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Tetrachlorido[N^2,$N^{2'}$-(dimethylsilanediyl)bis(*N*-*tert*-butyl-3-methylbenzimidamidato)-κ^2N^2,$N^{2'}$]hafnium(IV)

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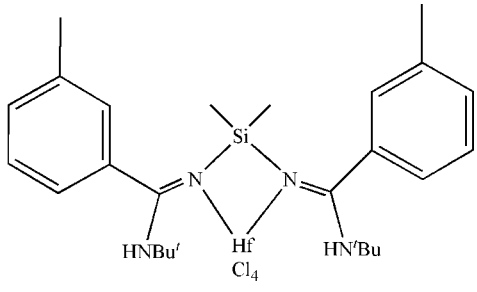
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.007 Å; R factor = 0.028; wR factor = 0.061; data-to-parameter ratio = 17.3.

The symmetric title molecule, $[Hf(C_{26}H_{40}N_4Si)Cl_4]$, lies about a twofold rotation axis. The Hf^{IV} and Si atoms lie on the rotation axis with all other atoms being in general positions. The Hf^{IV} atom is six-coordinated by two N atoms from the N^2,$N^{2'}$-(dimethylsilanediyl)bis(*N*-*tert*-butyl-3-methylbenzimidamidate) ligand and four Cl^- ions in a slightly distorted octahedral geometry. The two amidinate moieties are connected through the central Si atom with Si—N bond length of 1.762 (3) Å, generating the characteristic N—C—N—Si—N—C—N skeleton of a silyl-linked *ansa*-bis(amidine) species.

Related literature

For reviews of related amidinate ligands and their applications, see: Edelmann (2012); Lei *et al.* (2011); Münch *et al.* (2008). For a review of the modification of the steric and electronic properties of amidinate ligands by varying their substitution patterns, see: Liu *et al.* (2013); Qian *et al.* (2010). For related silyl-linked bis(amidinate) ligands and the synthesis of their metal complexes, including a closely related Hf complex, see: Bai *et al.* (2013).



Experimental

Crystal data

$[Hf(C_{26}H_{40}N_4Si)Cl_4]$
M_r = 757
Monoclinic, $C2/c$
a = 9.4373 (14) Å
b = 17.992 (3) Å
c = 19.966 (3) Å
β = 103.276 (3)°
V = 3299.5 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 3.54 mm^{-1}
T = 296 K
0.08 × 0.05 × 0.05 mm

Data collection

Bruker SMART area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.765, T_{max} = 0.843
7121 measured reflections
2920 independent reflections
2446 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.061
S = 1.02
2920 reflections
169 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

Table 1
Selected bond lengths (Å).

Hf1—N2	2.233 (3)	Si1—C13	1.857 (5)
Hf1—Cl2	2.4261 (11)	N1—C5	1.318 (5)
Hf1—Cl1	2.4366 (11)	N1—C1	1.504 (5)
Hf1—Si1	3.0588 (16)	N1—H1	0.8600
Si1—N2	1.762 (3)		

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL/PC* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ5366).

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Tetrachlorido[N^2,$N^{2'}$-(dimethylsilanediyl)bis(*N*-*tert*-butyl-3-methylbenzimidamidato)-κ^2N^2,$N^{2'}$]hafnium(IV)

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S1. Comment

Anionic N,*N*-chelating amidinate ligands, have been widely used in the synthesis of organometallic complexes of the s-, *p*-, d-, and f-block metals for a number of years (Edelmann, 2012; Münch *et al.*, 2008). Their steric and electronic properties can easily be modified by a simple variation of the substitution pattern (Liu *et al.*, 2013; Qian *et al.*, 2010). In the search for ancillary ligands to replace cyclopentadienyls to create non-metallocene species, amidinate anions have found many applications in coordination chemistry, and also as ancillary ligands to form metal complexes which act as catalysts in organic transformations and ethylene polymerizations (Lei *et al.*, 2011).

Linked bis(amidinate) ligands are a very special branch of this class of compound and their chemistry has been developed in recent years. We explored a class of silyl linked bis(amidinate) ligands, and applied them to the synthesis of metal complexes. They imposed a close contact between the two amidinate moieties and had the advantage of affording binuclear complexes analogous to an "ansa-metallocene" (Bai *et al.*, 2013). Here, the synthesis and characterization of the Hf(IV) complex $SiMe_2[NC(m\text{-MePh})N(Bu^t)H]_2HfCl_4$ bearing the silyl-linked ansa-bis(amidine) ligands will be described.

