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$[\eta^5\text{-}2,3\text{-Bis(trimethylsilyl)-}2,3\text{-dicarbano-}nido\text{-hexaborane}(2-)]\text{chlorido}(N,N,N',N'\text{-tetramethylethylenediamine})\text{-dysprosium(III)}$

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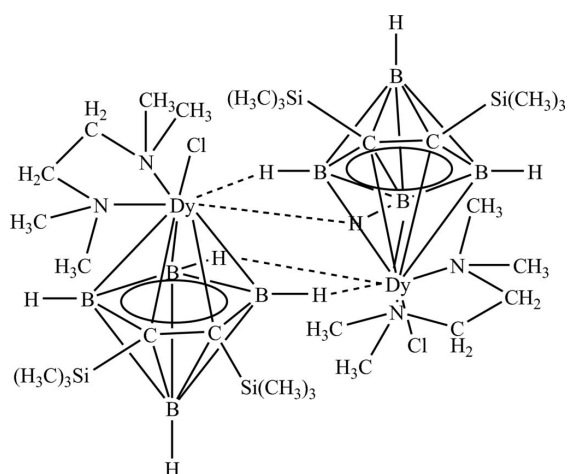
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 Key indicators: single-crystal X-ray study; $T = 293\text{ K}$; mean $\sigma(\text{C}-\text{C}) = 0.005\text{ \AA}$; R factor = 0.024; wR factor = 0.051; data-to-parameter ratio = 18.0.

The structure of the title compound, $[\text{Dy}(\text{C}_8\text{H}_{22}\text{B}_4\text{Si}_2)\text{Cl}(\text{C}_6\text{H}_{16}\text{N}_2)]$, reveals that a center of symmetry exists within the dimeric half-sandwich units. Within each half-sandwich, the Dy^{III} ion is coordinated by the five-membered ring of the carborane, tetramethylethylenediamine and the chloride ion.

Related literature

 For related literature, see: Bazan *et al.* (1993); Tomlinson *et al.* (2005); Wang *et al.* (2006).


Experimental

Crystal data

 $[\text{Dy}(\text{C}_8\text{H}_{22}\text{B}_4\text{Si}_2)\text{Cl}(\text{C}_6\text{H}_{16}\text{N}_2)]$
 $M_r = 531.84$
 Monoclinic, $P2_1/n$
 $a = 11.4467(8)\text{ \AA}$
 $b = 14.862(1)\text{ \AA}$
 $c = 13.8615(9)\text{ \AA}$
 $\beta = 92.770(1)^\circ$
 $V = 2355.4(3)\text{ \AA}^3$
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 3.39\text{ mm}^{-1}$
 $T = 293(2)\text{ K}$
 $0.80 \times 0.30 \times 0.30\text{ mm}$

Data collection

 Bruker SMART CCD PLATFORM diffractometer
 Absorption correction: multi-scan *SADABS* (Sheldrick, 2006)
 $T_{\text{min}} = 0.257$, $T_{\text{max}} = 0.362$
 17271 measured reflections
 4151 independent reflections
 4142 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.019$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.024$
 $wR(F^2) = 0.050$
 $S = 1.44$
 4151 reflections
 231 parameters

 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.64\text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.61\text{ e \AA}^{-3}$

Data collection: *SMART* (Bruker, 2003); cell refinement: *SMART* and *SAINT* (Bruker, 2003); data reduction: *SAINT* (Bruker, 2003); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2008); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SI2081).

