an adipate anion, two halves of oxalate anions and one coordinating water molecule. The adipate and oxalate ions are located on centres of inversion. The Dy ${ }^{3+}$ cation has a distorted tricapped trigonal-prismatic geometry and is coordinated by nine O atoms, four belonging to three adipate anions, four to two oxalate anions and one from an aqua ligand. The cations are bridged by adipate ligands, generating a two-dimensional network parallel to (010). This network is further extended into three dimensions by coordination of the rigid oxalate ligands and is further consolidated by $\mathrm{O}-\mathrm{H} \cdots \mathrm{O}$ hydrogen bonds. A part of the adipate anion is disordered over two positions in a 0.75:0.25 ratio.

Keywords: crystal structure; dysprosium(III) complex; three-dimensional coordination polymer; oxalate; adipate.

CCDC reference: 1033325

## 1. Related literature

For the isotypic structures of $\mathrm{La}, \mathrm{Sm}$ and Gd complexes, see: Dan et al. (2005); Li \& Wang (2010); Li (2011).


## 2. Experimental

### 2.1. Crystal data

$\left[\mathrm{Dy}_{2}\left(\mathrm{C}_{6} \mathrm{H}_{8} \mathrm{O}_{4}\right)\left(\mathrm{C}_{2} \mathrm{O}_{4}\right)_{2}\left(\mathrm{H}_{2} \mathrm{O}\right)_{2}\right]$

$$
M_{r}=681.20
$$

$$
\text { Triclinic, } P \overline{1}
$$

$$
\begin{aligned}
& a=6.772(2) \AA \\
& b=6.929(2) \AA
\end{aligned}
$$

$$
c=8.949(3) \AA \quad T=295 \mathrm{~K}
$$

$$
\alpha=104.916
$$

$$
\begin{aligned}
& \gamma=104.306(4)^{\circ} \\
& V=360.5(2) \AA^{3} \\
& Z=1 \\
& \text { Mo } K \alpha \text { radiation } \\
& \mu=10.37 \mathrm{~mm}^{-1} \\
& T=295 \mathrm{~K} \\
& 0.21 \times 0.09 \times 0.07 \mathrm{~mm}
\end{aligned}
$$

1813 measured reflections 1234 independent reflections 1179 reflections with $I>2 \sigma(I)$ $R_{\text {int }}=0.014$

### 2.2. Data collection

Bruker SMART APEXII CCD
area-detector diffractometer
Absorption correction: multi-scan
(SADABS; Sheldrick, 2003)

122 parameters
H -atom parameters constrained
$\Delta \rho_{\text {max }}=1.41 \mathrm{e}^{-3}$
$\Delta \rho_{\min }=-1.45 \mathrm{e}^{-3}$

Table 1
Hydrogen-bond geometry $\left(\AA,{ }^{\circ}\right)$.

| $D-\mathrm{H} \cdots A$ | $D-\mathrm{H}$ | $\mathrm{H} \cdots A$ | $D \cdots A$ | $D-\mathrm{H} \cdots A$ |
| :--- | :--- | :--- | :--- | :--- |
| ${\text { O7-H7A } \cdots{ }^{-} 3^{\mathrm{i}}}^{\text {i }}$ | 0.85 | 1.96 | $2.787(4)$ | 166 |
| ${\text { O7-H7B } \cdots \text { O6 }^{\text {ii }}}^{2}$ | 0.85 | 2.10 | $2.883(5)$ | 153 |

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

## Acknowledgements

This work was supported by the National Natural Science Foundation of China (51372104), the Natural Science Foundation of Jiangxi Province (2010GQC0064), the Science and Technology Support Fundation of Jiangxi Province (2012BBE500038, 20141BBE50019) and the Jiangxi University of Science and Technology Foundation (3304000027).

## data reports

Supporting information for this paper is available from the IUCr electronic archives (Reference: GK2622).

Dan, M., Cottereau, G. \& Rao, C. N. R. (2005). Solid State Sci. 7, 437-443.
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Li, Z.-F. \& Wang, C.-X. (2010). Acta Cryst. E66, m1263.
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Bruker (2004). APEX2 and SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.

## supporting information

Acta Cryst. (2014). E70, m399-m400 [doi:10.1107/S1600536814024544]

