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# Bis(μ-2-{[2-(1,3-benzothiazol-2-yl)-hydrazinylidene]methyl}-6-methoxyphenolato)bis[dinitratodysprosium(III)] methanol disolvate

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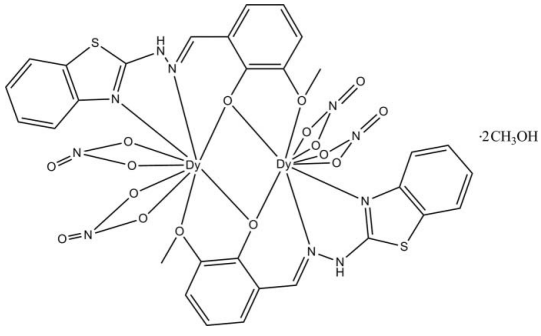
Key indicators: single-crystal X-ray study; $T$ = 159 K; mean $\sigma$(C–C) = 0.007 Å; $R$ factor = 0.025; $wR$ factor = 0.057; data-to-parameter ratio = 12.0.

In the centrosymmetric dinuclear title compound, $[Dy_2(C_{15}H_{12}N_3O_2S)_2(NO_3)_4]\cdot 2CH_3OH$, the two $Dy^{III}$ atoms are coordinated by two deprotonated 2-{[2-(1,3-benzothiazol-2-yl)hydrazinylidene]methyl}-6-methoxyphenol ligands and four nitrate ions, all of which are chelating. The crystal packing is stabilized by intermolecular N—H···O hydrogen bonds and weak O—H···O interactions, forming a two-dimensional network parallel to (010).

## Related literature

For applications of dysprosium complexes in data storage and processing, see: Lin *et al.* (2010). For the preparation of the 2-{[2-(1,3-benzothiazol-2-yl)hydrazinylidene]methyl}-6-methoxyphenol ligand, see: Patil *et al.* (2009). For related structures, see: Lin & Hong (2009); Lin *et al.* (2008); Xu *et al.* (2010).




## Experimental

### Crystal data

$[Dy_2(C_{15}H_{12}N_3O_2S)_2(NO_3)_4]\cdot 2CH_4O$
$M_r$ = 1233.82
Triclinic, $P\overline{1}$
$a$ = 9.6191 (6) Å
$b$ = 10.1002 (7) Å
$c$ = 11.6151 (8) Å
$\alpha$ = 112.045 (1)°
$\beta$ = 105.065 (1)°
$\gamma$ = 93.154 (1)°
$V$ = 995.23 (12) Å$^3$
$Z$ = 1
Mo $K\alpha$ radiation
$\mu$ = 3.92 mm$^{-1}$
$T$ = 159 K
0.20 × 0.10 × 0.10 mm

### Data collection

Rigaku Saturn CCD area-detector diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2002)
$T_{min}$ = 0.631, $T_{max}$ = 0.676
5083 measured reflections
3502 independent reflections
3162 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.016

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.057
$S$ = 1.07
3502 reflections
292 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.83 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.51 e Å$^{-3}$

**Table 1**
Selected bond lengths (Å).

| | | | |
|---|---|---|---|
| Dy1—O2 | 2.280 (3) | Dy1—N4 | 2.461 (3) |
| Dy1—O2$^{i}$ | 2.374 (2) | Dy1—O6 | 2.471 (3) |
| Dy1—O1$^{i}$ | 2.394 (3) | Dy1—N5 | 2.494 (3) |
| Dy1—O4 | 2.422 (3) | Dy1—O8 | 2.530 (3) |
| Dy1—O3 | 2.433 (3) | | |

Symmetry code: (i) $-x+1, -y, -z+2$.

**Table 2**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O9—H9$A$···O3$^{ii}$ | 0.84 | 2.64 | 3.206 (4) | 126 |
| O9—H9$A$···O8$^{iii}$ | 0.84 | 2.24 | 3.049 (4) | 161 |
| N9—H9···O9$^{iv}$ | 0.88 | 1.91 | 2.751 (4) | 160 |

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

Data collection: *CrystalClear* (Rigaku, 2002); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2006) and *XP* (Siemens, 1994); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JJ2083).

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# supplementary materials

*Acta Cryst.* (2011). E**67**, m796-m797 [ **doi:10.1107/S1600536811019118** ]

# Bis(μ-2-{[2-(1,3-benzothiazol-2-yl)hydrazinylidene]methyl}-6-methoxyphenolato)bis[dinitratodysprosium(III)] methanol disolvate

**X. Xu, S. Ding, S. Shen, J. Tang and Z. Liu**

## Comment

Dysprosium complexes continue to attract significant attention because of their potential applications in data storage and processing (Lin *et al.* 2010). In order to explore the relationship between these applications and their structures, a series of dinuclear dysprosium coordination compounds have been structural characterized (Lin *et al.* 2008; Xu *et al.* 2010). In support of our continued research in this area, we report here a new dinuclear dysprosium complex, $[Dy_2(C_{15}H_{12}N_3O_2S)_2(NO_3)_4].CH_3OH$.