References

- Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
- Bazan, G. C., Schaefer, W. P. & Bercaw, J. E. (1993). *Organometallics*, **12**, 2126–2130.
- Bruker (2003). *SMART* and *SAINT*. Bruker Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Sheldrick, G. M. (2006). *SADABS*. University of Göttingen, Germany.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Tomlinson, S., Zheng, C., Hosmane, N. S., Yang, J., Wang, Y., Zhang, H., Gray, T. G., Demissie, T., Maguire, J. A., Baumann, F., Klein, A., Sarkar, B., Kaim, W. & Lipscomb, W. N. (2005). *Organometallics* **24**, 2177–2187.
- Wang, J., Smith, J., Zheng, C., Maguire, J. A. & Hosmane, N. S. (2006). *Organometallics*, **25**, 4118–4130.

supplementary materials

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[η^5 -2,3-Bis(trimethylsilyl)-2,3-dicarba-*nido*-hexaborane(2-)]chlorido(*N,N,N',N'*-tetramethylethylenediamine)dysprosium(III)

C. Zheng, G. Canseco-Melchor, J. A. Maguire and N. S. Hosmane

Comment

The lanthanacarboranes (LnC_2B_4) cage systems are of interest in that their structural chemistry depends on the number and nature of the metal ligands other than the particular carborane. For example, the reaction of the tetrahydrofuran (THF) solvated dilithium compounds of $\text{B}_4\text{C}_8\text{H}_{22}\text{Si}_2^{2-}$ with LnCl_3 in 2:1 molar ratios produced exclusively a trinuclear clusters (Tomlinson *et al.* 2005, Wang *et al.* 2006, and literature cited therein). The methoxide and oxide ions were thought to be the result of a degradation of the THF molecules. This is consistent with the observation that the reaction of the TMEDA lithi-acarboranes with LnCl_3 in 2:1 molar ratios gave the expected full sandwich lanthanacarboranes. This tendency for TMEDA to disrupt complex aggregation and alter the course of a metalation reactions was also found in the d-block metals. The reaction of the THF solvated dilithium salts of $\text{B}_4\text{C}_8\text{H}_{22}\text{Si}_2^{2-}$ with either CoCl_2 or NiCl_2 induced a metal disproportion reaction yielding *commo*-complexes and the respective metals. On the other hand, similar reactions with the TMEDA-solvated carborane dianions produced the corresponding *closo*-compound (Tomlinson *et al.* 2005). The reaction of a *nido*-compound with a number of lanthanide halides in 2:1 carborane-to-lanthanide molar ratios produced only the corresponding dimers (Wang *et al.* 2006). The presence of the reactive chlorides made these compounds potentially useful precursors in the syntheses of other lanthanacarboranes. Since dimer formation is thought to inhibit the reactions of the metallacarboranes (Bazan *et al.* 1993), the synthesis of a monomeric dysprosacarborane was attempted by the reaction of DyCl_3 and the *nido*-compound, giving the title compound. The structure of the title compound, shown in Figure 1, is somewhat surprising in that it still remains a dimer. The structure consists of two half-sandwich units bridged at the B4, B5 positions by two pairs of B—H—Dy bonds. The average coordination bond distances (Å) are: Dy—Cl 2.5851 (9), Dy—N1 2.598 (3), Dy—N2 2.698 (3), Dy—B4,B5 2.728 (4), 2.692 (4), Dy-av(C1, C2, B3, B4, B5) 2.720 and the corresponding bond angles (°) are Cl—Dy—N1 95.98 (7), Cl—Dy—B5 143.74 (8), N(1)—Dy—N(2) 68.3 (1), B(5)—Dy—B(4) 35.7 (1). The Dy-av(C_2B_3) distance of 2.712 Å and the Dy—Cl distance of 2.5851 (9) Å in the structure are similar to the values of 2.717 Å and 2.5757 (14) Å, respectively, found in another Dy compound (Wang *et al.* 2006). This is one of the few instances where the substitution of THF by TMEDA molecules does not change either the cluster or intermolecular geometries.

Experimental

The reaction of DyCl_3 with the TMEDA solvated monosodium *nido*- compound, *nido*-1-Na(TMEDA)₂-2,3-(SiMe₃)₂-2,3-C₂B₄H₄, in a 1:2 molar ratio in dry benzene at 60° C produced the corresponding half sandwich carborane in 88% yield.

