Bis[$\mu_3$-2-(pyridin-3-yl)acetato-$\kappa^3$O:O'O']bis[$\mu_2$-2-(pyridin-3-yl)acetato-$\kappa^2$O'O']bis[chlorido(1,10-phenanthroline-$\kappa^2$N,N')dysprosium(III)]

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The title Dy$^{III}$ complex, [Dy$_2$(C$_7$H$_6$NO$_2$)$_4$Cl$_2$(C$_{12}$H$_8$N$_2$)$_2$] or [Dy$_2$(\mu$_3$-PAA)$_4$-\(\text{Cl})_2(\text{phen})_2\] (PAA = 3-pyridylacetate, phen = 1,10-phenanthroline), obtained by reaction of Dy(ClO$_4$)$_3$, 3-pyridylacetic acid ligands and 1,10-phenanthroline, exhibits a dinuclear structure. Adjacent binuclear dimers are further connected via face-to-face $\pi$-$\pi$ stacking interactions resulting in supramolecular chains along the $c$-axis direction.

Structure description

Coordination complexes composed of metal cations and organic ligands have received much attention because of their diverse structures and intriguing properties such as photoluminescence, magnetism, proton conduction and so on. Lanthanide ions are considered to be excellent metal ions for the construction of such systems because of their unique 4f electrons and can show remarkable photoluminescent, magnetic and catalytic properties. Among numerous ligands, pyridinecarboxylate ligands bearing O and N coordination atoms have attracted considerable interest and have proved to be a class of excellent bridging linkers in fabricating metal coordination complexes with appealing structures. The 3-pyridylacetate ligand (3-PAA), one of the most simple pyridinecarboxylate ligands, has attracted considerable interest owing to its strong coordination and varied coordination modes, resulting in diverse structures with excellent properties. So far, coordination complexes constructed by the 3-PAA ligand have focused on transition-metal cations, but lanthanide complexes based on the 3-PAA ligand are still rare. Thus, in this work, we prepared the title compound [Dy$_2$(\mu$_2$-PAA)$_4$\(\text{Cl})_2(\text{phen})_2\] (3-PAA = 3-pyridylacetate, phen = 1,10-phenanthroline), which displays a dinuclear structure.
The asymmetric unit of 1 (Fig. 1) consists of one crystallographically independent Dy$^{III}$ ion, one Cl$^-$/O$^-$ anion, two PAA ligands and one phen molecule. The Dy$^{III}$ cation is eight-coordinated by five carboxylate oxygen atoms from four different PAA$^-$/O$^-$ ligands, one Cl$^-$ ion, and two nitrogen atoms from one chelating phen molecule. The Dy–O bond lengths range from 2.3069 (17) to 2.5170 (15) Å, and the Dy–N bond distances are 2.5386 (18) and 2.5516 (17) Å, which are similar to those in the complex [Zn(L2)(Cl2)Dy(NO3)2]$\cdot$H2O{L = N,N’-dimethyl-N,N’-bis(2-hydroxy-3-formyl-5-bromobenzyl, dicl = deprotonated diclofenac = 2-[(2,6-dichlorophenyl)amino] benzene acetate; Echenique-Errandonea et al., 2019]. The two PAA$^-$ ligands exhibit two different coordination modes. One acts as a tridentate ligand with a $\mu_2$-$\eta^2$1-O:2-O coordination mode, while the other serves as a bidentate ligand with a $\mu_2$-$\kappa^1O^-$:2:O$^-$ coordination mode. It is worth emphasizing that the N atom of the PAA ligand is noncoordinating in 1. As shown in Fig. 2, two neighboring Dy$^{III}$ ions are linked by four bridging carboxyl groups of four PAA$^-$ ligands, forming the binuclear structure of 1, in which the nearest Dy$^{III}$-Dy$^{III}$ separation is 3.8976 (19) Å. These adjacent binuclear dimers are further connected via face-to-face $\pi$–$\pi$ stacking interactions involving the phenyl and pyridine rings of the phen ligands, the centroid-to-centroid distance being 3.6116 (10) Å, leading to the formation of supramolecular chains along the c-axis direction (Fig. 3). For background information on the lanthanide ions and the 3-pyridylacetic acid ligand, see: Chakraborty et al. (2021); Xin et al. (2019); Ma et al. (2020); Wang et al. (2011); Teo et al. (2009); Adams et al. (2006).