In the title compound the asymmetric unit consists of one $Dy^{III}$ ion, one deprotonated 2-[(benzothiazol-2-yl)hydrazonomethyl]-6-methoxyphenol ligand and two nitrate ions (Fig. 1). The centrosymmetric dinuclear complex is composed of two nine-coordinate $Dy^{III}$ ions bridged by phenoxo groups (O2, O2a) from the ligands with a Dy1—O2—Dy1a angle of 106.041 (100) ° and a Dy···Dy distance equal to 3.7184 (3) Å. The central core $Dy_2O_2$ appears to be nearly rhombic with the two Dy—O2 distances being 2.37 Å and 2.28 Å, respectively. Crystal packing is stabiized by N—H···O hydrogen bonds and weak O—H···O intermolecular interactions forming a two-dimensional network (Fig. 2).

## Experimental

A mixture of $Dy(NO_3)_3.6H_2O$ (0.1 mmol), 2-hydrazino benzothiazole (0.1 mmol), 2-hydroxy-3-methoxy-5-((4-methoxyphenyl)diazenyl)benzaldehyde (0.1 mmol), methanol (10 ml), $Et_3N$ (0.3 mmol) was sealed in a glass vessel (20 ml, capacity) and the solution was heated at 363 K for 1 h under autogenous pressure. After the mixture was allowed to cool to room temperature, yellow block single crystals were isolated from the vessel.

## Refinement

The H atoms were placed in geometrically idealized positions (C—H = 0.95 Å and O—H = 0.82–0.84 Å), with $U_{iso}$(H) = 1.2Ueq(C) and $U_{iso}$(H) = 1.5Ueq(O).

## Figures

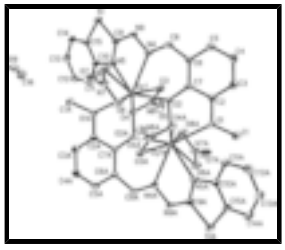

Fig. 1. The molecular structure of the title compound, with atom labels and 30% probability displacement ellipsoids. Hydrogen atoms are omitted for clarity.

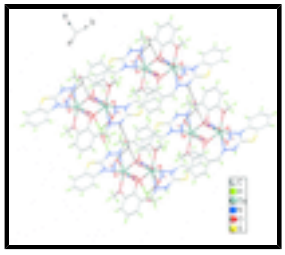

Fig. 2. Packing diagram of the title compound. Hydrogen bonds are shown as dashed lines.

**Bis(μ-2-{[2-(1,3-benzothiazol-2-yl)hydrazinylidene]methyl}-6- methoxyphenolato)bis[dinitratodysprosium(III)] methanol disolvate**

*Crystal data*

| | |
|---|---|
| $[Dy_2(C_{15}H_{12}N_3O_2S)_2(NO_3)_4]\cdot 2CH_4O$ | $Z = 1$ |
| $M_r = 1233.82$ | $F(000) = 602$ |
| Triclinic, $P\overline{1}$ | $D_x = 2.059$ Mg m$^{-3}$ |
| Hall symbol: -P 1 | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| $a = 9.6191$ (6) Å | Cell parameters from 3776 reflections |
| $b = 10.1002$ (7) Å | $\theta = 2.3$–$25.1°$ |
| $c = 11.6151$ (8) Å | $\mu = 3.92$ mm$^{-1}$ |
| $\alpha = 112.045$ (1)° | $T = 159$ K |
| $\beta = 105.065$ (1)° | Block, yellow |
| $\gamma = 93.154$ (1)° | 0.20 × 0.10 × 0.10 mm |
| $V = 995.23$ (12) Å$^3$ | |

*Data collection*

| | |
|---|---|
| Rigaku Saturn CCD area-detector diffractometer | 3502 independent reflections |
| Radiation source: rotating anode | 3162 reflections with $I > 2\sigma(I)$ |
| confocal | $R_{int} = 0.016$ |
| Detector resolution: 7.31 pixels mm$^{-1}$ | $\theta_{max} = 25.1°$, $\theta_{min} = 2.0°$ |
| ω and φ scans | $h = -11\rightarrow11$ |
| Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2002) | $k = -12\rightarrow7$ |
| $T_{min} = 0.631$, $T_{max} = 0.676$ | $l = -13\rightarrow13$ |
| 5083 measured reflections | |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full | Secondary atom site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)] = 0.025$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.057$ | H-atom parameters constrained |
| $S = 1.07$ | $w = 1/[\sigma^2(F_o^2) + (0.0289P)^2 + 0.127P]$ where $P = (F_o^2 + 2F_c^2)/3$ |

| | |
|---|---|
| 3502 reflections | $(\Delta/\sigma)_{max} = 0.001$ |
| 292 parameters | $\Delta\rho_{max} = 0.83$ e Å$^{-3}$ |
| 0 restraints | $\Delta\rho_{min} = -0.51$ e Å$^{-3}$ |