Refinement

The hydrogen atoms on carbon atoms were refined using the riding model in *SHELXL* with the U_{iso} equal to 1.5 times of that of the preceding carbon atoms for the methyl groups and 1.3 times for the rings. The C—H distances are equal to 0.97 and 0.96 Å for the CH₂ and CH₃ groups, respectively. The hydrogen atoms attached to boron atoms were located using the

supplementary materials

difference map, those of the carborane ring were refined using the riding model with B—H distances 0.93 Å. The hydrogen atom attached to B6 was refined with an isotropic displacement parameter with B—H distance 0.96 Å. The $U_{\text{iso}}(\text{H}) = 1.2$ times $U_{\text{eq}}(\text{B3}, \text{B4}, \text{B5})$.

Figures

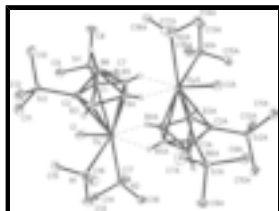


Fig. 1. Thermal ellipsoid drawing of the title compound shown at the 30% probability level. Atoms of the symmetry related moiety of the dimer complex are indicated "A" (symmetry code 1 - x, 2 - y, 1 - z).



Fig. 2. Reaction scheme

[η^5 -2,3-Bis(trimethylsilyl)-2,3-dicarba-*nido*-hexaborane(2-)]chlorido(N,N,N',N'-tetramethylethylenediamine)dysprosium(III)

Crystal data

[Dy(C₈H₂₂B₄Si₂)Cl(C₆H₁₆N₂)]

$M_r = 531.84$

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 11.4467$ (8) Å

$b = 14.8624$ (10) Å

$c = 13.8615$ (9) Å

$\beta = 92.7700$ (10)°

$V = 2355.4$ (3) Å³

$Z = 4$

$F_{000} = 1068$

$D_x = 1.500$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 562 reflections

$\theta = -14-14^\circ$

$\mu = 3.39$ mm⁻¹

$T = 293$ (2) K

Column, colorless

$0.80 \times 0.30 \times 0.30$ mm

Data collection

Bruker SMART CCD PLATFORM
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

Detector resolution: 0 pixels mm⁻¹

$T = 293$ (2) K

ω scans

4151 independent reflections

4142 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.019$

$\theta_{\text{max}} = 25.0^\circ$

$\theta_{\text{min}} = 2.0^\circ$

$h = -13 \rightarrow 13$

Absorption correction: multi-scan
 SADABS (Sheldrick, 2006) $k = -17 \rightarrow 17$
 $T_{\min} = 0.257$, $T_{\max} = 0.362$ $l = -16 \rightarrow 16$
 17271 measured reflections

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.024$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.050$	$w = 1/[\sigma^2(F_o^2) + 3.9928P]$
$S = 1.44$	where $P = (F_o^2 + 2F_c^2)/3$
4151 reflections	$(\Delta/\sigma)_{\max} = 0.013$
231 parameters	$\Delta\rho_{\max} = 0.64 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\min} = -0.61 \text{ e } \text{\AA}^{-3}$
	Extinction correction: none

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Dy	0.584689 (12)	0.954047 (10)	0.626856 (11)	0.02053 (6)
Cl	0.65147 (8)	0.79787 (6)	0.69040 (8)	0.0406 (2)
Si1	0.37951 (8)	0.73248 (6)	0.55959 (7)	0.0268 (2)
Si2	0.31576 (8)	0.84186 (6)	0.80092 (7)	0.0269 (2)
C1	0.3894 (3)	0.8543 (2)	0.5966 (2)	0.0205 (6)
C2	0.3757 (3)	0.8993 (2)	0.6921 (2)	0.0221 (7)
B3	0.3682 (3)	1.0040 (3)	0.6794 (3)	0.0249 (8)
H3	0.3640	1.0465	0.7285	0.030*
B4	0.3693 (3)	1.0245 (2)	0.5630 (3)	0.0233 (8)
H4	0.3622	1.0799	0.5320	0.028*
B5	0.3862 (3)	0.9239 (2)	0.5134 (3)	0.0217 (7)
H5	0.3926	0.9117	0.4481	0.026*
B6	0.2804 (3)	0.9338 (3)	0.6029 (3)	0.0239 (8)