![Figure 1](image1.png)

Figure 1
The asymmetric unit of 1 with 40% probability displacement ellipsoids. H atoms are omitted for clarity. Symmetry code: (A) 1 – x, 1 – y, 2 – z.

![Figure 2](image2.png)

Figure 2
The dinuclear structure of 1. H atoms are omitted for clarity. Symmetry code: (A) 1 – x, 1 – y, 2 – z.

![Figure 3](image3.png)

Figure 3
The supramolecular chain along the c-axis direction formed by face-to-face $\pi$–$\pi$ stacking interactions.

### Table 1
Experimental details.

| Property | Value |
|----------|-------|
| Chemical formula | [Dy$_2$(C$_7$H$_6$NO$_2$)$_4$Cl$_2$(C$_{12}$H$_8$N$_2$)$_2$] |
| $M_r$ | 1300.82 |
| Crystal system, space group | Monoclinic, $P2_1/c$ |
| Temperature (K) | 296 |
| $a$, $b$, $c$ (Å) | 8.8922 (1), 21.5425 (3), 12.9887 (1) |
| $\beta$ (°) | 101.755 (1) |
| $V$ (Å$^3$) | 2435.94 (5) |
| $Z$ | 2 |
| Radiation type | Mo Kα |
| $\mu$ (mm$^{-1}$) | 3.22 |
| Crystal size (mm) | 0.22 × 0.20 × 0.19 |
| Data collection | Bruker SAINT CCD area detector Multi-scan (SADABS; Bruker, 2008) |
| T$_{\text{min}}$, T$_{\text{max}}$ | 0.626, 0.746 |
| No. of measured, independent and observed $|I > 2\sigma(I)|$ reflections | 16420, 4487, 3954 |
| $R_{\text{int}}$ | 0.024 |
| $\langle \sin \theta/\lambda \rangle_{\text{max}}$ (Å$^{-1}$) | 0.606 |
| Refinement | $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ |
| No. of reflections | 4487 |
| No. of parameters | 325 |
| No. of restraints | 1 |
| H-atom treatment | H-atom parameters constrained |
| $\Delta \rho_{\text{max}}$, $\Delta \rho_{\text{min}}$ (e Å$^{-3}$) | 0.59, –0.34 |

Computer programs: SMART and SAINT (Bruker, 2008), SHELXS97 (Sheldrick, 2008), and SHELXTL (Sheldrick, 2008).
Synthesis and crystallization

Dy(ClO₄)₃ (0.2 mmol), 3-pyridylacetic acid (3-PAA, 0.25 mmol), 1,10-phenanthroline (0.25 mmol), HCl (0.25 mmol) and Et₃N were dissolved in 5 mL of acetonitrile and then sealed into a 25 mL Teflon-lined stainless steel vessel. The vessel was kept at 433 K for 3 d under autogenous pressure and then cooled to room temperature at a rate of 5.63 K h⁻¹. Colorless block-shaped crystals were obtained by filtration of the resulting solution. Yield based on Dy: 38%.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. The instruction "delu 0.002 0.001 C11 C12" was used during the refinement to limit the displacement parameters of the specified atoms.

Funding information

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References

Adams, C. J., Crawford, P. C., Guy Orpen, A. & Podesta, T. J. (2006). Dalton Trans. pp. 4078–4092.
Bruker (2008). SMART, SAINT and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.
Chakraborty, G., Park, I.-H., Medishetty, R. & Vittal, J. J. (2021). Chem. Rev. 121, 3751–3891.
Echenique-Errandonea, E., Zabala-Lekuona, A., Cepeda, J., Rodríguez-Diéguez, A., Seco, J. M., Oyarzabal, I. & Colacio, E. (2019). Dalton Trans. 48, 190–201.
Ma, Y. J., Hu, J. X., Han, S. D., Pan, J., Li, J. H. & Wang, G. M. (2020). J. Am. Chem. Soc. 142, 2682–2689.
Sheldrick, G. M. (2008). Acta Cryst. A64, 112–122.
Teo, P., Koh, L. L. & Hor, T. S. A. (2009). Dalton Trans. pp. 5637–5646.
Wang, L., Yin, X. H., Hao, H. J., Lin, W., Tan, X. H. & Lin, C. W. (2011). Z. Kristallogr. New. Cryst. Struct. pp. 219-220.
Xin, Y., Wang, J. H., Zychowicz, M., Zakrzewski, J. J., Nakabayashi, K., Sieklucka, B., Chorazy, S. & Ohtkoshi, S. (2019). J. Am. Chem. Soc. 141, 18211–18220.
**full crystallographic data**