*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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.5488 (5) | −0.1481 (6) | 1.3423 (4) | 0.0419 (12) |
| H1A | 0.4829 | −0.0989 | 1.3895 | 0.063* |
| H1B | 0.6488 | −0.0946 | 1.3876 | 0.063* |
| H1C | 0.5453 | −0.2468 | 1.3387 | 0.063* |
| C2 | 0.3550 (4) | −0.2085 (4) | 1.1420 (4) | 0.0262 (9) |
| C3 | 0.2744 (5) | −0.3007 (5) | 1.1704 (4) | 0.0328 (10) |
| H3 | 0.3194 | −0.3323 | 1.2363 | 0.039* |
| C4 | 0.1277 (5) | −0.3481 (5) | 1.1034 (4) | 0.0349 (10) |
| H4 | 0.0711 | −0.4143 | 1.1208 | 0.042* |
| C5 | 0.0654 (4) | −0.2974 (5) | 1.0113 (4) | 0.0314 (10) |
| H5 | −0.0365 | −0.3263 | 0.9677 | 0.038* |
| C6 | 0.1455 (4) | −0.2054 (4) | 0.9789 (4) | 0.0259 (9) |
| C7 | 0.2972 (4) | −0.1615 (4) | 1.0421 (4) | 0.0237 (8) |
| C8 | 0.0613 (4) | −0.1613 (5) | 0.8805 (4) | 0.0308 (10) |
| H8 | −0.0395 | −0.2025 | 0.8423 | 0.037* |
| C9 | 0.0364 (4) | 0.0701 (5) | 0.7244 (4) | 0.0285 (9) |
| C10 | 0.1564 (4) | 0.2767 (4) | 0.7467 (4) | 0.0265 (9) |
| C11 | 0.2636 (4) | 0.3998 (5) | 0.7988 (4) | 0.0309 (10) |
| H11 | 0.3530 | 0.4075 | 0.8617 | 0.037* |
| C12 | 0.2378 (5) | 0.5099 (5) | 0.7573 (4) | 0.0335 (10) |
| H12 | 0.3112 | 0.5930 | 0.7904 | 0.040* |
| C13 | 0.1059 (5) | 0.5011 (5) | 0.6680 (4) | 0.0354 (10) |
| H13 | 0.0902 | 0.5791 | 0.6421 | 0.042* |
| C14 | −0.0026 (5) | 0.3818 (5) | 0.6163 (4) | 0.0335 (10) |
| H14 | −0.0930 | 0.3764 | 0.5557 | 0.040* |
| C15 | 0.0251 (4) | 0.2701 (5) | 0.6558 (4) | 0.0284 (9) |
| C16 | 0.2951 (6) | 0.3287 (6) | 0.4354 (5) | 0.0540 (14) |
| H16A | 0.2585 | 0.3862 | 0.3858 | 0.081* |
| H16C | 0.3978 | 0.3682 | 0.4862 | 0.081* |

| | | | | |
|---|---|---|---|---|
| H16B | 0.2374 | 0.3326 | 0.4945 | 0.081* |
| Dy1 | 0.358298 (18) | 0.05304 (2) | 0.890365 (17) | 0.02346 (8) |
| N4 | 0.1097 (3) | −0.0726 (4) | 0.8405 (3) | 0.0283 (8) |
| N5 | 0.1632 (3) | 0.1587 (4) | 0.7829 (3) | 0.0274 (8) |
| N6 | 0.3542 (4) | 0.3093 (4) | 1.1063 (3) | 0.0345 (9) |
| N7 | 0.3601 (4) | −0.1898 (4) | 0.6517 (3) | 0.0327 (8) |
| N9 | 0.0038 (3) | −0.0478 (4) | 0.7456 (3) | 0.0327 (8) |
| H9 | −0.0802 | −0.1070 | 0.7020 | 0.039* |
| O1 | 0.5034 (3) | −0.1543 (3) | 1.2102 (3) | 0.0291 (6) |
| O2 | 0.3863 (3) | −0.0823 (3) | 1.0113 (2) | 0.0249 (6) |
| O3 | 0.2717 (3) | 0.1897 (3) | 1.0724 (3) | 0.0340 (7) |
| O4 | 0.4335 (3) | 0.3079 (3) | 1.0330 (3) | 0.0337 (7) |
| O5 | 0.3585 (4) | 0.4152 (4) | 1.2012 (3) | 0.0574 (10) |
| O6 | 0.2613 (3) | −0.1127 (3) | 0.6575 (3) | 0.0371 (7) |
| O7 | 0.3620 (4) | −0.2850 (4) | 0.5512 (3) | 0.0580 (10) |
| O8 | 0.4579 (3) | −0.1600 (3) | 0.7589 (3) | 0.0416 (8) |
| O9 | 0.2828 (3) | 0.1836 (4) | 0.3486 (3) | 0.0452 (8) |
| H9A | 0.3398 | 0.1790 | 0.3038 | 0.068* |
| S1 | −0.09430 (11) | 0.11034 (12) | 0.61321 (10) | 0.0315 (2) |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.033 (2) | 0.062 (3) | 0.028 (2) | 0.002 (2) | 0.0030 (19) | 0.020 (2) |
| C2 | 0.021 (2) | 0.029 (2) | 0.027 (2) | 0.0045 (17) | 0.0058 (17) | 0.0110 (18) |
| C3 | 0.036 (2) | 0.029 (2) | 0.033 (2) | 0.006 (2) | 0.0075 (19) | 0.014 (2) |
| C4 | 0.033 (2) | 0.033 (2) | 0.041 (3) | 0.002 (2) | 0.011 (2) | 0.017 (2) |
| C5 | 0.022 (2) | 0.035 (2) | 0.034 (2) | 0.0027 (18) | 0.0075 (18) | 0.011 (2) |
| C6 | 0.0188 (19) | 0.028 (2) | 0.030 (2) | 0.0037 (17) | 0.0051 (17) | 0.0118 (18) |
| C7 | 0.0183 (19) | 0.022 (2) | 0.028 (2) | 0.0031 (16) | 0.0038 (16) | 0.0095 (18) |
| C8 | 0.016 (2) | 0.037 (3) | 0.033 (2) | 0.0030 (18) | 0.0006 (17) | 0.013 (2) |
| C9 | 0.019 (2) | 0.037 (2) | 0.028 (2) | 0.0086 (18) | 0.0036 (17) | 0.013 (2) |
| C10 | 0.023 (2) | 0.032 (2) | 0.024 (2) | 0.0070 (18) | 0.0049 (17) | 0.0117 (18) |
| C11 | 0.022 (2) | 0.039 (3) | 0.030 (2) | 0.0060 (19) | 0.0039 (17) | 0.014 (2) |
| C12 | 0.029 (2) | 0.035 (2) | 0.032 (2) | 0.003 (2) | 0.0046 (19) | 0.012 (2) |
| C13 | 0.039 (2) | 0.039 (3) | 0.031 (2) | 0.011 (2) | 0.006 (2) | 0.019 (2) |
| C14 | 0.031 (2) | 0.042 (3) | 0.028 (2) | 0.012 (2) | 0.0021 (19) | 0.019 (2) |
| C15 | 0.020 (2) | 0.038 (2) | 0.022 (2) | 0.0059 (18) | 0.0028 (16) | 0.0086 (19) |
| C16 | 0.043 (3) | 0.069 (4) | 0.044 (3) | 0.009 (3) | 0.011 (2) | 0.018 (3) |
| Dy1 | 0.01390 (10) | 0.03118 (12) | 0.02375 (11) | 0.00321 (8) | 0.00021 (7) | 0.01318 (9) |
| N4 | 0.0175 (16) | 0.033 (2) | 0.0301 (18) | −0.0005 (15) | −0.0019 (14) | 0.0156 (16) |
| N5 | 0.0207 (17) | 0.0310 (19) | 0.0281 (18) | 0.0054 (15) | 0.0046 (14) | 0.0110 (16) |
| N6 | 0.033 (2) | 0.039 (2) | 0.029 (2) | 0.0144 (18) | 0.0035 (17) | 0.0152 (19) |
| N7 | 0.0199 (18) | 0.036 (2) | 0.031 (2) | 0.0019 (16) | −0.0010 (15) | 0.0067 (18) |
| N9 | 0.0144 (16) | 0.045 (2) | 0.0347 (19) | −0.0018 (16) | −0.0072 (14) | 0.0229 (18) |
| O1 | 0.0195 (13) | 0.0415 (17) | 0.0277 (15) | 0.0034 (13) | 0.0010 (11) | 0.0199 (14) |
| O2 | 0.0146 (13) | 0.0316 (16) | 0.0279 (14) | 0.0016 (12) | 0.0007 (11) | 0.0156 (13) |
| O3 | 0.0291 (16) | 0.0430 (19) | 0.0312 (16) | 0.0091 (15) | 0.0063 (13) | 0.0177 (15) |