N′,*N*″-(dimethylsilanediyl)bis(*N*-*tert*-butyl-3-methylbenzimidamide $SiMe_2[NC(m\text{-MePh})NH(^tBu)]_2$ was prepared by treating $^tBuNH_2$ with one equivalent of $LiBu^n$, *m*-MePhCN, and half equivalent of $SiMe_2Cl_2$ in a one-pot reaction. Treating the ansa-bis(amidine) $SiMe_2[NC(m\text{-MePh})NH(^tBu)]_2$ with $HfCl_4$ in CH_2Cl_2 gave the title compound. Crystals suitable for *X*–ray investigation were obtained by recrystallization from toluene and its molecular structure is presented in Fig. 1. It is a symmetric molecule lying about a 2-fold rotation axis (*e* in Wyckoff notation). The Hf1 and Si1 atoms lie on this axis with all other atoms on general positions. The two amidinate moieties connect the central Si atom with Si–N2 distances 1.762 (3) Å, which matched our original proposal of forming a dianionic N–C–N–Si–N–C–N framework. The structure shows that all the substituents and the silyl bridge are on the same side of the N—C—N skeletons, resulting in two E-anti forms of the amidinate units. The two inner nitrogen atoms bind to Hf1 at a distance of 2.233 (3) Å, and the N2—Hf2—$N2^i$ angle is 69.28 (11)° (i = -*x* + 1, *y*, -*z* + 1/2). The Hf center also exhibits a slightly distorted octahedral geometry.

S2. Experimental

A solution of $LiBu^n$ (2.2 *M*, 2.27 ml, 5.0 mmol) in hexane was added to a stirred solution of $^tBuNH_2$ (0.53 ml, 5.0 mmol) in THF (*ca* 30 ml) by syringe at 273 K. The reaction mixture was warmed to room temperature and kept stirring for 4 h and then *m*-MePhCN (0.59 ml, 5.0 mmol) was added by syringe at 273 K. The reaction mixture was warmed to room temperature and kept stirring for 4 h. Then $SiMe_2Cl_2$ (0.3 ml, 2.55 mmol) was added by syringe at 273 K. After stirring at room temperature for 4 h, it was dried in vacuum to remove all volatiles. The residue was extracted with CH_2Cl_2 (30 ml) and then $HfCl_4$ (0.812 g, 2.5 mmol) was added to this stirred solution at 273 K. The reaction mixture was warmed to room temperature, after stirring for 4 h the solution was dried in vacuum to remove all volatiles. The residue was

dissolved with toluene and then concentrated to yield colorless crystals of the title compound. Yield: 0.422 g (22.3%). ^{1}H NMR (30 MHz, $CDCl_3$) δ (p.p.m.): 8.813 (s, 2H; N*H*), 7.398, 7.204 (d, 8H; *m*-Me*phenyls*), 2.511 (s, 6H; *m-Me*phenyls), 1.034 (s, 18H; ^{t}*Bu*), -0.272 (s, 6H; Si*Me*$_2$). ^{13}C NMR (75 MHz, $CDCl_3$) δ (p.p.m.): 172.566 (N-*C*-N), 139.174–126.831 (*m*-Me*phenyl*), 78.569–77.724 (*m-Me*phenyls), 57.109 (*C*Me$_3$), 32.080, 22.363 (C*Me*$_3$), 3.012 (Si*Me*$_2$). Anal. Calcd. for $C_{26}H_{40}Cl_4HfN_4Si$ (Mr = 757.00): C, 45.25; H, 5.33; N, 3.30%. Found: C, 45.56; H, 5.48; N, 3.44%.

S3. Refinement

The methyl H atoms were constrained to an ideal geometry, with C—H distances of 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C), but each group was allowed to rotate freely about its C–C and C–Si bonds. The amino H atoms were constrained with N—H distances of 0.86 Å and U_{iso}(H) = 1.2U_{eq}(N). The phenyl H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances of 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C).