*IUCrData* (2022). 7, x220231  [https://doi.org/10.1107/S2414314622002310]

Bis[µ₃-2-(pyridin-3-yl)acetato-κ³O:O’:O’']bis[µ₂-2-(pyridin-3-yl)acetato-κ²O:O’']bis[chlorido(1,10-phenanthroline-κ²N,N’)dysprosium(III)]

Yan Lin and Jian-Ping Yu

\[\text{Bis[µ₃-2-(pyridin-3-yl)acetato-κ³O:O’:O’’] \text{bis[µ₂-2-(pyridin-3-yl)acetato-κ²O:O’’]bis[chlorido(1,10-phenanthroline-κ²N,N’)dysprosium(III)]},}\]

**Crystal data**

\[\text{[Dy₂(C₇H₆NO₂)₄Cl₂(C₁₂H₈N₂)₂]}\]

\[M_r = 1300.82\]

Monoclinic, \(P\text{₂₁/c}\)

\(a = 8.8922\) (1) Å

\(b = 21.5425\) (3) Å

\(c = 12.9887\) (1) Å

\(β = 101.755\) (1)°

\(V = 2435.94\) (5) Å³

\(Z = 2\)

**Data collection**

Bruker SAINT CCD area detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

\(\phi\) and \(ω\) scans

Absorption correction: multi-scan

(SADABS; Bruker, 2008)

\(T_{\text{min}} = 0.626, T_{\text{max}} = 0.746\)

16420 measured reflections

4487 independent reflections

3954 reflections with \(I > 2\sigma(I)\)

\(R_{\text{int}} = 0.024\)

\(θ_{\text{max}} = 25.5°, θ_{\text{min}} = 1.9°\)

\(h = -10→10\)

\(k = -26→23\)

\(l = -15→15\)

**Refinement**

Refinement on \(F^2\)

Least-squares matrix: full

\(R(F^2 > 2σ(F^2)) = 0.021\)

\(wR(F^2) = 0.056\)

\(S = 1.00\)

4487 reflections

325 parameters

1 restraint

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

\(w = 1/[σ(F^2) + (0.035P)^2]\)

\(\text{where } P = (F^2 + 2F'O^2)/3\)

\((Δ/σ)_{\text{max}} = 0.002\)

\(Δρ_{\text{max}} = 0.59\) e Å⁻³

\(Δρ_{\text{min}} = -0.34\) e Å⁻³

\(F(000) = 1276\)

\(D_x = 1.773\) Mg m⁻³

Mo \(Kα\) radiation, \(λ = 0.71073\) Å

Cell parameters from 7687 reflections

\(\mu = 3.22\) mm⁻¹

\(T = 296\) K

Block, yellow

0.22 × 0.20 × 0.19 mm
Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\sigma(F^2)$ 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.

H atoms were placed in calculated positions with C—H = 0.93 Å in phenyl and pyridine rings while C—H = 0.97 Å in CH$_2$ groups and refined in riding mode with $U_{iso}$(H) = 1.2$U_{eq}$(C).

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)

