

## 6-(1-Methylethyl)-12-phenyl-5,6,7,12-tetrahydrodibenz[c,f][1,5]azasilocene

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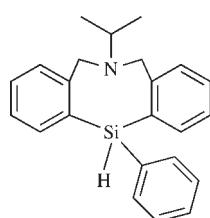
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Key indicators: single-crystal X-ray study;  $T = 120$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.041;  $wR$  factor = 0.100; data-to-parameter ratio = 14.1.

The title compound, C<sub>23</sub>H<sub>25</sub>NSi, has an eight-membered silicon-containing heterocyclic ring with an intramolecular N···Si close contact, the transannular distance of which is 2.6294 (18) Å. The resulting geometry about the Si atom is distorted trigonal-bypyramidal, with the N and H atoms occupying apical sites. The dihedral angle between the aromatic rings fused to the eight-membered ring is 63.27 (7)°.

### Related literature

For highly coordinated organosilanes, see: Brellère *et al.* (1986); Carré *et al.* (1997); Paton *et al.* (1977); Woning & Verkade (1991); Yoshida *et al.* (2006). For a related structure, see: Saruhashi *et al.* (2001).



### Experimental

#### Crystal data

C<sub>23</sub>H<sub>25</sub>NSi  
 $M_r = 343.53$

Monoclinic, P<sub>2</sub><sub>1</sub>/c  
 $a = 9.756(7)$  Å

#### Data collection

Rigaku Mercury CCD diffractometer  
Absorption correction: multi-scan (REQAB; Jacobson, 1998)  
 $T_{\min} = 0.975$ ,  $T_{\max} = 0.987$

11962 measured reflections  
3278 independent reflections  
2798 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.029$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.041$   
 $wR(F^2) = 0.100$   
 $S = 1.08$   
3278 reflections  
232 parameters

H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\max} = 0.30$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.30$  e Å<sup>-3</sup>

Data collection: *CrystalClear* (Rigaku, 2004); 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: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *yadokari-XG* (Wakita, 2005).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2498).

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# supporting information

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## 6-(1-Methylethyl)-12-phenyl-5,6,7,12-tetrahydrodibenz[c,f][1,5]azasilocine

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### S1. Comment

Highly coordinated hydrosilanes have been of great interest for their unique structures and reactivities. It has been known that, in highly coordinated monohydrosilanes, the Si—H bond has high affinity for equatorial position (Brellère *et al.*, 1986) and there are only a few examples with the Si—H bond at the apical position (Woning & Verkade, 1991). A dibenz[c,f][1,5]azasilocine framework has been utilized for the synthesis of various highly coordinated silicon compounds (Paton *et al.*, 1977; Carré *et al.*, 1997; Yoshida *et al.*, 2006). Recently, we reported the synthesis and structural characterization of a pentacoordinated monohydrosilane bearing this molecular framework with the apical Si—H bond (Saruhashi *et al.*, 2001). As a further investigation of this work, the crystal structure of the title new hydrosilane is reported.

The title compound was synthesized by the reaction of *N,N*-bis(2-bromobenzyl)isopropylamine (Carré *et al.*, 1997) with *n*-butyllithium followed by treatment with phenylsilane. The molecular structure of the title compound is shown in Fig. 1. It was found that the geometry around the silicon atom is that of a distorted trigonal bipyramidal with the sum of the equatorial C—Si—C bond angles of 346.3°. The SiH hydrogen atom occupies the apical site in spite of its lower apicophilicity than that of a phenyl group, which is similar to the related *N*-butyl compound we previously reported (Saruhashi *et al.*, 2001). The Si···N transannular distance is 2.6294 (18) Å, which is slightly longer than that of the *N*-butyl derivative [2.516 (2) Å] probably because of the steric repulsion between the isopropyl group and the phenyl ring.