# Crystal structure of poly[ $\mu_{6}$-adipato-diaquadi- $\mu_{2}$-oxalato-didysprosium(III)] 

Zhi-Feng Li, Yi-Chao Zhang, Xiao-Qin Hu and Chun-Xiang Wang

## S1. Structural commentary

The title compound is isostructural with $\left[\mathrm{M}_{2}\left(\mathrm{C}_{6} \mathrm{H}_{8} \mathrm{O}_{4}\right)\left(\mathrm{C}_{2} \mathrm{O}_{4}\right)_{2}\left(\mathrm{H}_{2} \mathrm{O}\right)_{2}\right][M=\mathrm{La}, \mathrm{Sm}$, Gd$]$ (Dan et al., 2005; Li \& Wang, 2010; $\mathrm{Li}, 2011$ ). the asymmetric unit consists of one $\mathrm{Dy}^{3+}$ cation, a half of adipate anion, two half of oxalate anions and one aqua ligand (Fig. 1). The Dy atom is each coordinated by nine oxygen atoms, in which four oxygen atoms are from three adipate anions, four from two oxalate anions and one from a water molecule, to form a $\mathrm{DyO}_{9}$ polyhedron of a distorted tricapped trigonal-prismatic geometry. In the title complex, the adipate anions are located on a centre of symmetry and atom C3 is positionally disordered (C3A and C3B sites; occupancies $0.75 / 0.25$ ). The adipate ligands act in a $\eta^{2}, \mu_{3}-\eta^{2}, \mu_{3}$-chelating-bridging octadentate coordination modes and link the $\mathrm{DyO}_{9}$ polyhedra into layers parallel to ( 010 ), in which the adjacent Dy $\cdots$ Dy distances are 4.20 (2) $\AA$ and 4.223 (9) $\AA$, respectively. In the title complex, two symmetry independent oxalate ions are also located on centres of inversion and act as double bidentate (tetradentate) ligands in a zigzag chain along [001]. Through the oxalate and adipate ligands bridging interactions, the Dy atoms build up threedimensional framework. The aqua ligand provides hydrogen-bond donors form hydrogen bonds with oxalate atoms O3 and O6.

The structure of the title complex is similar to that of other lanthanide (gadolinium, samarium and lanthanum) coordination polymers with adipate and oxalate ligands, and the mean Dy-O distance in the title complex of $2.438 \AA$ is shorter than that of $\mathrm{Ga}-\mathrm{O}(2.463 \AA), \mathrm{Sm}-\mathrm{O}(2.482 \AA)$ and $\mathrm{La}-\mathrm{O}(2.566 \AA)$.

## S2. Synthesis and crystallization

A mixture of $\mathrm{DyCl}_{3} \cdot 6 \mathrm{H}_{2} \mathrm{O}(1.00 \mathrm{mmol}, 0.38 \mathrm{~g})$, oxalic acid ( $0.50 \mathrm{mmol}, 0.05 \mathrm{~g}$ ), adipic acid $(0.50 \mathrm{mmol}, 0.07 \mathrm{~g}), \mathrm{NaOH}$ $(2.00 \mathrm{mmol}, 0.08 \mathrm{~g})$ and $\mathrm{H}_{2} \mathrm{O}(10.0 \mathrm{ml})$ was heated in a 23 ml stainless steel reactor with a Teflon liner at 443 K for 48 h . A small amount of colorless plate-like crystals were filtered and washed with water and acetone. Yield 5\% based on Dy.

## S3. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. Atom C 3 of the adipate anion is positionally disordered ( C 3 A and C 3 B ) and these atoms were refined with occupancies of $0.75 / 0.25$. The C-bound H atoms were included in calculated positions and treated as riding atoms: $\mathrm{C}-\mathrm{H}=0.97 \AA$ and $\left.U_{\text {iso }}(\mathrm{H})=1.2 U_{\text {eq }}(\mathrm{C})\right]$. The water H -atoms were located in difference Fourier maps and were refined with distance restraints: $\mathrm{O}-\mathrm{H}$ distance of 0.85 $\AA$ and $U_{\text {iso }}(\mathrm{H})=1.5 U_{\text {eq }}(\mathrm{O})$. The highest density peak and deepest hole are located at $0.95 \AA$ and $0.87 \AA$ from the Dy atom, respectively.


## Figure 1

The fragment of the structure of the title compounds, with the atom-numbering scheme. Displacement ellipsoids are drawn at the $30 \%$ probability level and H atoms are shown as small spheres of arbitrary radii. Symmetry code: (i) $-x, 1-$ $y, 1-z$; (ii) $1-x, 1-y, 1-z$; (iii) $-x,-y, 1-z$; (iv) $-x,-y,-z$; (v) $1-x, 1-y, 2-z$.

## Poly $\left[\mu_{6}\right.$-adipato-diaquadi- $\mu_{2}$-oxalato-didysprosium(III)]

## Crystal data

$\left[\mathrm{Dy}_{2}\left(\mathrm{C}_{6} \mathrm{H}_{8} \mathrm{O}_{4}\right)\left(\mathrm{C}_{2} \mathrm{O}_{4}\right)_{2}\left(\mathrm{H}_{2} \mathrm{O}\right)_{2}\right]$
$M_{r}=681.20$
Triclinic, $P \overline{1}$
Hall symbol: -p 1
$a=6.772$ (2) $\AA$
$b=6.929$ (2) $\AA$
$c=8.949$ (3) $\AA$
$\alpha=104.916(5)^{\circ}$
$\beta=108.069(4)^{\circ}$
$\gamma=104.306(4)^{\circ}$
$V=360.5$ (2) $\AA^{3}$

## Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan
(SADABS; Sheldrick, 2003)
$T_{\text {min }}=0.245, T_{\text {max }}=0.531$

## Refinement

Refinement on $F^{2}$
Least-squares matrix: full
$R\left[F^{2}>2 \sigma\left(F^{2}\right)\right]=0.021$
$w R\left(F^{2}\right)=0.057$
$S=1.05$
1234 reflections
$Z=1$
$F(000)=316$
$D_{\mathrm{x}}=3.138 \mathrm{Mg} \mathrm{m}^{-3}$
Mo $K \alpha$ radiation, $\lambda=0.71073 \AA$
Cell parameters from 198 reflections
$\theta=4.6-28.3^{\circ}$
$\mu=10.37 \mathrm{~mm}^{-1}$
$T=295 \mathrm{~K}$
Plate, colorless
$0.21 \times 0.09 \times 0.07 \mathrm{~mm}$