|    | x               | y               | z               | $U_{iso}*/U_{eq}$ |
|----|----------------|----------------|----------------|------------------|
| Dy1 | 0.387207 (12) | 0.510416 (5)   | 0.855827 (7)   | 0.03127 (3)      |
| Cl1 | 0.13833 (7)   | 0.58009 (3)    | 0.81071 (5)    | 0.05938 (19)     |
| O3  | 0.57734 (18)  | 0.58550 (8)    | 0.90135 (12)   | 0.0487 (5)        |
| N3  | 0.4153 (2)    | 0.55350 (10)   | 0.67882 (13)   | 0.0420 (5)        |
| O1  | 0.57990 (18)  | 0.44419 (8)    | 0.80320 (11)   | 0.0472 (4)        |
| N4  | 0.2364 (2)    | 0.45183 (9)    | 0.69718 (13)   | 0.0383 (5)        |
| C7  | 0.6642 (3)    | 0.43988 (11)   | 0.89307 (17)   | 0.0365 (6)        |
| C14 | 0.6799 (3)    | 0.60069 (12)   | 0.97945 (18)   | 0.0455 (6)        |
| C18 | 0.3415 (3)    | 0.54877 (14)   | 0.48832 (18)   | 0.0544 (7)        |
| C26 | 0.3341 (3)    | 0.52662 (12)   | 0.58975 (17)   | 0.0416 (6)        |
| C25 | 0.2402 (3)    | 0.47369 (12)   | 0.59934 (17)   | 0.0420 (6)        |
| C21 | 0.1571 (3)    | 0.44537 (15)   | 0.50734 (18)   | 0.0545 (8)        |
| C6  | 0.8200 (3)    | 0.41057 (12)   | 0.90869 (18)   | 0.0441 (6)        |
| H6A | 0.8755        | 0.4280         | 0.8585         | 0.053*            |
| H6B | 0.8770        | 0.4202         | 0.9788         | 0.053*            |
| C22 | 0.1525 (3)    | 0.40185 (12)   | 0.7037 (2)     | 0.0498 (7)        |
| H24A| 0.1480        | 0.3871         | 0.7702         | 0.060*            |
| C5  | 0.8108 (3)    | 0.34153 (13)   | 0.8952 (2)     | 0.0476 (7)        |
| C19 | 0.2538 (4)    | 0.51931 (16)   | 0.3983 (2)     | 0.0687 (10)       |
| H19A| 0.2583        | 0.5344         | 0.3319         | 0.082*            |
| C22 | 0.0729 (3)    | 0.39253 (16)   | 0.5185 (2)     | 0.0696 (9)        |
| C12 | 0.3724        | 0.69858 (12)   | 0.8813 (2)     | 0.0494 (7)        |
| C8  | 0.7993 (3)    | 0.70351 (14)   | 0.7954 (2)     | 0.0662 (9)        |
| H8A | 0.8829        | 0.6781         | 0.7929         | 0.079*            |