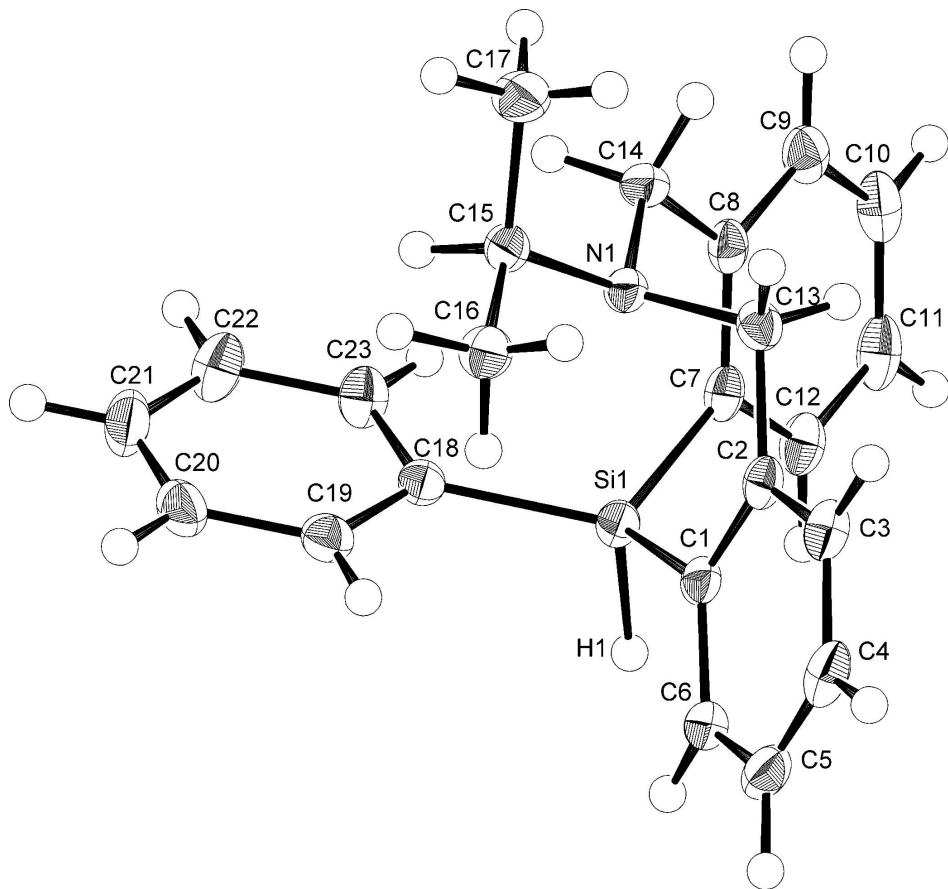
### S2. Experimental

A solution of *n*-butyllithium in hexane (1.6 M; 4.2 ml, 6.7 mmol) was added dropwise to a solution of *N,N*-bis(2-bromobenzyl)isopropylamine (1.25 g, 3.16 mmol) in ether (3 ml) at 233 K. The solution was stirred at the same temperature for 30 min and then allowed to warm to room temperature. After stirring for additional 2 h, the solution was cooled to 233 K, and a solution of phenylsilane (345 mg, 3.19 mmol) in ether (2 ml) was added dropwise. The mixture was allowed to warm to room temperature, and stirred overnight. After addition of water, the mixture was extracted with ether, and the organic layer was dried over anhydrous magnesium sulfate. After filtration and removal of the solvent, the residue was purified by gel permeation liquid chromatography (eluting with chloroform) and then recrystallization from hexane to give the title compound (101 mg, 0.295 mmol, 9.3%) as colorless crystals. Physical data: m.p. 354.1–355.8 K (decomposition);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ , 300 K):  $\delta$  0.76 (br, 6H), 2.53 (br, 1H), 3.78 (s, 4H), 5.53 (brs, 1H), 7.12–7.32 (m, 9H), 7.51 (br, 2H), 7.72 (br, 2H). Anal. Calcd for  $\text{C}_{23}\text{H}_{25}\text{NSi}$ : C 80.41, H 7.34, N 4.08%. Found: C 80.19, H 7.48, N, 3.94%.

### S3. Refinement

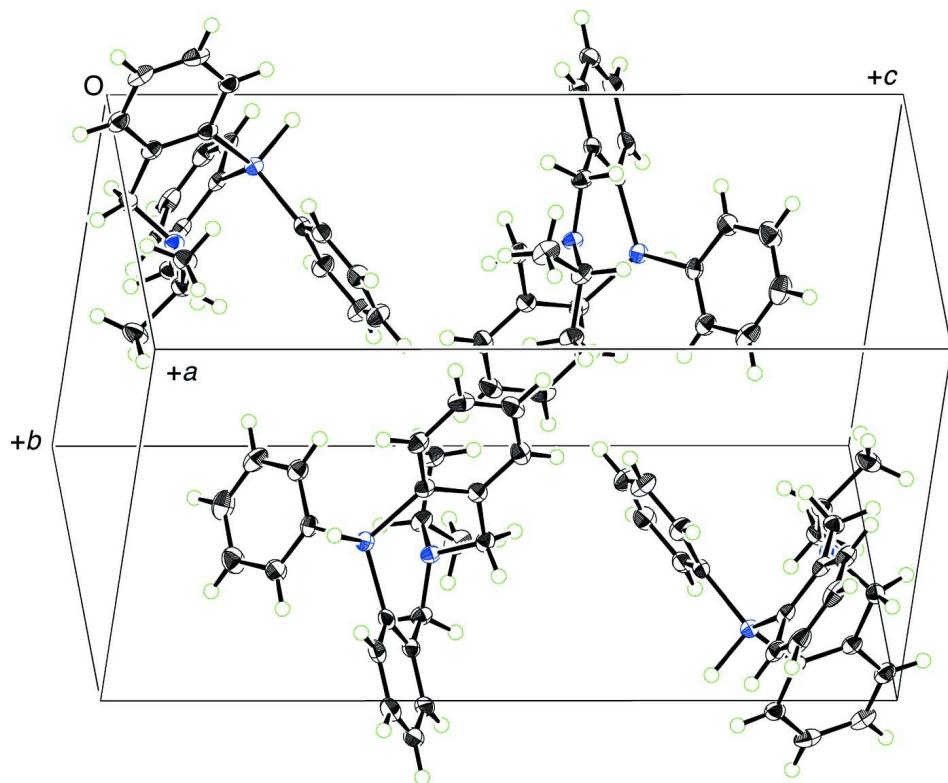
The H atom of the SiH group was found in a difference Fourier map and refined isotropically, while the C-bound H atoms were treated as riding, with C—H = 0.95–0.99 Å, and with  $U_{\text{iso}}(\text{H}) = 1.2$  (1.5 for methyl groups) times  $U_{\text{eq}}(\text{C})$ . The methyl