supplementary materials

C7	0.4863 (4)	0.7136 (3)	0.4645 (3)	0.0501 (11)
H7A	0.4791	0.6531	0.4410	0.075*
H7B	0.5642	0.7232	0.4914	0.075*
H7C	0.4707	0.7550	0.4123	0.075*
C8	0.2302 (4)	0.7083 (3)	0.5048 (3)	0.0472 (11)
H8A	0.1723	0.7344	0.5441	0.071*
H8B	0.2185	0.6444	0.5012	0.071*
H8C	0.2230	0.7336	0.4411	0.071*
C9	0.4155 (3)	0.6475 (2)	0.6554 (3)	0.0380 (9)
H9A	0.3536	0.6453	0.6996	0.057*
H9B	0.4873	0.6639	0.6896	0.057*
H9C	0.4242	0.5894	0.6262	0.057*
C10	0.1876 (3)	0.7697 (3)	0.7643 (3)	0.0372 (9)
H10A	0.1693	0.7311	0.8170	0.056*
H10B	0.2062	0.7338	0.7095	0.056*
H10C	0.1214	0.8072	0.7474	0.056*
C11	0.4250 (4)	0.7748 (3)	0.8752 (3)	0.0450 (10)
H11A	0.4679	0.8139	0.9191	0.068*
H11B	0.4780	0.7458	0.8335	0.068*
H11C	0.3850	0.7300	0.9112	0.068*
C12	0.2567 (4)	0.9287 (3)	0.8824 (3)	0.0436 (10)
H12A	0.3179	0.9698	0.9023	0.065*
H12B	0.2270	0.9000	0.9382	0.065*
H12C	0.1947	0.9611	0.8487	0.065*
N1	0.6388 (3)	1.0410 (2)	0.7853 (2)	0.0323 (7)
C13	0.7672 (3)	1.0466 (3)	0.8066 (3)	0.0430 (10)
H13A	0.7826	1.0429	0.8760	0.052*
H13B	0.7950	1.1046	0.7854	0.052*
C14	0.8344 (3)	0.9737 (3)	0.7587 (3)	0.0367 (9)
H14A	0.9169	0.9798	0.7768	0.044*
H14B	0.8086	0.9155	0.7812	0.044*
N2	0.8178 (3)	0.9778 (2)	0.6525 (2)	0.0345 (7)
C15	0.5868 (4)	0.9852 (3)	0.8603 (3)	0.0442 (10)
H15A	0.6170	0.9250	0.8572	0.066*
H15B	0.5034	0.9840	0.8496	0.066*
H15C	0.6064	1.0102	0.9229	0.066*
C16	0.5897 (4)	1.1327 (3)	0.7910 (3)	0.0464 (10)
H16A	0.6069	1.1570	0.8543	0.070*
H16B	0.5064	1.1303	0.7788	0.070*
H16C	0.6237	1.1704	0.7436	0.070*
C17	0.8846 (3)	0.9040 (3)	0.6097 (4)	0.0554 (12)
H17A	0.9663	0.9113	0.6268	0.083*
H17B	0.8727	0.9054	0.5407	0.083*
H17C	0.8581	0.8474	0.6338	0.083*
C18	0.8693 (4)	1.0633 (3)	0.6189 (3)	0.0517 (12)
H18A	0.9477	1.0692	0.6460	0.078*
H18B	0.8230	1.1130	0.6392	0.078*
H18C	0.8706	1.0628	0.5497	0.078*