| C11 | 0.6079 (3)    | 0.73727 (15)   | 0.8813 (2)     | 0.0690 (9)        |
| H11A| 0.5574        | 0.7359         | 0.9374         | 0.083*            |
| C23 | 0.0701 (3)    | 0.36993 (15)   | 0.6167 (2)     | 0.0657 (9)        |
| H23A| 0.0149        | 0.3343         | 0.6252         | 0.079*            |
| N2  | 0.7518 (3)    | 0.74312 (14)   | 0.7148 (2)     | 0.0894 (9)        |
| C17 | 0.4394 (4)    | 0.59840 (15)   | 0.4816 (2)     | 0.0658 (8)        |
| H17A| 0.4493        | 0.6133         | 0.4161         | 0.079*            |
| C13 | 0.7883 (3)    | 0.65317 (14)   | 0.9671 (2)     | 0.0682 (9)        |
| Atom  | x    | y    | z    | U11  | U22  | U33  |
|-------|------|------|------|------|------|------|
| H13A  | 0.8129 | 0.6757 | 1.0331 | 0.082* |
| H13B  | 0.8831 | 0.6352 | 0.9547 | 0.082* |
| O2    | 0.61601 (17) | 0.46036 (8) | 0.97223 (11) | 0.0421 (4) |
| O4    | 0.70410 (19) | 0.57482 (8) | 1.06850 (12) | 0.0509 (5) |
| C15   | 0.5041 (3) | 0.60095 (13) | 0.6677 (2) | 0.0565 (8) |
| H15A  | 0.5597 | 0.6197 | 0.7281 | 0.068* |
| C20   | 0.1653 (4) | 0.47066 (18) | 0.4064 (2) | 0.0720 (10) |
| H20A  | 0.1078 | 0.4527 | 0.3458 | 0.086* |
| C16   | 0.5196 (4) | 0.62483 (14) | 0.5697 (2) | 0.0690 (9) |
| H16A  | 0.5842 | 0.6583 | 0.5659 | 0.083* |
| C1    | 0.7804 (4) | 0.30426 (16) | 0.9734 (3) | 0.0882 (12) |
| H1A   | 0.7679 | 0.3235 | 1.0352 | 0.106* |
| C10   | 0.5589 (4) | 0.77733 (16) | 0.8009 (3) | 0.0875 (12) |
| H10A  | 0.4759 | 0.8035 | 0.8011 | 0.105* |
| C4    | 0.8326 (4) | 0.31262 (18) | 0.8051 (3) | 0.0958 (12) |
| H4A   | 0.8526 | 0.3356 | 0.7487 | 0.115* |
| N1    | 0.7671 (5) | 0.24248 (16) | 0.9688 (3) | 0.1415 (16) |
| C3    | 0.8239 (5) | 0.2480 (2) | 0.8001 (4) | 0.1398 (18) |
| H3A   | 0.8416 | 0.2267 | 0.7413 | 0.168* |
| C9    | 0.6330 (5) | 0.77806 (17) | 0.7218 (3) | 0.0939 (13) |
| H9A   | 0.5980 | 0.8055 | 0.6668 | 0.113* |
| C2    | 0.7889 (5) | 0.2167 (2) | 0.8833 (5) | 0.140 (2) |
| H2A   | 0.7800 | 0.1738 | 0.8781 | 0.168* |