| | | | | | | |
|---|---|---|---|---|---|---|
| O4 | 0.0285 (15) | 0.0393 (18) | 0.0298 (15) | 0.0044 (14) | 0.0036 (13) | 0.0138 (14) |
| O5 | 0.086 (3) | 0.041 (2) | 0.041 (2) | 0.021 (2) | 0.0250 (19) | 0.0073 (18) |
| O6 | 0.0229 (15) | 0.0449 (19) | 0.0318 (16) | 0.0119 (14) | −0.0016 (13) | 0.0087 (15) |
| O7 | 0.0373 (19) | 0.066 (3) | 0.037 (2) | 0.0132 (18) | −0.0008 (16) | −0.0066 (19) |
| O8 | 0.0351 (17) | 0.0463 (19) | 0.0321 (16) | 0.0152 (15) | −0.0033 (14) | 0.0117 (15) |
| O9 | 0.0258 (17) | 0.057 (2) | 0.046 (2) | −0.0010 (16) | 0.0079 (14) | 0.0163 (18) |
| S1 | 0.0212 (5) | 0.0388 (6) | 0.0302 (5) | 0.0037 (5) | −0.0020 (4) | 0.0161 (5) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| C1—O1 | 1.459 (5) | C13—H13 | 0.9500 |
| C1—H1A | 0.9800 | C14—C15 | 1.384 (6) |
| C1—H1B | 0.9800 | C14—H14 | 0.9500 |
| C1—H1C | 0.9800 | C15—S1 | 1.752 (4) |
| C2—C3 | 1.364 (6) | C16—O9 | 1.410 (6) |
| C2—O1 | 1.402 (4) | C16—H16A | 0.9800 |
| C2—C7 | 1.410 (5) | C16—H16C | 0.9800 |
| C3—C4 | 1.379 (6) | C16—H16B | 0.9800 |
| C3—H3 | 0.9500 | Dy1—O2 | 2.280 (3) |
| C4—C5 | 1.370 (6) | Dy1—O2$^{i}$ | 2.374 (2) |
| C4—H4 | 0.9500 | Dy1—O1$^{i}$ | 2.394 (3) |
| C5—C6 | 1.390 (5) | Dy1—O4 | 2.422 (3) |
| C5—H5 | 0.9500 | Dy1—O3 | 2.433 (3) |
| C6—C7 | 1.412 (5) | Dy1—N4 | 2.461 (3) |
| C6—C8 | 1.451 (5) | Dy1—O6 | 2.471 (3) |
| C7—O2 | 1.342 (4) | Dy1—N5 | 2.494 (3) |
| C8—N4 | 1.270 (5) | Dy1—O8 | 2.530 (3) |
| C8—H8 | 0.9500 | Dy1—Dy1$^{i}$ | 3.7184 (4) |
| C9—N5 | 1.320 (5) | N4—N9 | 1.402 (4) |
| C9—N9 | 1.340 (5) | N6—O5 | 1.203 (5) |
| C9—S1 | 1.738 (4) | N6—O3 | 1.273 (5) |
| C10—C11 | 1.395 (6) | N6—O4 | 1.279 (4) |
| C10—C15 | 1.399 (5) | N7—O7 | 1.208 (5) |
| C10—N5 | 1.405 (5) | N7—O6 | 1.261 (4) |
| C11—C12 | 1.379 (6) | N7—O8 | 1.266 (4) |
| C11—H11 | 0.9500 | N9—H9 | 0.8800 |
| C12—C13 | 1.388 (6) | O1—Dy1$^{i}$ | 2.394 (3) |
| C12—H12 | 0.9500 | O2—Dy1$^{i}$ | 2.374 (2) |
| C13—C14 | 1.376 (6) | O9—H9A | 0.8400 |
| O1—C1—H1A | 109.5 | O4—Dy1—O3 | 52.63 (10) |
| O1—C1—H1B | 109.5 | O2—Dy1—N4 | 75.53 (10) |
| H1A—C1—H1B | 109.5 | O2$^{i}$—Dy1—N4 | 149.07 (10) |
| O1—C1—H1C | 109.5 | O1$^{i}$—Dy1—N4 | 138.80 (10) |
| H1A—C1—H1C | 109.5 | O4—Dy1—N4 | 121.11 (11) |
| H1B—C1—H1C | 109.5 | O3—Dy1—N4 | 74.17 (11) |
| C3—C2—O1 | 122.0 (4) | O2—Dy1—O6 | 108.45 (10) |
| C3—C2—C7 | 123.2 (4) | O2$^{i}$—Dy1—O6 | 116.45 (9) |