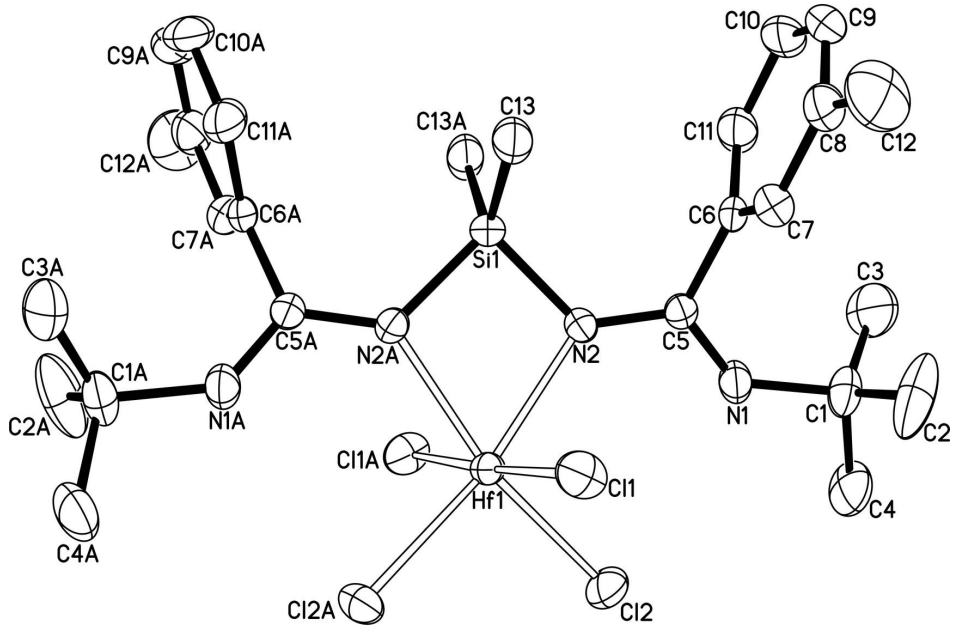

Figure 1

The molecular structure, showing the atom–numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Hydrogen atoms are omitted for clarity.

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Crystal data

$[Hf(C_{26}H_{40}N_4Si)Cl_4]$
M_r = 757
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
a = 9.4373 (14) Å
b = 17.992 (3) Å
c = 19.966 (3) Å
β = 103.276 (3)°
V = 3299.5 (8) Å^3
Z = 4
F(000) = 1512
D_x = 1.524 Mg m^{-3}
Mo *K*α radiation, λ = 0.71073 Å
Cell parameters from 2487 reflections
θ = 2.5–22.1°
μ = 3.54 mm^{-1}
T = 296 K
Block, colorless
0.08 × 0.05 × 0.05 mm

Data collection

Bruker SMART area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.765, T_{max} = 0.843
7121 measured reflections
2920 independent reflections
2446 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 25.1°, θ_{min} = 2.3°
h = −11→7
k = −21→19
l = −23→23

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.061
S = 1.02
2920 reflections
169 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
$w = 1/[\sigma^2(F_o^2) + (0.0266P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
Hf1	0.5000	0.540155 (12)	0.2500	0.03621 (10)
Cl1	0.63951 (14)	0.53649 (6)	0.36889 (6)	0.0584 (3)
Cl2	0.33063 (15)	0.63063 (6)	0.27686 (7)	0.0666 (4)
Si1	0.5000	0.37014 (8)	0.2500	0.0430 (4)
N1	0.2205 (4)	0.47999 (19)	0.33427 (18)	0.0510 (10)
H1	0.2186	0.5176	0.3074	0.061*
N2	0.3964 (3)	0.43808 (16)	0.28086 (16)	0.0363 (8)
C1	0.1179 (5)	0.4877 (3)	0.3814 (2)	0.0589 (13)
C2	0.2003 (7)	0.4884 (4)	0.4547 (3)	0.120 (3)
H2A	0.2335	0.4390	0.4683	0.180*
H2B	0.1379	0.5055	0.4833	0.180*
H2C	0.2825	0.5210	0.4596	0.180*
C3	0.0055 (7)	0.4271 (3)	0.3680 (4)	0.112 (3)
H3A	−0.0378	0.4246	0.3196	0.168*
H3B	−0.0685	0.4375	0.3926	0.168*
H3C	0.0509	0.3804	0.3832	0.168*