H6	0.191 (3)	0.916 (2)	0.600 (3)	0.029 (9)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Dy	0.01951 (8)	0.01862 (9)	0.02317 (9)	-0.00146 (6)	-0.00200 (6)	0.00244 (6)
Cl	0.0311 (5)	0.0249 (4)	0.0647 (6)	0.0002 (4)	-0.0081 (4)	0.0114 (4)
Si1	0.0314 (5)	0.0189 (5)	0.0301 (5)	-0.0025 (4)	0.0010 (4)	-0.0016 (4)
Si2	0.0303 (5)	0.0268 (5)	0.0238 (5)	-0.0015 (4)	0.0049 (4)	0.0036 (4)
C1	0.0178 (15)	0.0198 (16)	0.0238 (16)	-0.0006 (12)	0.0005 (12)	0.0010 (13)
C2	0.0196 (16)	0.0224 (16)	0.0242 (17)	-0.0003 (13)	0.0008 (13)	0.0005 (13)
B3	0.0251 (19)	0.0226 (19)	0.027 (2)	0.0016 (15)	0.0014 (15)	-0.0026 (15)
B4	0.0221 (18)	0.0163 (18)	0.031 (2)	0.0009 (14)	0.0003 (15)	0.0034 (15)
B5	0.0184 (17)	0.0230 (19)	0.0237 (18)	-0.0028 (14)	-0.0002 (14)	-0.0003 (15)
B6	0.0207 (18)	0.0231 (19)	0.028 (2)	0.0002 (15)	0.0010 (15)	0.0021 (15)
C7	0.067 (3)	0.036 (2)	0.049 (3)	0.008 (2)	0.022 (2)	-0.0054 (19)
C8	0.050 (3)	0.036 (2)	0.054 (3)	-0.0117 (19)	-0.019 (2)	-0.0039 (19)
C9	0.039 (2)	0.0247 (19)	0.049 (2)	-0.0006 (16)	-0.0026 (18)	0.0039 (17)
C10	0.033 (2)	0.040 (2)	0.040 (2)	-0.0074 (17)	0.0114 (17)	0.0021 (17)
C11	0.048 (2)	0.045 (2)	0.041 (2)	0.0010 (19)	-0.0038 (19)	0.0168 (19)
C12	0.053 (3)	0.042 (2)	0.036 (2)	-0.0025 (19)	0.0161 (19)	-0.0034 (18)
N1	0.0360 (17)	0.0301 (16)	0.0303 (16)	-0.0061 (13)	-0.0058 (13)	-0.0029 (13)
C13	0.037 (2)	0.047 (2)	0.044 (2)	-0.0102 (18)	-0.0129 (18)	-0.0051 (19)
C14	0.0300 (19)	0.039 (2)	0.039 (2)	-0.0056 (16)	-0.0141 (16)	0.0089 (17)
N2	0.0266 (15)	0.0383 (18)	0.0377 (17)	-0.0059 (13)	-0.0058 (13)	0.0031 (14)
C15	0.052 (2)	0.053 (3)	0.027 (2)	-0.011 (2)	-0.0055 (18)	0.0033 (18)
C16	0.053 (3)	0.034 (2)	0.052 (3)	-0.0002 (19)	-0.005 (2)	-0.0141 (19)
C17	0.025 (2)	0.068 (3)	0.073 (3)	-0.001 (2)	0.002 (2)	-0.016 (3)
C18	0.037 (2)	0.068 (3)	0.049 (3)	-0.023 (2)	-0.0137 (19)	0.020 (2)