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| O1—C2—C7 | 114.8 (3) | $O1^{i}$—Dy1—O6 | 76.95 (10) |
| C2—C3—C4 | 120.1 (4) | O4—Dy1—O6 | 141.79 (10) |
| C2—C3—H3 | 120.0 | O3—Dy1—O6 | 139.92 (10) |
| C4—C3—H3 | 120.0 | N4—Dy1—O6 | 69.16 (10) |
| C5—C4—C3 | 118.5 (4) | O2—Dy1—N5 | 139.17 (10) |
| C5—C4—H4 | 120.8 | $O2^{i}$—Dy1—N5 | 144.17 (10) |
| C3—C4—H4 | 120.8 | $O1^{i}$—Dy1—N5 | 81.46 (10) |
| C4—C5—C6 | 122.6 (4) | O4—Dy1—N5 | 79.22 (10) |
| C4—C5—H5 | 118.7 | O3—Dy1—N5 | 79.08 (10) |
| C6—C5—H5 | 118.7 | N4—Dy1—N5 | 66.52 (11) |
| C5—C6—C7 | 119.6 (4) | O6—Dy1—N5 | 72.05 (10) |
| C5—C6—C8 | 115.0 (4) | O2—Dy1—O8 | 77.32 (10) |
| C7—C6—C8 | 125.3 (4) | $O2^{i}$—Dy1—O8 | 70.22 (9) |
| O2—C7—C2 | 119.8 (3) | $O1^{i}$—Dy1—O8 | 76.42 (10) |
| O2—C7—C6 | 124.3 (3) | O4—Dy1—O8 | 140.65 (10) |
| C2—C7—C6 | 115.8 (3) | O3—Dy1—O8 | 153.86 (10) |
| N4—C8—C6 | 126.1 (4) | N4—Dy1—O8 | 98.23 (11) |
| N4—C8—H8 | 117.0 | O6—Dy1—O8 | 50.52 (9) |
| C6—C8—H8 | 117.0 | N5—Dy1—O8 | 121.51 (10) |
| N5—C9—N9 | 123.9 (4) | O2—Dy1—$Dy1^{i}$ | 37.86 (6) |
| N5—C9—S1 | 117.3 (3) | $O2^{i}$—Dy1—$Dy1^{i}$ | 36.10 (6) |
| N9—C9—S1 | 118.8 (3) | $O1^{i}$—Dy1—$Dy1^{i}$ | 103.10 (6) |
| C11—C10—C15 | 119.0 (4) | O4—Dy1—$Dy1^{i}$ | 92.27 (7) |
| C11—C10—N5 | 125.8 (3) | O3—Dy1—$Dy1^{i}$ | 90.23 (7) |
| C15—C10—N5 | 115.2 (4) | N4—Dy1—$Dy1^{i}$ | 113.24 (8) |
| C12—C11—C10 | 118.9 (4) | O6—Dy1—$Dy1^{i}$ | 118.59 (7) |
| C12—C11—H11 | 120.6 | N5—Dy1—$Dy1^{i}$ | 169.00 (8) |
| C10—C11—H11 | 120.6 | O8—Dy1—$Dy1^{i}$ | 69.47 (6) |
| C11—C12—C13 | 120.9 (4) | C8—N4—N9 | 114.2 (3) |
| C11—C12—H12 | 119.5 | C8—N4—Dy1 | 131.6 (3) |
| C13—C12—H12 | 119.5 | N9—N4—Dy1 | 114.1 (2) |
| C14—C13—C12 | 121.5 (4) | C9—N5—C10 | 109.1 (3) |
| C14—C13—H13 | 119.3 | C9—N5—Dy1 | 113.1 (3) |
| C12—C13—H13 | 119.3 | C10—N5—Dy1 | 136.6 (2) |
| C13—C14—C15 | 117.4 (4) | O5—N6—O3 | 122.4 (4) |
| C13—C14—H14 | 121.3 | O5—N6—O4 | 122.6 (4) |
| C15—C14—H14 | 121.3 | O3—N6—O4 | 115.0 (3) |
| C14—C15—C10 | 122.3 (4) | O7—N7—O6 | 122.5 (4) |
| C14—C15—S1 | 127.6 (3) | O7—N7—O8 | 122.2 (4) |
| C10—C15—S1 | 109.9 (3) | O6—N7—O8 | 115.2 (3) |
| O9—C16—H16A | 109.5 | C9—N9—N4 | 117.0 (3) |
| O9—C16—H16C | 109.5 | C9—N9—H9 | 121.5 |
| H16A—C16—H16C | 109.5 | N4—N9—H9 | 121.5 |
| O9—C16—H16B | 109.5 | C2—O1—C1 | 114.9 (3) |
| H16A—C16—H16B | 109.5 | C2—O1—$Dy1^{i}$ | 116.7 (2) |
| H16C—C16—H16B | 109.5 | C1—O1—$Dy1^{i}$ | 127.7 (2) |