1813 measured reflections
1234 independent reflections
1179 reflections with $I>2 \sigma(I)$
$R_{\text {int }}=0.014$
$\theta_{\text {max }}=25.0^{\circ}, \theta_{\text {min }}=2.6^{\circ}$
$h=-8 \rightarrow 7$
$k=-6 \rightarrow 8$
$l=-10 \rightarrow 7$

122 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites
H -atom parameters constrained
$w=1 /\left[\sigma^{2}\left(F_{0}^{2}\right)+(0.0422 P)^{2}+0.1716 P\right]$
where $P=\left(F_{0}{ }^{2}+2 F_{\mathrm{c}}{ }^{2}\right) / 3$
$(\Delta / \sigma)_{\text {max }}<0.001$

$$
\Delta \rho_{\max }=1.41 \mathrm{e} \AA^{-3}
$$

$\Delta \rho_{\text {min }}=-1.45 \mathrm{e}^{-3}$
Extinction correction: SHELXL97 (Sheldrick, 2008), $\mathrm{Fc}^{*}=\mathrm{kFc}\left[1+0.001 \mathrm{xFc}^{2} \lambda^{3} / \sin (2 \theta)\right]^{-1 / 4}$

Extinction coefficient: 0.0039 (11)

## 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 1.s. planes.
Refinement. Refinement of $F^{2}$ against ALL reflections. The weighted $R$-factor $w R$ 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 $F^{2}>\sigma\left(F^{2}\right)$ is used only for calculating $R$-factors $(\mathrm{gt})$ 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 ( $\dot{A}^{2}$ )

|  | $x$ | $y$ | $z$ | $U_{\text {iso }} * / U_{\text {eq }}$ | Occ. $(<1)$ |
| :---: | :---: | :---: | :---: | :---: | :---: |
| Dy | 0.15576 (3) | 0.34496 (3) | 0.36280 (2) | 0.01182 (14) |  |
| O1 | 0.1519 (6) | 0.5222 (6) | 0.6470 (4) | 0.0145 (7) |  |
| O2 | 0.4801 (6) | 0.5972 (6) | 0.6395 (4) | 0.0162 (8) |  |
| O3 | 0.2699 (6) | 0.1546 (6) | 0.5391 (4) | 0.0154 (8) |  |
| O4 | 0.1535 (6) | -0.0812 (6) | 0.6512 (5) | 0.0181 (8) |  |
| O5 | -0.0072 (6) | 0.2564 (6) | 0.0712 (4) | 0.0172 (8) |  |
| O6 | -0.1314 (7) | 0.0203 (6) | -0.1913 (5) | 0.0201 (8) |  |
| 07 | 0.2677 (6) | 0.6848 (6) | 0.3381 (5) | 0.0206 (8) |  |
| H7A | 0.4060 | 0.7539 | 0.3786 | 0.031* |  |
| H7B | 0.1863 | 0.7472 | 0.2931 | 0.031* |  |
| C1 | 0.3632 (9) | 0.6131 (8) | 0.7234 (6) | 0.0135 (11) |  |
| C2 | 0.4635 (9) | 0.7252 (9) | 0.9114 (7) | 0.0178 (12) |  |
| H2A | 0.3960 | 0.8298 | 0.9403 | 0.021* |  |
| H2B | 0.6211 | 0.8016 | 0.9479 | 0.021* |  |
| C3A | 0.4334 (12) | 0.5740 (12) | 1.0066 (9) | 0.0162 (14) | 0.75 |
| H3A1 | 0.2770 | 0.4877 | 0.9624 | 0.019* | 0.75 |
| H3A2 | 0.4788 | 0.6577 | 1.1245 | 0.019* | 0.75 |
| C3B | 0.576 (4) | 0.593 (4) | 1.005 (3) | 0.0162 (14) | 0.25 |
| H3B1 | 0.6770 | 0.5536 | 0.9564 | 0.019* | 0.25 |
| H3B2 | 0.6640 | 0.6824 | 1.1227 | 0.019* | 0.25 |
| C4 | 0.1215 (8) | 0.0215 (7) | 0.5549 (6) | 0.0127 (10) |  |
| C5 | -0.0395 (8) | 0.0806 (8) | -0.0350 (7) | 0.0150 (11) |  |

## Atomic displacement parameters $\left(\AA^{2}\right)$

|  | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
| :--- | :--- | :--- | :--- | :--- | :--- | :--- |
| Dy | $0.01277(19)$ | $0.01272(18)$ | $0.01252(19)$ | $0.00465(11)$ | $0.00650(12)$ | $0.00663(11)$ |
| O1 | $0.0132(17)$ | $0.0169(18)$ | $0.0168(18)$ | $0.0054(14)$ | $0.0085(15)$ | $0.0081(14)$ |
| O2 | $0.0179(19)$ | $0.0165(19)$ | $0.0181(19)$ | $0.0061(15)$ | $0.0117(16)$ | $0.0070(14)$ |
| O3 | $0.0135(18)$ | $0.0175(19)$ | $0.0164(18)$ | $0.0052(15)$ | $0.0049(15)$ | $0.0093(15)$ |