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

Dy—C1	2.5851 (9)	C9—H9B	0.9600
Dy—N1	2.598 (3)	C9—H9C	0.9600
Dy—B5 ⁱ	2.692 (4)	C10—H10A	0.9600
Dy—N2	2.698 (3)	C10—H10B	0.9600
Dy—C1	2.698 (3)	C10—H10C	0.9600
Dy—C2	2.723 (3)	C11—H11A	0.9600
Dy—B4 ⁱ	2.728 (4)	C11—H11B	0.9600
Si1—C7	1.861 (4)	C11—H11C	0.9600
Si1—C9	1.865 (4)	C12—H12A	0.9600
Si1—C8	1.872 (4)	C12—H12B	0.9600
Si1—C1	1.884 (3)	C12—H12C	0.9600
Si2—C12	1.864 (4)	N1—C15	1.477 (5)
Si2—C10	1.867 (4)	N1—C16	1.477 (5)
Si2—C11	1.869 (4)	N1—C13	1.487 (5)
Si2—C2	1.890 (3)	C13—C14	1.502 (6)
C1—C2	1.498 (4)	C13—H13A	0.9700
C1—B5	1.548 (5)	C13—H13B	0.9700

supplementary materials

C1—B6	1.724 (5)	C14—N2	1.477 (5)
C2—B3	1.568 (5)	C14—H14A	0.9700
C2—B6	1.689 (5)	C14—H14B	0.9700
B3—B4	1.642 (5)	N2—C17	1.478 (5)
B3—H3	0.9300	N2—C18	1.485 (5)
B4—B5	1.661 (5)	C15—H15A	0.9600
B4—Dy ⁱ	2.728 (4)	C15—H15B	0.9600
B4—H4	0.9300	C15—H15C	0.9600
B5—Dy ⁱ	2.692 (4)	C16—H16A	0.9600
B5—H5	0.9300	C16—H16B	0.9600
B6—H6	1.06 (4)	C16—H16C	0.9600
C7—H7A	0.9600	C17—H17A	0.9600
C7—H7B	0.9600	C17—H17B	0.9600
C7—H7C	0.9600	C17—H17C	0.9600
C8—H8A	0.9600	C18—H18A	0.9600
C8—H8B	0.9600	C18—H18B	0.9600
C8—H8C	0.9600	C18—H18C	0.9600
C9—H9A	0.9600		
C1—Dy—N1	95.98 (7)	Si1—C8—H8B	109.5
C1—Dy—B5 ⁱ	143.74 (8)	H8A—C8—H8B	109.5
N1—Dy—B5 ⁱ	104.06 (10)	Si1—C8—H8C	109.5
C1—Dy—N2	78.36 (7)	H8A—C8—H8C	109.5
N1—Dy—N2	68.26 (10)	H8B—C8—H8C	109.5
B5 ⁱ —Dy—N2	81.43 (10)	Si1—C9—H9A	109.5
C1—Dy—C1	77.81 (7)	Si1—C9—H9B	109.5
N1—Dy—C1	124.51 (10)	H9A—C9—H9B	109.5
B5 ⁱ —Dy—C1	112.86 (10)	Si1—C9—H9C	109.5
N2—Dy—C1	154.04 (9)	H9A—C9—H9C	109.5
C1—Dy—C2	82.45 (7)	H9B—C9—H9C	109.5
N1—Dy—C2	92.59 (10)	Si2—C10—H10A	109.5
B5 ⁱ —Dy—C2	125.64 (10)	Si2—C10—H10B	109.5
N2—Dy—C2	151.01 (9)	H10A—C10—H10B	109.5
C1—Dy—C2	32.08 (9)	Si2—C10—H10C	109.5
C1—Dy—B4 ⁱ	111.47 (8)	H10A—C10—H10C	109.5
N1—Dy—B4 ⁱ	135.13 (10)	H10B—C10—H10C	109.5
B5 ⁱ —Dy—B4 ⁱ	35.69 (11)	Si2—C11—H11A	109.5
N2—Dy—B4 ⁱ	82.89 (10)	Si2—C11—H11B	109.5
C1—Dy—B4 ⁱ	96.39 (10)	H11A—C11—H11B	109.5
C2—Dy—B4 ⁱ	124.68 (10)	Si2—C11—H11C	109.5
C7—Si1—C9	105.8 (2)	H11A—C11—H11C	109.5
C7—Si1—C8	107.6 (2)	H11B—C11—H11C	109.5
C9—Si1—C8	108.95 (19)	Si2—C12—H12A	109.5
C7—Si1—C1	107.74 (17)	Si2—C12—H12B	109.5
C9—Si1—C1	116.69 (16)	H12A—C12—H12B	109.5
C8—Si1—C1	109.69 (17)	Si2—C12—H12C	109.5
C12—Si2—C10	105.03 (19)	H12A—C12—H12C	109.5