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|---|---|---|---|
| O2—Dy1—O2$^{i}$ | 73.96 (10) | C7—O2—Dy1 | 136.2 (2) |
| O2—Dy1—O1$^{i}$ | 139.27 (9) | C7—O2—Dy1$^{i}$ | 117.7 (2) |
| O2$^{i}$—Dy1—O1$^{i}$ | 68.12 (9) | Dy1—O2—Dy1$^{i}$ | 106.04 (10) |
| O2—Dy1—O4 | 109.76 (9) | N6—O3—Dy1 | 95.9 (2) |
| O2$^{i}$—Dy1—O4 | 74.78 (9) | N6—O4—Dy1 | 96.3 (2) |
| O1$^{i}$—Dy1—O4 | 74.29 (10) | N7—O6—Dy1 | 98.6 (2) |
| O2—Dy1—O3 | 76.56 (10) | N7—O8—Dy1 | 95.6 (2) |
| O2$^{i}$—Dy1—O3 | 103.27 (9) | C16—O9—H9A | 109.5 |
| O1$^{i}$—Dy1—O3 | 125.83 (10) | C9—S1—C15 | 88.36 (19) |
| O1—C2—C3—C4 | 178.1 (4) | C8—N4—N9—C9 | 162.8 (4) |
| C7—C2—C3—C4 | −2.5 (7) | Dy1—N4—N9—C9 | −20.4 (4) |
| C2—C3—C4—C5 | −1.7 (6) | C3—C2—O1—C1 | −25.7 (5) |
| C3—C4—C5—C6 | 2.8 (7) | C7—C2—O1—C1 | 154.9 (4) |
| C4—C5—C6—C7 | 0.2 (6) | C3—C2—O1—Dy1$^{i}$ | 162.7 (3) |
| C4—C5—C6—C8 | −179.3 (4) | C7—C2—O1—Dy1$^{i}$ | −16.7 (4) |
| C3—C2—C7—O2 | −173.5 (4) | C2—C7—O2—Dy1 | −167.5 (3) |
| O1—C2—C7—O2 | 6.0 (5) | C6—C7—O2—Dy1 | 13.8 (6) |
| C3—C2—C7—C6 | 5.3 (6) | C2—C7—O2—Dy1$^{i}$ | 7.9 (5) |
| O1—C2—C7—C6 | −175.2 (3) | C6—C7—O2—Dy1$^{i}$ | −170.8 (3) |
| C5—C6—C7—O2 | 174.7 (4) | O2$^{i}$—Dy1—O2—C7 | 175.7 (4) |
| C8—C6—C7—O2 | −6.0 (6) | O1$^{i}$—Dy1—O2—C7 | −162.4 (3) |
| C5—C6—C7—C2 | −4.1 (6) | O4—Dy1—O2—C7 | 108.9 (3) |
| C8—C6—C7—C2 | 175.3 (4) | O3—Dy1—O2—C7 | 67.4 (3) |
| C5—C6—C8—N4 | 176.4 (4) | N4—Dy1—O2—C7 | −9.4 (3) |
| C7—C6—C8—N4 | −3.0 (7) | O6—Dy1—O2—C7 | −71.2 (3) |
| C15—C10—C11—C12 | −1.1 (6) | N5—Dy1—O2—C7 | 12.5 (4) |
| N5—C10—C11—C12 | −176.9 (4) | O8—Dy1—O2—C7 | −111.5 (3) |
| C10—C11—C12—C13 | 1.8 (6) | Dy1$^{i}$—Dy1—O2—C7 | 175.7 (4) |
| C11—C12—C13—C14 | −1.0 (7) | O2$^{i}$—Dy1—O2—Dy1$^{i}$ | 0.0 |
| C12—C13—C14—C15 | −0.5 (6) | O1$^{i}$—Dy1—O2—Dy1$^{i}$ | 21.83 (19) |
| C13—C14—C15—C10 | 1.2 (6) | O4—Dy1—O2—Dy1$^{i}$ | −66.82 (12) |
| C13—C14—C15—S1 | 176.6 (3) | O3—Dy1—O2—Dy1$^{i}$ | −108.32 (12) |
| C11—C10—C15—C14 | −0.5 (6) | N4—Dy1—O2—Dy1$^{i}$ | 174.87 (13) |
| N5—C10—C15—C14 | 175.8 (4) | O6—Dy1—O2—Dy1$^{i}$ | 113.12 (11) |
| C11—C10—C15—S1 | −176.5 (3) | N5—Dy1—O2—Dy1$^{i}$ | −163.24 (12) |
| N5—C10—C15—S1 | −0.2 (4) | O8—Dy1—O2—Dy1$^{i}$ | 72.77 (11) |
| C6—C8—N4—N9 | −179.2 (4) | O5—N6—O3—Dy1 | −175.9 (4) |
| C6—C8—N4—Dy1 | 4.8 (7) | O4—N6—O3—Dy1 | 3.2 (3) |
| O2—Dy1—N4—C8 | 0.1 (4) | O2—Dy1—O3—N6 | 126.4 (2) |
| O2$^{i}$—Dy1—N4—C8 | 9.7 (5) | O2$^{i}$—Dy1—O3—N6 | 56.8 (2) |
| O1$^{i}$—Dy1—N4—C8 | 153.4 (3) | O1$^{i}$—Dy1—O3—N6 | −15.6 (2) |
| O4—Dy1—N4—C8 | −104.5 (4) | O4—Dy1—O3—N6 | −1.92 (19) |
| O3—Dy1—N4—C8 | −79.8 (4) | N4—Dy1—O3—N6 | −155.1 (2) |
| O6—Dy1—N4—C8 | 116.7 (4) | O6—Dy1—O3—N6 | −130.8 (2) |