|  |  |  |  |  |  |  |
| :--- | :--- | :--- | :--- | :--- | :--- | :--- |
| O4 | $0.0134(18)$ | $0.022(2)$ | $0.0213(19)$ | $0.0068(15)$ | $0.0054(15)$ | $0.0131(16)$ |
| O5 | $0.0220(19)$ | $0.0161(19)$ | $0.0167(19)$ | $0.0095(15)$ | $0.0092(16)$ | $0.0062(15)$ |
| O6 | $0.027(2)$ | $0.022(2)$ | $0.014(2)$ | $0.0111(17)$ | $0.0083(17)$ | $0.0095(16)$ |
| O7 | $0.0178(19)$ | $0.020(2)$ | $0.031(2)$ | $0.0080(16)$ | $0.0105(17)$ | $0.0177(17)$ |
| C1 | $0.021(3)$ | $0.008(2)$ | $0.015(3)$ | $0.009(2)$ | $0.007(2)$ | $0.0074(19)$ |
| C2 | $0.017(3)$ | $0.019(3)$ | $0.019(3)$ | $0.006(2)$ | $0.009(2)$ | $0.007(2)$ |
| C3A | $0.017(3)$ | $0.029(4)$ | $0.013(3)$ | $0.013(3)$ | $0.010(3)$ | $0.012(3)$ |
| C3B | $0.017(3)$ | $0.029(4)$ | $0.013(3)$ | $0.013(3)$ | $0.010(3)$ | $0.012(3)$ |
| C4 | $0.018(3)$ | $0.008(2)$ | $0.015(2)$ | $0.0022(19)$ | $0.011(2)$ | $0.0058(19)$ |
| C5 | $0.012(2)$ | $0.019(3)$ | $0.019(3)$ | $0.007(2)$ | $0.009(2)$ | $0.009(2)$ |

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

| Dy-05 | 2.336 (4) | O7-H7A | 0.8468 |
| :---: | :---: | :---: | :---: |
| Dy-04 ${ }^{\text {i }}$ | 2.360 (4) | O7-H7B | 0.8496 |
| Dy-03 | 2.376 (4) | $\mathrm{C} 1-\mathrm{C} 2$ | 1.501 (7) |
| Dy-07 | 2.379 (4) | $\mathrm{C} 2-\mathrm{C} 3 \mathrm{~A}$ | 1.525 (9) |
| Dy-O2 ${ }^{\text {ii }}$ | 2.406 (3) | $\mathrm{C} 2-\mathrm{C} 3 \mathrm{~B}$ | 1.57 (2) |
| Dy-O1 ${ }^{\text {iii }}$ | 2.464 (4) | $\mathrm{C} 2-\mathrm{H} 2 \mathrm{~A}$ | 0.9700 |
| Dy-O6 ${ }^{\text {iv }}$ | 2.535 (4) | $\mathrm{C} 2-\mathrm{H} 2 \mathrm{~B}$ | 0.9700 |
| Dy-01 | 2.535 (3) | $\mathrm{C} 3 \mathrm{~A}-\mathrm{C} 3 \mathrm{~A}^{\text {v }}$ | 1.529 (14) |
| Dy-02 | 2.552 (4) | C3A-H3A1 | 0.9700 |
| $\mathrm{O} 1-\mathrm{C} 1$ | 1.279 (7) | C3A-H3A2 | 0.9700 |
| $\mathrm{O} 2-\mathrm{C} 1$ | 1.255 (6) | $\mathrm{C} 3 \mathrm{~B}-\mathrm{C} 3 \mathrm{~B}^{\text {v }}$ | 1.40 (4) |
| O3-C4 | 1.256 (6) | C3B-H3B1 | 0.9700 |
| O4-C4 | 1.253 (6) | C3B-H3B2 | 0.9700 |
| O5-C5 | 1.256 (6) | $\mathrm{C} 4-\mathrm{C} 4^{\text {i }}$ | 1.538 (10) |
| O6-C5 | 1.245 (7) | C5-C5 ${ }^{\text {iv }}$ | 1.540 (11) |
| O6-Dy ${ }^{\text {iv }}$ | 2.535 (4) |  |  |
| O5-Dy-O4 ${ }^{\text {i }}$ | 90.45 (13) | C1-O1-Dy | 95.2 (3) |
| $\mathrm{O} 5-\mathrm{Dy}-\mathrm{O} 3$ | 134.27 (12) | Dy ${ }^{\text {iii }}$-O1-Dy | 115.28 (14) |
| $\mathrm{O} 4{ }^{\text {i }}$ - Dy-O3 | 68.93 (12) | $\mathrm{C} 1-\mathrm{O} 2-\mathrm{Dy}{ }^{\text {ii }}$ | 148.4 (3) |
| O5-Dy-07 | 78.20 (13) | C1-O2-Dy | 95.1 (3) |
| O4--Dy-07 | 142.35 (13) | Dyii-O2-Dy | 115.66 (13) |
| O3-Dy-07 | 141.46 (12) | C4-O3-Dy | 117.8 (3) |
| $\mathrm{O} 5-\mathrm{Dy}-\mathrm{O} 2^{\text {ii }}$ | 91.79 (12) | C4-O4-Dy ${ }^{\text {i }}$ | 118.7 (3) |
| $\mathrm{O} 4{ }^{\text {i }}$-Dy-O2 $2^{\text {ii }}$ | 142.20 (13) | C5-O5-Dy | 123.8 (3) |
| $\mathrm{O} 3-\mathrm{Dy}-\mathrm{O} 2{ }^{\text {ii }}$ | 82.95 (12) | C5-O6-Dy ${ }^{\text {iv }}$ | 117.5 (3) |
| $\mathrm{O} 7-\mathrm{Dy}-\mathrm{O} 2^{\text {ii }}$ | 74.65 (13) | Dy-07-H7A | 116.9 |
| $\mathrm{O} 5-\mathrm{Dy}-\mathrm{O} 1^{\text {iii }}$ | 82.15 (12) | Dy-07-H7B | 128.4 |
| O 4 - Dy-O1 ${ }^{\text {iii }}$ | 69.07 (12) | H7A-O7-H7B | 114.8 |
| $\mathrm{O} 3-\mathrm{Dy}-\mathrm{O} 1^{\text {iii }}$ | 122.91 (11) | $\mathrm{O} 2-\mathrm{C} 1-\mathrm{O} 1$ | 118.8 (5) |
| $\mathrm{O} 7-\mathrm{Dy}-\mathrm{O}^{1 \mathrm{iii}}$ | 73.84 (12) | $\mathrm{O} 2-\mathrm{C} 1-\mathrm{C} 2$ | 122.2 (5) |
| $\mathrm{O} 2{ }^{\text {iii }}-\mathrm{Dy}-\mathrm{O} 1^{\text {iii }}$ | 148.49 (12) | $\mathrm{O} 1-\mathrm{C} 1-\mathrm{C} 2$ | 119.0 (4) |
| $\mathrm{O} 5-\mathrm{Dy}-\mathrm{OG}^{\text {iv }}$ | 65.84 (12) | $\mathrm{O} 2-\mathrm{C} 1-\mathrm{Dy}$ | 59.8 (3) |
| O 4 - Dy- $\mathrm{O}^{\text {iv }}$ | 70.76 (13) | O1-C1-Dy | 59.1 (2) |
| $\mathrm{O} 3-\mathrm{Dy}-\mathrm{Of}^{\text {iv }}$ | 68.84 (12) | C2-C1-Dy | 173.1 (4) |