C12—Si2—C11	106.8 (2)	H12B—C12—H12C	109.5
C10—Si2—C11	109.84 (19)	C15—N1—C16	108.3 (3)
C12—Si2—C2	109.07 (17)	C15—N1—C13	108.6 (3)
C10—Si2—C2	110.65 (16)	C16—N1—C13	108.3 (3)
C11—Si2—C2	114.95 (17)	C15—N1—Dy	103.2 (2)
C2—C1—B5	111.1 (3)	C16—N1—Dy	115.3 (2)
C2—C1—B6	62.8 (2)	C13—N1—Dy	112.9 (2)
B5—C1—B6	65.7 (2)	N1—C13—C14	113.2 (3)
C2—C1—Si1	131.5 (2)	N1—C13—H13A	108.9
B5—C1—Si1	116.1 (2)	C14—C13—H13A	108.9
B6—C1—Si1	129.7 (2)	N1—C13—H13B	108.9
C2—C1—Dy	74.86 (17)	C14—C13—H13B	108.9
B5—C1—Dy	74.80 (17)	H13A—C13—H13B	107.7
B6—C1—Dy	102.23 (18)	N2—C14—C13	111.5 (3)
Si1—C1—Dy	127.49 (14)	N2—C14—H14A	109.3
C1—C2—B3	110.6 (3)	C13—C14—H14A	109.3
C1—C2—B6	65.2 (2)	N2—C14—H14B	109.3
B3—C2—B6	65.6 (2)	C13—C14—H14B	109.3
C1—C2—Si2	124.1 (2)	H14A—C14—H14B	108.0
B3—C2—Si2	121.2 (2)	C14—N2—C17	109.0 (3)
B6—C2—Si2	118.4 (2)	C14—N2—C18	108.3 (3)
C1—C2—Dy	73.05 (17)	C17—N2—C18	106.7 (3)
B3—C2—Dy	73.14 (18)	C14—N2—Dy	101.8 (2)
B6—C2—Dy	102.27 (19)	C17—N2—Dy	112.2 (2)
Si2—C2—Dy	139.32 (15)	C18—N2—Dy	118.4 (2)
C2—B3—B4	106.9 (3)	N1—C15—H15A	109.5
C2—B3—H3	126.5	N1—C15—H15B	109.5
B4—B3—H3	126.5	H15A—C15—H15B	109.5
B3—B4—B5	104.3 (3)	N1—C15—H15C	109.5
B3—B4—Dy ⁱ	168.7 (2)	H15A—C15—H15C	109.5
B5—B4—Dy ⁱ	70.97 (18)	H15B—C15—H15C	109.5
B3—B4—H4	127.9	N1—C16—H16A	109.5
B5—B4—H4	127.9	N1—C16—H16B	109.5
Dy ⁱ —B4—H4	57.8	H16A—C16—H16B	109.5
C1—B5—B4	107.0 (3)	N1—C16—H16C	109.5
C1—B5—Dy ⁱ	171.5 (2)	H16A—C16—H16C	109.5
B4—B5—Dy ⁱ	73.34 (18)	H16B—C16—H16C	109.5
C1—B5—H5	126.5	N2—C17—H17A	109.5
B4—B5—H5	126.5	N2—C17—H17B	109.5
Dy ⁱ —B5—H5	53.8	H17A—C17—H17B	109.5
C2—B6—C1	52.07 (18)	N2—C17—H17C	109.5
C2—B6—H6	122.4 (19)	H17A—C17—H17C	109.5
C1—B6—H6	122 (2)	H17B—C17—H17C	109.5
Si1—C7—H7A	109.5	N2—C18—H18A	109.5
Si1—C7—H7B	109.5	N2—C18—H18B	109.5
H7A—C7—H7B	109.5	H18A—C18—H18B	109.5
Si1—C7—H7C	109.5	N2—C18—H18C	109.5
H7A—C7—H7C	109.5	H18A—C18—H18C	109.5

supplementary materials

H7B—C7—H7C 109.5

H18B—C18—H18C

109.5

Si1—C8—H8A 109.5

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