| | | | |
|---|---|---|---|
| N5—Dy1—N4—C8 | −164.5 (4) | N5—Dy1—O3—N6 | −86.6 (2) |
| O8—Dy1—N4—C8 | 74.6 (4) | O8—Dy1—O3—N6 | 128.8 (3) |
| Dy1$^{i}$—Dy1—N4—C8 | 3.5 (4) | Dy1$^{i}$—Dy1—O3—N6 | 90.8 (2) |
| O2—Dy1—N4—N9 | −176.0 (3) | O5—N6—O4—Dy1 | 175.9 (4) |
| O2$^{i}$—Dy1—N4—N9 | −166.4 (2) | O3—N6—O4—Dy1 | −3.2 (3) |
| O1$^{i}$—Dy1—N4—N9 | −22.7 (3) | O2—Dy1—O4—N6 | −52.3 (2) |
| O4—Dy1—N4—N9 | 79.4 (3) | O2$^{i}$—Dy1—O4—N6 | −118.6 (2) |
| O3—Dy1—N4—N9 | 104.2 (3) | O1$^{i}$—Dy1—O4—N6 | 170.4 (2) |
| O6—Dy1—N4—N9 | −59.4 (3) | O3—Dy1—O4—N6 | 1.91 (19) |
| N5—Dy1—N4—N9 | 19.4 (2) | N4—Dy1—O4—N6 | 32.4 (2) |
| O8—Dy1—N4—N9 | −101.4 (3) | O6—Dy1—O4—N6 | 127.8 (2) |
| Dy1$^{i}$—Dy1—N4—N9 | −172.6 (2) | N5—Dy1—O4—N6 | 86.3 (2) |
| N9—C9—N5—C10 | −175.2 (4) | O8—Dy1—O4—N6 | −146.3 (2) |
| S1—C9—N5—C10 | 5.1 (4) | Dy1$^{i}$—Dy1—O4—N6 | −86.6 (2) |
| N9—C9—N5—Dy1 | 15.4 (5) | O7—N7—O6—Dy1 | −176.6 (4) |
| S1—C9—N5—Dy1 | −164.29 (19) | O8—N7—O6—Dy1 | 3.1 (4) |
| C11—C10—N5—C9 | 173.0 (4) | O2—Dy1—O6—N7 | −56.8 (2) |
| C15—C10—N5—C9 | −3.0 (5) | O2$^{i}$—Dy1—O6—N7 | 24.1 (3) |
| C11—C10—N5—Dy1 | −21.3 (6) | O1$^{i}$—Dy1—O6—N7 | 81.2 (2) |
| C15—C10—N5—Dy1 | 162.7 (3) | O4—Dy1—O6—N7 | 123.1 (2) |
| O2—Dy1—N5—C9 | −40.9 (3) | O3—Dy1—O6—N7 | −147.7 (2) |
| O2$^{i}$—Dy1—N5—C9 | 167.3 (2) | N4—Dy1—O6—N7 | −122.6 (2) |
| O1$^{i}$—Dy1—N5—C9 | 135.7 (3) | N5—Dy1—O6—N7 | 166.3 (3) |
| O4—Dy1—N5—C9 | −148.7 (3) | O8—Dy1—O6—N7 | −1.8 (2) |
| O3—Dy1—N5—C9 | −95.1 (3) | Dy1$^{i}$—Dy1—O6—N7 | −16.8 (2) |
| N4—Dy1—N5—C9 | −17.7 (3) | O7—N7—O8—Dy1 | 176.7 (4) |
| O6—Dy1—N5—C9 | 56.8 (3) | O6—N7—O8—Dy1 | −3.0 (4) |
| O8—Dy1—N5—C9 | 67.5 (3) | O2—Dy1—O8—N7 | 129.1 (2) |
| Dy1$^{i}$—Dy1—N5—C9 | −108.9 (4) | O2$^{i}$—Dy1—O8—N7 | −153.6 (3) |
| O2—Dy1—N5—C10 | 153.8 (3) | O1$^{i}$—Dy1—O8—N7 | −82.3 (2) |
| O2$^{i}$—Dy1—N5—C10 | 2.0 (5) | O4—Dy1—O8—N7 | −125.1 (2) |
| O1$^{i}$—Dy1—N5—C10 | −29.6 (4) | O3—Dy1—O8—N7 | 126.7 (3) |
| O4—Dy1—N5—C10 | 46.0 (4) | N4—Dy1—O8—N7 | 56.0 (2) |
| O3—Dy1—N5—C10 | 99.6 (4) | O6—Dy1—O8—N7 | 1.8 (2) |
| N4—Dy1—N5—C10 | 177.0 (4) | N5—Dy1—O8—N7 | −11.4 (3) |
| O6—Dy1—N5—C10 | −108.5 (4) | Dy1$^{i}$—Dy1—O8—N7 | 167.8 (3) |
| O8—Dy1—N5—C10 | −97.8 (4) | N5—C9—S1—C15 | −4.6 (3) |
| Dy1$^{i}$—Dy1—N5—C10 | 85.8 (5) | N9—C9—S1—C15 | 175.7 (4) |
| N5—C9—N9—N4 | 3.2 (6) | C14—C15—S1—C9 | −173.3 (4) |
| S1—C9—N9—N4 | −177.1 (3) | C10—C15—S1—C9 | 2.4 (3) |