| O7-Dy-O6 ${ }^{\text {iv }}$ | 132.07 (13) |
| :---: | :---: |
| $\mathrm{O} 2{ }^{\text {ii- }}$ - $\mathrm{Dy}-\mathrm{O} 6^{\mathrm{iv}}$ | 75.88 (12) |
| $\mathrm{O} 1^{\text {iii- }}$ - $\mathrm{Dy}-\mathrm{O}^{\text {iv }}$ | 127.55 (12) |
| O5-Dy-O1 | 146.78 (12) |
| O4--Dy-O1 | 80.36 (12) |
| $\mathrm{O} 3-\mathrm{Dy}-\mathrm{O} 1$ | 71.64 (12) |
| O7-Dy-O1 | 89.76 (13) |
| $\mathrm{O} 2 \mathrm{ii}-\mathrm{Dy}-\mathrm{O} 1$ | 114.93 (11) |
| $\mathrm{O} 1 \mathrm{iii}-\mathrm{Dy}-\mathrm{O} 1$ | 64.72 (14) |
| O6 ${ }^{\text {iv }}-\mathrm{Dy}-\mathrm{O} 1$ | 137.19 (12) |
| $\mathrm{O} 5-\mathrm{Dy}-\mathrm{O} 2$ | 146.25 (12) |
| O 4 - $\mathrm{Dy}^{\text {- }}$ O2 | 123.15 (12) |
| $\mathrm{O} 3-\mathrm{Dy}-\mathrm{O} 2$ | 69.30 (12) |
| O7-Dy-O2 | 72.79 (13) |
| $\mathrm{O} 2 \mathrm{ii}-\mathrm{Dy}-\mathrm{O} 2$ | 64.34 (13) |
| $\mathrm{O} 1{ }^{\text {iii }}$-Dy-O2 | 105.43 (11) |
| $\mathrm{O}^{\text {iv }}-\mathrm{Dy}-\mathrm{O} 2$ | 124.52 (12) |
| $\mathrm{O} 1-\mathrm{Dy}-\mathrm{O} 2$ | 50.76 (11) |
| O5-Dy-C1 | 158.48 (13) |
| O4i-Dy-C1 | 101.76 (14) |
| O3-Dy-C1 | 67.18 (13) |
| O7-Dy-C1 | 81.38 (14) |
| $\mathrm{O} 2 \mathrm{ii}-\mathrm{Dy}-\mathrm{C} 1$ | 89.29 (13) |
| $\mathrm{O} 1^{\text {iii }}-\mathrm{Dy}-\mathrm{C} 1$ | 85.74 (13) |
| O6 ${ }^{\text {iv }}-\mathrm{Dy}-\mathrm{C} 1$ | 134.90 (13) |
| O1-Dy-C1 | 25.66 (13) |
| $\mathrm{O} 2-\mathrm{Dy}-\mathrm{C} 1$ | 25.14 (13) |
| C1-O1-Dy ${ }^{\text {iii }}$ | 132.5 (3) |
| $\mathrm{O} 5-\mathrm{Dy}-\mathrm{O} 1-\mathrm{C} 1$ | -138.0 (3) |
| $\mathrm{O} 4-\mathrm{Dy}-\mathrm{O} 1-\mathrm{Cl}$ | 146.2 (3) |
| $\mathrm{O} 3-\mathrm{Dy}-\mathrm{O} 1-\mathrm{C} 1$ | 75.4 (3) |
| O7-Dy-O1-C1 | -70.3 (3) |
| $\mathrm{O} 2 \mathrm{ii}-\mathrm{Dy}-\mathrm{O} 1-\mathrm{C} 1$ | 2.6 (3) |
| $\mathrm{O} 1{ }^{\text {iii }}-\mathrm{Dy}-\mathrm{O} 1-\mathrm{C} 1$ | -142.5 (4) |
| $\mathrm{O}^{\text {iv }}-\mathrm{Dy}-\mathrm{O} 1-\mathrm{C} 1$ | 98.7 (3) |
| $\mathrm{O} 2-\mathrm{Dy}-\mathrm{O} 1-\mathrm{C} 1$ | -2.5 (3) |
| O5-Dy-O1-Dy ${ }^{\text {iii }}$ | 4.5 (3) |
| O4-Dy-O1-Dy ${ }^{\text {iii }}$ | -71.31 (15) |
| O3-Dy-O1-Dy ${ }^{\text {iii }}$ | -142.17 (17) |
| O7-Dy-O1-Dy ${ }^{\text {iii }}$ | 72.19 (15) |
| $\mathrm{O} 2 \mathrm{ii}-\mathrm{Dy}-\mathrm{O} 1-\mathrm{Dy}{ }^{\text {iii }}$ | 145.10 (14) |
| O1 ${ }^{\text {iii- }}$ - Dy-O1-Dy ${ }^{\text {iii }}$ | 0.0 |
| O6 ${ }^{\text {iv }}$ - Dy-O1-Dy ${ }^{\text {iii }}$ | -118.82 (18) |
| O2-Dy-O1-Dy ${ }^{\text {iii }}$ | 139.9 (2) |
| C1-Dy-O1-Dy ${ }^{\text {iii }}$ | 142.5 (4) |
| $\mathrm{O} 5-\mathrm{Dy}-\mathrm{O} 2-\mathrm{C} 1$ | 138.8 (3) |
| $\mathrm{O} 4-\mathrm{Dy}-\mathrm{O} 2-\mathrm{Cl}$ | -35.1 (3) |