**Atomic displacement parameters (Å²)**

| Atom  | U11  | U22  | U33  | U12  | U13  | U23  |
|-------|------|------|------|------|------|------|
| Dy1   | 0.03380 (6) | 0.03284 (7) | 0.02427 (6) | 0.00167 (4) | −0.00091 (4) | 0.00130 (4) |
| C11   | 0.0556 (4) | 0.0664 (4) | 0.0473 (3) | 0.0255 (3) | −0.0104 (3) | −0.0088 (3) |
| O3    | 0.0544 (10) | 0.0502 (11) | 0.0343 (8) | −0.0156 (8) | −0.0077 (7) | 0.0103 (8) |
| N3    | 0.0500 (11) | 0.0439 (12) | 0.0303 (9) | 0.0042 (10) | 0.0043 (8) | 0.0054 (8) |
| O1    | 0.0509 (9) | 0.0605 (11) | 0.0286 (8) | 0.0160 (9) | 0.0047 (7) | −0.0012 (7) |
| N4    | 0.0372 (10) | 0.0439 (12) | 0.0312 (9) | 0.0055 (9) | 0.0009 (8) | −0.0030 (8) |
| C7    | 0.0383 (12) | 0.0338 (13) | 0.0362 (12) | 0.0016 (10) | 0.0051 (10) | 0.0017 (10) |
| C14   | 0.0493 (14) | 0.0429 (15) | 0.0403 (13) | −0.0087 (12) | −0.0003 (11) | 0.0052 (11) |
| C18   | 0.0604 (16) | 0.0681 (19) | 0.0349 (13) | 0.0217 (14) | 0.0098 (11) | 0.0097 (12) |
| C26   | 0.0482 (14) | 0.0459 (15) | 0.0290 (11) | 0.0127 (12) | 0.0041 (10) | 0.0035 (10) |
| C25   | 0.0397 (13) | 0.0538 (15) | 0.0303 (12) | 0.0140 (12) | 0.0020 (10) | −0.0057 (11) |
| C21   | 0.0440 (14) | 0.080 (2) | 0.0353 (13) | 0.0073 (14) | −0.0017 (11) | −0.0138 (13) |
| C6    | 0.0365 (12) | 0.0509 (16) | 0.0445 (13) | 0.0049 (11) | 0.0071 (10) | −0.0035 (11) |
| C24   | 0.0494 (14) | 0.0535 (17) | 0.0448 (14) | −0.0060 (13) | 0.0053 (11) | −0.0079 (12) |
| C5    | 0.0319 (12) | 0.0499 (16) | 0.0566 (15) | 0.0096 (11) | −0.0013 (11) | −0.0084 (12) |
| C19   | 0.078 (2) | 0.095 (3) | 0.0318 (14) | 0.0236 (18) | 0.0077 (14) | 0.0053 (14) |
| C22   | 0.0583 (17) | 0.097 (2) | 0.0471 (15) | −0.0097 (18) | −0.0034 (13) | −0.0313 (16) |
| C12   | 0.0444 (13) | 0.0429 (15) | 0.0568 (15) | −0.0134 (10) | 0.0005 (12) | 0.0090 (12) |
| C8    | 0.0567 (17) | 0.0577 (19) | 0.086 (2) | −0.0059 (15) | 0.0176 (16) | 0.0155 (16) |
| C11   | 0.0628 (16) | 0.067 (2) | 0.078 (2) | −0.0037 (13) | 0.0156 (16) | −0.0113 (17) |
| C23   | 0.0564 (16) | 0.072 (2) | 0.0648 (17) | −0.0175 (15) | 0.0045 (14) | −0.0239 (15) |
|     | x          | y          | z          | dx         | dy         | dz         |
|-----|------------|------------|------------|------------|------------|------------|
| N2  | 0.101 (2)  | 0.090 (2)  | 0.0763 (17)| -0.0263 (18)| 0.0171 (16)| 0.0270 (16) |
| C17 | 0.0842 (19)| 0.076 (2)  | 0.0398 (14)| 0.0175 (18)| 0.0187 (13)| 0.0244 (14) |
| C13 | 0.0551 (16)| 0.072 (2)  | 0.0658 (18)| -0.0288 (15)| -0.0150 (14)| 0.0267 (15) |
| O2  | 0.0440 (9) | 0.0520 (10)| 0.0284 (8) | 0.0135 (8) | 0.0028 (7) | -0.0033 (7) |
| O4  | 0.0602 (11)| 0.0502 (11)| 0.0347 (8) | -0.0181 (9) | -0.0083 (8) | 0.0070 (8)  |
| C15 | 0.0703 (18)| 0.0530 (17)| 0.0455 (14)| -0.0066 (15)| 0.0101 (13)| 0.0103 (12) |
| C20 | 0.072 (2)  | 0.111 (3)  | 0.0272 (13)| 0.016 (2)  | -0.0040 (13)| -0.0135 (16)|
| C16 | 0.094 (2)  | 0.0609 (19)| 0.0574 (17)| -0.0034 (17)| 0.0266 (15)| 0.0194 (14) |
| C1  | 0.128 (3)  | 0.057 (2)  | 0.065 (2)  | -0.012 (2) | -0.016 (2) | 0.0032 (17) |
| C10 | 0.069 (2)  | 0.051 (2)  | 0.129 (3)  | 0.0161 (17)| -0.011 (2) | 0.004 (2)   |
| C4  | 0.077 (2)  | 0.103 (3)  | 0.116 (3)  | -0.003 (2) | 0.041 (2)  | -0.048 (2) |
| N1  | 0.183 (3)  | 0.057 (2)  | 0.146 (3)  | -0.026 (2) | -0.057 (3) | 0.026 (2)  |
| C3  | 0.079 (2)  | 0.124 (3)  | 0.222 (4)  | -0.001 (3) | 0.044 (3)  | -0.118 (3) |
| C9  | 0.103 (3)  | 0.060 (2)  | 0.100 (3)  | -0.015 (2) | -0.025 (2) | 0.026 (2)  |
| C2  | 0.082 (3)  | 0.062 (3)  | 0.240 (6)  | 0.028 (2)  | -0.052 (3) | -0.042 (3) |