Symmetry codes: (i) −*x*+1, −*y*, −*z*+2.

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|

| | | | | |
|---|---|---|---|---|
| O9—H9A···O3$^{ii}$ | 0.84 | 2.64 | 3.206 (4) | 126 |
| O9—H9A···O8$^{iii}$ | 0.84 | 2.24 | 3.049 (4) | 161 |
| N9—H9···O9$^{iv}$ | 0.88 | 1.91 | 2.751 (4) | 160 |

Symmetry codes: (ii) *x*, *y*, *z*−1; (iii) −*x*+1, −*y*, −*z*+1; (iv) −*x*, −*y*, −*z*+1.

**Fig. 1**

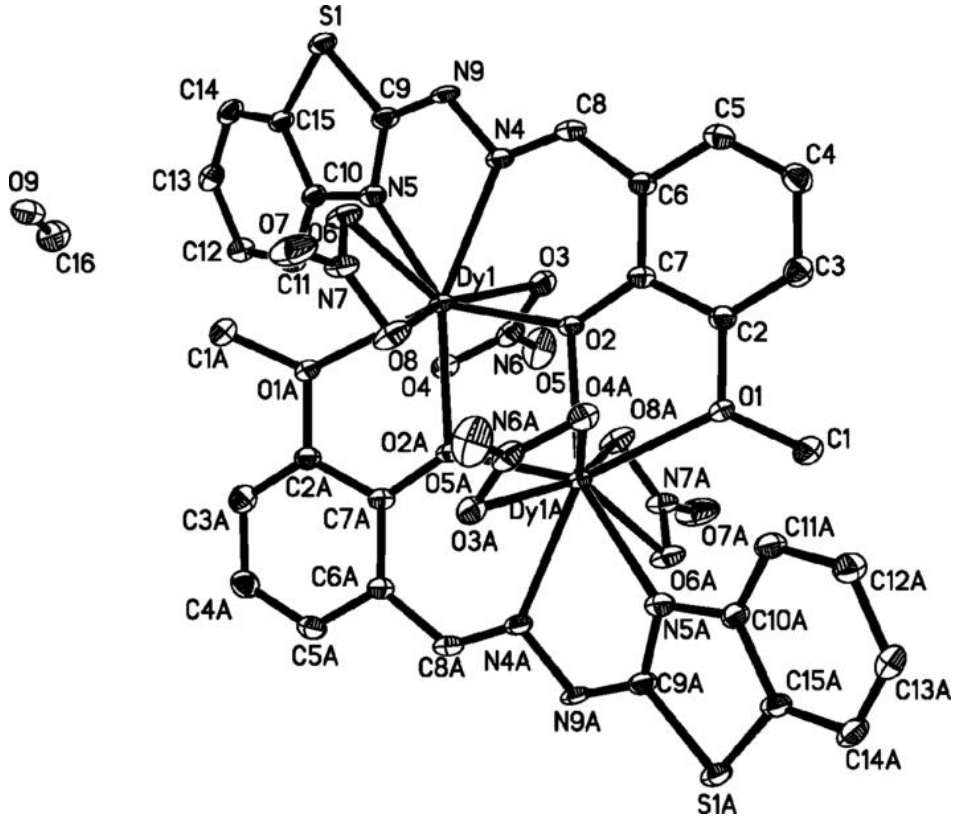
S1
C14
C9
N9
C15
N4
C8
C5
C4
C13
C10
N5
C6
O9
O7
O6
C12
C16
C11
N7
Dy1
O3
C7
C3
C1A
O8
O2
C2
N6
O4
O5
O4A
O1A
O8A
O1
N6A
O2A
C1
O5A
N7A
C2A
Dy1A
C3A
C7A
O3A
O7A
C11A
C6A
O6A
C12A
C4A
N5A
C10A
C5A
C8A
N4A
C9A
C15A
C13A
N9A
C14A
S1A

**Fig. 2**