| $\mathrm{C} 1-\mathrm{C} 2-\mathrm{C} 3 \mathrm{~A}$ | 113.3 (5) |
| :---: | :---: |
| C1-C2-C3B | 111.8 (9) |
| $\mathrm{C} 1-\mathrm{C} 2-\mathrm{H} 2 \mathrm{~A}$ | 108.9 |
| $\mathrm{C} 3 \mathrm{~A}-\mathrm{C} 2-\mathrm{H} 2 \mathrm{~A}$ | 108.9 |
| $\mathrm{C} 3 \mathrm{~B}-\mathrm{C} 2-\mathrm{H} 2 \mathrm{~A}$ | 134.9 |
| $\mathrm{C} 1-\mathrm{C} 2-\mathrm{H} 2 \mathrm{~B}$ | 108.9 |
| $\mathrm{C} 3 \mathrm{~A}-\mathrm{C} 2-\mathrm{H} 2 \mathrm{~B}$ | 108.9 |
| $\mathrm{C} 3 \mathrm{~B}-\mathrm{C} 2-\mathrm{H} 2 \mathrm{~B}$ | 76.5 |
| $\mathrm{H} 2 \mathrm{~A}-\mathrm{C} 2-\mathrm{H} 2 \mathrm{~B}$ | 107.7 |
| $\mathrm{C} 2-\mathrm{C} 3 \mathrm{~A}-\mathrm{C} 3 \mathrm{~A}^{\text {v }}$ | 113.2 (7) |
| C2-C3A-H3A1 | 108.9 |
| C3A ${ }^{\text {- }}$ - 3 A -H 3 A 1 | 108.9 |
| C2-C3A-H3A2 | 108.9 |
| $\mathrm{C} 3 \mathrm{~A}^{\mathrm{v}}-\mathrm{C} 3 \mathrm{~A}-\mathrm{H} 3 \mathrm{~A} 2$ | 108.9 |
| H3A1-C3A-H3A2 | 107.8 |
| $\mathrm{C} 3 \mathrm{~B}^{v}-\mathrm{C} 3 \mathrm{~B}-\mathrm{C} 2$ | 113 (2) |
| $\mathrm{C} 3 \mathrm{~B}^{v}-\mathrm{C} 3 \mathrm{~B}-\mathrm{H} 3 \mathrm{~B} 1$ | 108.9 |
| C2-C3B-H3B1 | 108.9 |
| $\mathrm{C} 3 \mathrm{~B}-\mathrm{C} 3 \mathrm{~B}-\mathrm{H} 3 \mathrm{~B} 2$ | 108.9 |
| C2-C3B-H3B2 | 108.9 |
| H3B1-C3B-H3B2 | 107.7 |
| $\mathrm{O} 4-\mathrm{C} 4-\mathrm{O} 3$ | 125.9 (5) |
| O4-C4-C4 ${ }^{\text {i }}$ | 116.9 (5) |
| O3-C4-C4 ${ }^{\text {i }}$ | 117.2 (5) |
| O6-C5-O5 | 127.2 (5) |
| O6-C5-C5 ${ }^{\text {iv }}$ | 116.0 (6) |
| O5-C5-C5 ${ }^{\text {iv }}$ | 116.8 (6) |