**Geometric parameters (Å, °)**

- Dy1—O4i: 2.3069 (17)  C19—C20: 1.327 (5)
- Dy1—O3: 2.3275 (16)  C19—H19A: 0.930
- Dy1—O2i: 2.3261 (14)  C22—C23: 1.371 (4)
- Dy1—O1: 2.4323 (16)  C22—H22A: 0.930
- Dy1—O2: 2.5170 (15)  C12—C11: 1.376 (4)
- Dy1—N3: 2.5386 (18)  C12—C8: 1.378 (4)
- Dy1—N4: 2.5516 (17)  C12—C13: 1.493 (4)
- Dy1—C11: 2.6392 (6)  C8—N2: 1.350 (4)
- Dy1—C7: 2.850 (2)  C8—H8A: 0.930
- Dy1—Dy1: 3.8976 (2)  C11—C10: 1.356 (4)
- O3—C14: 1.261 (3)  C11—H11A: 0.930
- N3—C15: 1.317 (3)  C23—H23A: 0.930
- N3—C26: 1.362 (3)  N2—C9: 1.315 (5)
- O1—C7: 1.256 (2)  C17—C16: 1.346 (4)
- N4—C24: 1.323 (3)  C17—H17A: 0.930
- N4—C25: 1.362 (3)  C13—H13A: 0.970
- C7—O2: 1.271 (3)  C13—H13B: 0.970
- C7—C6: 1.498 (3)  O2—Dy1: 2.3261 (14)
- C14—O4: 1.262 (3)  O4—Dy1: 2.3069 (17)
- C14—C13: 1.515 (4)  C15—C16: 1.405 (4)
- C18—C19: 1.417 (4)  C15—H15A: 0.930
- C18—C26: 1.415 (3)  C20—H20A: 0.930
- C18—C17: 1.393 (4)  C16—H16A: 0.930
- C26—C25: 1.434 (4)  C1—N1: 1.336 (5)
- C25—C21: 1.409 (3)  C1—H1A: 0.930
- C21—C22: 1.386 (4)  C10—C9: 1.329 (5)
- C21—C20: 1.435 (4)  C10—H10A: 0.930
- C6—C5: 1.498 (4)  C4—C3: 1.395 (6)
- C6—H6A: 0.9700  C4—H4A: 0.930
- C6—H6B: 0.9700  N1—C2: 1.291 (7)
| Bond                  | Distance  | Bond                  | Distance  | Bond                  | Distance  |
|----------------------|-----------|----------------------|-----------|----------------------|-----------|
| C24—C23              | 1.396 (4) | C3—C2                | 1.362 (7) |                     |           |
| C24—H24A             | 0.9300    | C3—H3A               | 0.9300    |                     |           |
| C5—C1                | 1.364 (4) | C9—H9A               | 0.9300    |                     |           |
| C5—C4                | 1.374 (4) | C2—H2A               | 0.9300    |                     |           |
| O4i—Dy1—O3           | 138.19 (5)| C25—C21—C20          | 119.7 (3) |                     |           |
| O4i—Dy1—O2i          | 74.46 (6) | C5—C6—C7             | 112.05 (19)|                    |           |
| O3—Dy1—O2i           | 73.64 (6) | C5—C6—H6A            | 109.2     |                     |           |
| O4i—Dy1—O1           | 88.97 (6) | C7—C6—H6A            | 109.2     |                     |           |
| O3—Dy1—O1            | 87.84 (6) | C5—C6—H6B            | 109.2     |                     |           |
| O2i—Dy1—O1           | 125.15 (5)| C7—C6—H6B            | 109.2     |                     |           |
| O4i—Dy1—O2           | 73.42 (6) | H6A—C6—H6B           | 107.9     |                     |           |
| O3—Dy1—O2            | 71.86 (6) | N4—C24—C23           | 124.0 (3) |                     |           |
| O2i—Dy1—O2           | 72.89 (6) | N4—C24—H24A          | 118.0     |                     |           |
| O1—Dy1—O2            | 52.27 (5) | C23—C24—H24A         | 118.0     |                     |           |
| O4i—Dy1—N3           | 141.73 (6)| C1—C5—C4             | 116.8 (3) |                     |           |
| O3—Dy1—N3            | 77.03 (6) | C1—C5—C6             | 120.8 (3) |                     |           |
| O2i—Dy1—N3           | 142.50 (7)| C4—C5—C6             | 122.4 (3) |                     |           |
| O1—Dy1—N3            | 75.81 (6) | C20—C19—C18          | 121.6 (3) |                     |           |
| O2—Dy1—N3            | 118.94 (6)| C20—C19—H19A         | 119.2     |                     |           |
| O4i—Dy1—N4           | 77.16 (6) | C18—C19—H19A         | 119.2     |                     |           |
| O3—Dy1—N4            | 141.62 (6)| C21—C22—C23          | 120.1 (3) |                     |           |
| O2i—Dy1—N4           | 143.32 (6)| C21—C22—H22A         | 120.0     |                     |           |
| O1—Dy1—N4            | 76.54 (5) | C23—C22—H22A         | 120.0     |                     |           |
| O2—Dy1—N4            | 120.04 (6)| C11—C12—C8           | 115.8 (3) |                     |           |
| N3—Dy1—N4            | 65.29 (6) | C11—C12—C13          | 123.2 (3) |                     |           |
| O4i—Dy1—Cl1          | 101.26 (5)| C8—C12—C13           | 121.0 (3) |                     |           |
| O3—Dy1—Cl1           | 101.19 (5)| N2—C8—C12            | 123.8 (3) |                     |           |
| O2i—Dy1—Cl1          | 83.42 (4) | N2—C8—H8A            | 118.1     |                     |           |