C4	0.0435 (7)	0.5621 (3)	0.3634 (3)	0.0891 (19)
H4A	0.1146	0.6012	0.3732	0.134*
H4B	−0.0279	0.5693	0.3901	0.134*
H4C	−0.0033	0.5630	0.3153	0.134*
C5	0.3128 (4)	0.4280 (2)	0.32526 (19)	0.0337 (9)
C6	0.3272 (5)	0.3587 (2)	0.3673 (2)	0.0415 (10)
C7	0.4307 (5)	0.3561 (2)	0.4283 (2)	0.0510 (11)
H7	0.4860	0.3984	0.4429	0.061*
C8	0.4556 (7)	0.2921 (3)	0.4691 (3)	0.0682 (15)
C9	0.3727 (8)	0.2312 (3)	0.4451 (3)	0.089 (2)
H9	0.3867	0.1879	0.4712	0.107*
C10	0.2714 (8)	0.2312 (3)	0.3850 (3)	0.088 (2)
H10	0.2182	0.1883	0.3706	0.105*
C11	0.2455 (6)	0.2957 (2)	0.3440 (3)	0.0652 (14)
H11	0.1760	0.2960	0.3026	0.078*
C12	0.5689 (8)	0.2921 (4)	0.5360 (3)	0.112 (2)
H12A	0.5769	0.2430	0.5554	0.167*
H12B	0.5408	0.3264	0.5674	0.167*
H12C	0.6611	0.3068	0.5276	0.167*
C13	0.6240 (6)	0.3125 (3)	0.3154 (2)	0.0676 (15)
H13A	0.6920	0.2872	0.2943	0.101*
H13B	0.5681	0.2768	0.3340	0.101*
H13C	0.6761	0.3439	0.3517	0.101*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Hf1	0.04771 (16)	0.03029 (14)	0.03166 (14)	0.000	0.01126 (11)	0.000
Cl1	0.0724 (8)	0.0574 (7)	0.0391 (6)	−0.0115 (6)	−0.0002 (5)	0.0005 (5)
Cl2	0.0921 (10)	0.0479 (7)	0.0683 (8)	0.0238 (7)	0.0361 (8)	0.0044 (6)
Si1	0.0556 (11)	0.0302 (8)	0.0490 (11)	0.000	0.0241 (9)	0.000
N1	0.056 (2)	0.056 (2)	0.048 (2)	0.0136 (19)	0.0266 (19)	0.0120 (17)
N2	0.0409 (19)	0.0351 (17)	0.0345 (18)	−0.0027 (15)	0.0123 (16)	0.0020 (14)
C1	0.054 (3)	0.080 (3)	0.049 (3)	0.014 (3)	0.025 (2)	0.000 (2)
C2	0.109 (5)	0.207 (7)	0.044 (4)	0.072 (5)	0.018 (4)	−0.010 (4)
C3	0.096 (5)	0.095 (4)	0.174 (8)	−0.010 (4)	0.093 (5)	−0.014 (5)
C4	0.091 (4)	0.092 (4)	0.100 (5)	0.042 (4)	0.055 (4)	0.014 (3)
C5	0.035 (2)	0.037 (2)	0.028 (2)	−0.0086 (19)	0.0047 (18)	−0.0062 (16)
C6	0.058 (3)	0.036 (2)	0.039 (3)	−0.004 (2)	0.028 (2)	−0.0045 (18)
C7	0.061 (3)	0.047 (3)	0.048 (3)	0.000 (2)	0.020 (2)	0.005 (2)
C8	0.097 (4)	0.062 (3)	0.054 (3)	0.020 (3)	0.035 (3)	0.015 (3)
C9	0.174 (7)	0.041 (3)	0.076 (4)	0.008 (4)	0.076 (5)	0.011 (3)
C10	0.160 (7)	0.041 (3)	0.080 (4)	−0.033 (3)	0.062 (5)	−0.015 (3)
C11	0.085 (4)	0.058 (3)	0.061 (3)	−0.023 (3)	0.032 (3)	−0.013 (3)
C12	0.134 (6)	0.123 (5)	0.072 (5)	0.036 (5)	0.013 (4)	0.039 (4)
C13	0.084 (4)	0.062 (3)	0.071 (4)	0.030 (3)	0.045 (3)	0.025 (3)

Geometric parameters (Å, º)

Hf1—N2	2.233 (3)	C3—H3C	0.9600
Hf1—N2^{i}	2.233 (3)	C4—H4A	0.9600
Hf1—Cl2^{i}	2.4261 (11)	C4—H4B	0.9600
Hf1—Cl2	2.4261 (11)	C4—H4C	0.9600
Hf1—Cl1^{i}	2.4366 (11)	C5—C6	1.491 (5)
Hf1—Cl1	2.4366 (11)	C6—C7	1.377 (6)
Hf1—Si1	3.0588 (16)	C6—C11	1.390 (6)
Si1—N2^{i}	1.762 (3)	C7—C8	1.399 (6)
Si1—N2	1.762 (3)	C7—H7	0.9300
Si1—C13^{i}	1.857 (5)	C8—C9	1.368 (8)
Si1—C13	1.857 (5)	C8—C12	1.507 (8)
N1—C5	1.318 (5)	C9—C10	1.351 (8)
N1—C1	1.504 (5)	C9—H9	0.9300
N1—H1	0.8600	C10—C11	1.409 (7)
N2—C5	1.327 (5)	C10—H10	0.9300
C1—C2	1.491 (7)	C11—H11	0.9300
C1—C3	1.502 (7)	C12—H12A	0.9600
C1—C4	1.516 (7)	C12—H12B	0.9600
C2—H2A	0.9600	C12—H12C	0.9600
C2—H2B	0.9600	C13—H13A	0.9600
C2—H2C	0.9600	C13—H13B	0.9600
C3—H3A	0.9600	C13—H13C	0.9600
C3—H3B	0.9600		