| $\mathrm{O} 1{ }^{\text {iii- }}$ - $\mathrm{Dy}-\mathrm{O} 5-\mathrm{C} 5$ | -135.5 (4) |
| :---: | :---: |
| O6 ${ }^{\text {iv }}$ - $\mathrm{Dy}-\mathrm{O} 5-\mathrm{C} 5$ | 1.9 (4) |
| $\mathrm{O} 1-\mathrm{Dy}-\mathrm{O} 5-\mathrm{C} 5$ | -139.6 (4) |
| $\mathrm{O} 2-\mathrm{Dy}-\mathrm{O} 5-\mathrm{C} 5$ | 118.4 (4) |
| $\mathrm{C} 1-\mathrm{Dy}-\mathrm{O} 5-\mathrm{C} 5$ | 168.2 (4) |
| Dy ${ }^{\text {ii }}-\mathrm{O} 2-\mathrm{C} 1-\mathrm{O} 1$ | -171.1 (4) |
| Dy-O2-C1-O1 | -4.5 (5) |
| Dyiin $\mathrm{O} 2-\mathrm{C} 1-\mathrm{C} 2$ | 5.7 (9) |
| Dy-O2-C1-C2 | 172.3 (4) |
| Dy ${ }^{\text {iii }} \mathrm{O} 2-\mathrm{C} 1-\mathrm{Dy}$ | -166.6 (6) |
| Dyiii- $\mathrm{O} 1-\mathrm{C} 1-\mathrm{O} 2$ | -127.1 (4) |
| Dy-O1-C1-O2 | 4.5 (5) |
| Dy ${ }^{\text {iii }}-\mathrm{O} 1-\mathrm{C} 1-\mathrm{C} 2$ | 56.0 (6) |
| Dy-O1-C1-C2 | -172.3 (4) |
| Dy ${ }^{\text {iii }}$-O1-C1-Dy | -131.7 (4) |
| $\mathrm{O} 5-\mathrm{Dy}-\mathrm{C} 1-\mathrm{O} 2$ | -86.1 (5) |
| $\mathrm{O} 4-\mathrm{Dy}-\mathrm{C} 1-\mathrm{O} 2$ | 150.6 (3) |
| $\mathrm{O} 3-\mathrm{Dy}-\mathrm{C} 1-\mathrm{O} 2$ | 89.7 (3) |
| $\mathrm{O} 7-\mathrm{Dy}-\mathrm{C} 1-\mathrm{O} 2$ | -67.6 (3) |


| O3-Dy-O2-C1 | -80.2 (3) |
| :---: | :---: |
| $\mathrm{O} 7-\mathrm{Dy}-\mathrm{O} 2-\mathrm{C} 1$ | 106.9 (3) |
| $\mathrm{O} 2 \mathrm{ii}-\mathrm{Dy}-\mathrm{O} 2-\mathrm{C} 1$ | -172.2 (4) |
| $\mathrm{O} 1{ }^{\text {iii- }}$ - $\mathrm{Dy}-\mathrm{O} 2-\mathrm{C} 1$ | 39.7 (3) |
| $\mathrm{O6}^{\text {iv }}-\mathrm{Dy}-\mathrm{O} 2-\mathrm{C} 1$ | -123.4 (3) |
| $\mathrm{O} 1-\mathrm{Dy}-\mathrm{O} 2-\mathrm{C} 1$ | 2.6 (3) |
| O5-Dy-O2-Dy ${ }^{\text {ii }}$ | -49.0 (3) |
| O4-Dy-O2-Dy ${ }^{\text {ii }}$ | 137.16 (15) |
| $\mathrm{O} 3-\mathrm{Dy}-\mathrm{O} 2-\mathrm{Dy}{ }^{\text {ii }}$ | 92.06 (16) |
| $\mathrm{O} 7-\mathrm{Dy}-\mathrm{O} 2-\mathrm{Dy}{ }^{\text {ii }}$ | -80.87 (16) |
| $\mathrm{O} 2{ }^{\text {ii }}$-Dy-O2-Dy ${ }^{\text {ii }}$ | 0.001 (1) |
| $\mathrm{O} 1^{\text {iiii- }}$ - ${ }^{\text {dy }}$-O2-Dy ${ }^{\text {ii }}$ | -148.06 (15) |
| O6 ${ }^{\text {iv }}-\mathrm{Dy}-\mathrm{O} 2-\mathrm{Dy}{ }^{\text {ii }}$ | 48.8 (2) |
| O1-Dy-O2-Dy ${ }^{\text {ii }}$ | 174.8 (2) |
| $\mathrm{C} 1-\mathrm{Dy}-\mathrm{O} 2-\mathrm{Dy}{ }^{\text {ii }}$ | 172.2 (4) |
| O5-Dy-O3-C4 | -74.6 (4) |
| $\mathrm{O} 4-\mathrm{Dy}-\mathrm{O} 3-\mathrm{C} 4$ | -5.9 (3) |
| $\mathrm{O} 7-\mathrm{Dy}-\mathrm{O} 3-\mathrm{C} 4$ | 145.5 (3) |
| $\mathrm{O} 2 \mathrm{ii}-\mathrm{Dy}-\mathrm{O} 3-\mathrm{C} 4$ | -160.2 (3) |
| $\mathrm{O} 1^{\text {iiii- }}$ - ${ }^{\text {dy }}-\mathrm{O} 3-\mathrm{C} 4$ | 39.2 (4) |
| O6 ${ }^{\text {iv }}-\mathrm{Dy}-\mathrm{O} 3-\mathrm{C} 4$ | -82.6 (3) |
| $\mathrm{O} 1-\mathrm{Dy}-\mathrm{O} 3-\mathrm{C} 4$ | 80.6 (3) |
| $\mathrm{O} 2-\mathrm{Dy}-\mathrm{O} 3-\mathrm{C} 4$ | 134.6 (4) |
| C1-Dy-O3-C4 | 107.6 (4) |
| $\mathrm{O} 4-\mathrm{Dy}-\mathrm{O} 5-\mathrm{C} 5$ | -66.7 (4) |
| O3-Dy-O5-C5 | -6.3 (5) |
| O7-Dy-O5-C5 | 149.5 (4) |
| O2 ${ }^{\text {iii-Dy }}$-O5-C5 | 75.6 (4) |