| O1—Dy1—Cl1           | 151.41 (4)| C12—C8—H8A           | 118.1     |                     |           |
| O2—Dy1—Cl1           | 156.30 (4)| C12—C11—C10          | 121.0 (3) |                     |           |
| N3—Dy1—Cl1           | 79.86 (5) | C12—C11—H11A         | 119.5     |                     |           |
| N4—Dy1—Cl1           | 79.81 (4) | C10—C11—H11A         | 119.5     |                     |           |
| O4i—Dy1—C7           | 82.55 (6) | C24—C23—C22          | 118.2 (3) |                     |           |
| O3—Dy1—C7            | 76.68 (6) | C24—C23—H23A         | 120.9     |                     |           |
| O2i—Dy1—C7           | 99.24 (6) | C22—C23—H23A         | 120.9     |                     |           |
| O1—Dy1—C7            | 25.99 (5) | C9—N2—C8             | 115.9 (3) |                     |           |
| O2—Dy1—C7            | 26.47 (5) | C16—C17—C18          | 120.1 (3) |                     |           |
| N3—Dy1—C7            | 96.17 (6) | C16—C17—H17A         | 120.0     |                     |           |
| N4—Dy1—C7            | 99.62 (6) | C18—C17—H17A         | 120.0     |                     |           |
| Cl1—Dy1—C7           | 175.87 (5)| C12—C13—C14          | 116.1 (2) |                     |           |
| O4i—Dy1—Dy1i         | 69.87 (4) | C12—C13—H13A         | 108.3     |                     |           |
| O3—Dy1—Dy1i          | 68.34 (4) | C14—C13—H13A         | 108.3     |                     |           |
| O2—Dy1—Dy1i          | 38.11 (4) | C12—C13—H13B         | 108.3     |                     |           |
| O1—Dy1—Dy1i          | 87.04 (3) | C14—C13—H13B         | 108.3     |                     |           |
| O2—Dy1—Dy1i          | 34.78 (3) | H13A—C13—H13B        | 107.4     |                     |           |
| N3—Dy1—Dy1i          | 141.81 (4)| C7—O2—Dy1I           | 160.55 (14)|                   |           |
| N4—Dy1—Dy1i          | 143.32 (4)| C7—O2—Dy1            | 91.55 (12)|                     |           |
| Bond                   | Distance (Å) | Error (Å) | Angle (°) | Error (°) |
|------------------------|--------------|-----------|-----------|-----------|
| C1—Dy1—Dy1<sup>i</sup> | 121.533      | 15        | 107.11    | 6         |
| C7—Dy1—Dy1<sup>i</sup> | 61.17        | 4         | 137.13    | 15        |
| C14—O3—Dy1            | 138.89       | 15        | 123.7     | 3         |
| C15—N3—C26            | 117.5        | 2         | 118.1     |           |
| C15—N3—Dy1            | 123.72       | 16        | 118.1     |           |
| C26—N3—Dy1            | 118.73       | 16        | 120.9     | 3         |
| C7—O1—Dy1             | 95.93        | 14        | 119.6     |           |
| C24—N4—C25            | 117.5        | 2         | 119.6     |           |
| C24—N4—Dy1            | 124.18       | 15        | 118.6     |           |
| O1—C7—Dy1             | 119.4        | 2         | 120.6     |           |
| O1—C7—C6              | 121.2        | 2         | 125.6     | 4         |
| O2—C7—C6              | 119.44       | 19        | 117.2     |           |
| O2—C7—Dy1             | 58.08        | 12        | 117.2     |           |
| O2—C7—Dy1             | 61.98        | 11        | 118.1     | 3         |
| O4—C14—O3             | 125.6        | 2         | 120.9     |           |
| O4—C14—C13            | 115.7        | 2         | 118.4     | 4         |
| O3—C14—C13            | 118.7        | 2         | 120.8     |           |
| C19—C18—C26           | 119.7        | 3         | 120.8     |           |
| C19—C18—C17           | 122.6        | 3         | 116.0     | 4         |
| C26—C18—C17           | 117.7        | 2         | 118.4     | 4         |
| N3—C26—C25            | 122.0        | 2         | 120.8     |           |
| N3—C26—C25            | 118.8        | 2         | 120.8     |           |
| C18—C26—C25           | 119.2        | 2         | 125.3     | 3         |
| N4—C25—C21            | 122.2        | 2         | 117.4     |           |
| N4—C25—C26            | 118.8        | 2         | 117.4     |           |
| C21—C25—C26           | 119.0        | 2         | 124.7     | 4         |
| C22—C21—C25           | 117.9        | 2         | 117.6     |           |
| C22—C21—C20           | 122.4        | 3         | 117.6     |           |

Symmetry code: (i) −x+1, −y+1, −z+2.