N2—Hf1—N2^{i}	69.32 (16)	H2A—C2—H2C	109.5
N2—Hf1—Cl2^{i}	164.88 (9)	H2B—C2—H2C	109.5
N2^{i}—Hf1—Cl2^{i}	97.96 (9)	C1—C3—H3A	109.5
N2—Hf1—Cl2	97.96 (9)	C1—C3—H3B	109.5
N2^{i}—Hf1—Cl2	164.88 (9)	H3A—C3—H3B	109.5
Cl2^{i}—Hf1—Cl2	95.71 (6)	C1—C3—H3C	109.5
N2—Hf1—Cl1^{i}	94.22 (9)	H3A—C3—H3C	109.5
N2^{i}—Hf1—Cl1^{i}	83.21 (9)	H3B—C3—H3C	109.5
Cl2^{i}—Hf1—Cl1^{i}	92.23 (4)	C1—C4—H4A	109.5
Cl2—Hf1—Cl1^{i}	89.86 (4)	C1—C4—H4B	109.5
N2—Hf1—Cl1	83.21 (9)	H4A—C4—H4B	109.5
N2^{i}—Hf1—Cl1	94.22 (9)	C1—C4—H4C	109.5
Cl2^{i}—Hf1—Cl1	89.86 (4)	H4A—C4—H4C	109.5
Cl2—Hf1—Cl1	92.23 (4)	H4B—C4—H4C	109.5
Cl1^{i}—Hf1—Cl1	176.89 (5)	N1—C5—N2	120.5 (3)
N2—Hf1—Si1	34.66 (8)	N1—C5—C6	119.6 (3)
N2^{i}—Hf1—Si1	34.66 (8)	N2—C5—C6	119.9 (3)
Cl2^{i}—Hf1—Si1	132.14 (3)	C7—C6—C11	119.6 (4)
Cl2—Hf1—Si1	132.14 (3)	C7—C6—C5	118.7 (4)
Cl1^{i}—Hf1—Si1	88.45 (3)	C11—C6—C5	121.5 (4)
Cl1—Hf1—Si1	88.45 (3)	C6—C7—C8	122.2 (5)
N2^{i}—Si1—N2	92.2 (2)	C6—C7—H7	118.9

N2^{i}—Si1—C13^{i}	116.87 (18)	C8—C7—H7	118.9
N2—Si1—C13^{i}	108.80 (19)	C9—C8—C7	116.7 (5)
N2^{i}—Si1—C13	108.80 (19)	C9—C8—C12	122.9 (5)
N2—Si1—C13	116.87 (18)	C7—C8—C12	120.4 (5)
C13^{i}—Si1—C13	112.1 (3)	C10—C9—C8	122.8 (5)
N2^{i}—Si1—Hf1	46.09 (10)	C10—C9—H9	118.6
N2—Si1—Hf1	46.09 (10)	C8—C9—H9	118.6
C13^{i}—Si1—Hf1	123.93 (17)	C9—C10—C11	120.6 (5)
C13—Si1—Hf1	123.93 (17)	C9—C10—H10	119.7
C5—N1—C1	133.8 (4)	C11—C10—H10	119.7
C5—N1—H1	113.1	C6—C11—C10	117.9 (5)
C1—N1—H1	113.1	C6—C11—H11	121.0
C5—N2—Si1	127.0 (3)	C10—C11—H11	121.0
C5—N2—Hf1	131.4 (2)	C8—C12—H12A	109.5
Si1—N2—Hf1	99.25 (14)	C8—C12—H12B	109.5
C2—C1—C3	111.6 (5)	H12A—C12—H12B	109.5
C2—C1—N1	110.4 (4)	C8—C12—H12C	109.5
C3—C1—N1	110.6 (4)	H12A—C12—H12C	109.5
C2—C1—C4	109.5 (5)	H12B—C12—H12C	109.5
C3—C1—C4	109.2 (5)	Si1—C13—H13A	109.5
N1—C1—C4	105.2 (4)	Si1—C13—H13B	109.5
C1—C2—H2A	109.5	H13A—C13—H13B	109.5
C1—C2—H2B	109.5	Si1—C13—H13C	109.5
H2A—C2—H2B	109.5	H13A—C13—H13C	109.5
C1—C2—H2C	109.5	H13B—C13—H13C	109.5

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