| $\mathrm{O} 2{ }^{\text {iii }}-\mathrm{Dy}-\mathrm{C} 1-\mathrm{O} 2$ | 7.0 (3) |
| :---: | :---: |
| $\mathrm{O} 1 \mathrm{iii}-\mathrm{Dy}-\mathrm{C} 1-\mathrm{O} 2$ | -141.9 (3) |
| $\mathrm{O} 6^{\mathrm{iv}}-\mathrm{Dy}-\mathrm{C} 1-\mathrm{O} 2$ | 76.1 (3) |
| $\mathrm{O} 1-\mathrm{Dy}-\mathrm{C} 1-\mathrm{O} 2$ | -175.4 (5) |
| $\mathrm{O} 5-\mathrm{Dy}-\mathrm{C} 1-\mathrm{O} 1$ | 89.3 (5) |
| $\mathrm{O} 4-\mathrm{Dy}-\mathrm{C} 1-\mathrm{O} 1$ | -34.1 (3) |
| $\mathrm{O} 3-\mathrm{Dy}-\mathrm{C} 1-\mathrm{O} 1$ | -95.0 (3) |
| O7-Dy-C1-O1 | 107.8 (3) |
| $\mathrm{O} 2{ }^{\text {iii }}-\mathrm{Dy}-\mathrm{C} 1-\mathrm{O} 1$ | -177.6 (3) |
| $\mathrm{O} 1 \mathrm{iii}-\mathrm{Dy}-\mathrm{C} 1-\mathrm{O} 1$ | 33.5 (3) |
| $\mathrm{O} 6^{\text {iv }}-\mathrm{Dy}-\mathrm{C} 1-\mathrm{O} 1$ | -108.5 (3) |
| $\mathrm{O} 2-\mathrm{Dy}-\mathrm{C} 1-\mathrm{O} 1$ | 175.4 (5) |
| $\mathrm{O} 2-\mathrm{C} 1-\mathrm{C} 2-\mathrm{C} 3 \mathrm{~A}$ | -110.4 (6) |
| $\mathrm{O} 1-\mathrm{C} 1-\mathrm{C} 2-\mathrm{C} 3 \mathrm{~A}$ | 66.3 (7) |
| $\mathrm{O} 2-\mathrm{C} 1-\mathrm{C} 2-\mathrm{C} 3 \mathrm{~B}$ | -71.7 (11) |
| $\mathrm{O} 1-\mathrm{C} 1-\mathrm{C} 2-\mathrm{C} 3 \mathrm{~B}$ | 105.1 (10) |
|  | 68.2 (9) |
| $\mathrm{C} 3 \mathrm{~B}-\mathrm{C} 2-\mathrm{C} 3 \mathrm{~A}-\mathrm{C} 3 \mathrm{~A}^{\text {v }}$ | -27.2 (14) |
| $\mathrm{C} 1-\mathrm{C} 2-\mathrm{C} 3 \mathrm{~B}-\mathrm{C} 3 \mathrm{~B}^{\text {v }}$ | -70 (2) |
| C3A-C2-C3B-C3B ${ }^{\text {v }}$ | 29.9 (15) |
| Dy ${ }^{\text {i }}$-O4-C4-O3 | -174.4 (4) |
| Dyi- $04-\mathrm{C} 4-\mathrm{C} 4{ }^{\text {i }}$ | 4.9 (7) |
| Dy-O3-C4-O4 | -175.0 (4) |
| Dy-O3-C4-C4 ${ }^{\text {i }}$ | 5.8 (7) |
| Dy ${ }^{\text {iv }}$-O6-C5-O5 | 178.5 (4) |
| Dy ${ }^{\text {iv }}$-O6-C5-C5 ${ }^{\text {iv }}$ | -2.6 (7) |
| Dy-O5-C5-O6 | 177.5 (4) |
| Dy-O5-C5-C5iv | -1.4 (7) |

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

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

| $D — \mathrm{H} \cdots A$ | $D-\mathrm{H}$ | $\mathrm{H} \cdots A$ | $D \cdots A$ | $D-\mathrm{H}^{\cdots} A$ |
| :--- | :--- | :--- | :--- | :--- |
| O7—H7A $\cdots \mathrm{O}^{\text {ii }}$ | 0.85 | 1.96 | $2.787(4)$ | 166 |
| O7—H7 $^{\mathrm{H}} \cdots$ O6 $^{\text {vi }}$ | 0.85 | 2.10 | $2.883(5)$ | 153 |

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