groups were allowed to rotate freely about the C-C bond.



**Figure 1**

The molecular structure of (I) with 50% probability displacement ellipsoids (arbitrary spheres for H atoms).

**Figure 2**

Packing diagram.

**6-(1-Methylethyl)-12-phenyl-5,6,7,12-tetrahydrobibenz[c,f][1,5]azasilocene***Crystal data*

$C_{23}H_{25}NSi$   
 $M_r = 343.53$   
Monoclinic,  $P2_1/c$   
Hall symbol: -P 2ybc  
 $a = 9.756 (7) \text{ \AA}$   
 $b = 10.269 (7) \text{ \AA}$   
 $c = 18.912 (12) \text{ \AA}$   
 $\beta = 92.745 (3)^\circ$   
 $V = 1893 (2) \text{ \AA}^3$

$Z = 4$   
 $F(000) = 736$   
 $D_x = 1.206 \text{ Mg m}^{-3}$   
Mo  $K\alpha$  radiation,  $\lambda = 0.71070 \text{ \AA}$   
 $\theta = 3.0\text{--}25.0^\circ$   
 $\mu = 0.13 \text{ mm}^{-1}$   
 $T = 120 \text{ K}$   
Block, colourless  
 $0.20 \times 0.20 \times 0.10 \text{ mm}$

*Data collection*

Rigaku Mercury CCD  
diffractometer  
Radiation source: fine-focus sealed tube  
Graphite monochromator  
 $\omega$  scans  
Absorption correction: multi-scan  
(*REQAB*; Jacobson, 1998)  
 $T_{\min} = 0.975$ ,  $T_{\max} = 0.987$

11962 measured reflections  
3278 independent reflections  
2798 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.029$   
 $\theta_{\max} = 25.0^\circ$ ,  $\theta_{\min} = 3.0^\circ$   
 $h = -11 \rightarrow 11$   
 $k = -12 \rightarrow 9$   
 $l = -21 \rightarrow 21$

*Refinement*Refinement on  $F^2$ 

Least-squares matrix: full

$$R[F^2 > 2\sigma(F^2)] = 0.041$$

$$wR(F^2) = 0.100$$

$$S = 1.08$$

3278 reflections

232 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 atoms treated by a mixture of independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0402P)^2 + 1.0221P]$$

where  $P = (F_o^2 + 2F_c^2)/3$

$$(\Delta/\sigma)_{\max} < 0.001$$

$$\Delta\rho_{\max} = 0.30 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.30 \text{ e } \text{\AA}^{-3}$$

*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 ( $\text{\AA}^2$ )*

|     | <i>x</i>      | <i>y</i>      | <i>z</i>      | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|---------------|---------------|---------------|----------------------------------|
| Si1 | 0.10808 (5)   | 0.12541 (5)   | 0.18691 (3)   | 0.02030 (15)                     |
| H1  | 0.0293 (18)   | 0.0512 (18)   | 0.2351 (9)    | 0.023 (5)*                       |
| C1  | 0.14424 (17)  | -0.00037 (17) | 0.11833 (9)   | 0.0204 (4)                       |
| C2  | 0.17207 (17)  | 0.03310 (17)  | 0.04842 (9)   | 0.0210 (4)                       |
| C3  | 0.19436 (18)  | -0.06347 (19) | -0.00083 (10) | 0.0260 (4)                       |
| H2  | 0.2108        | -0.0400       | -0.0483       | 0.031*                           |
| C4  | 0.19306 (18)  | -0.19363 (19) | 0.01801 (11)  | 0.0286 (4)                       |
| H3  | 0.2105        | -0.2589       | -0.0160       | 0.034*                           |
| C5  | 0.16617 (19)  | -0.22838 (18) | 0.08669 (11)  | 0.0297 (5)                       |
| H4  | 0.1659        | -0.3175       | 0.1001        | 0.036*                           |
| C6  | 0.13964 (18)  | -0.13231 (17) | 0.13580 (10)  | 0.0239 (4)                       |
| H5  | 0.1179        | -0.1569       | 0.1824        | 0.029*                           |
| C7  | -0.01585 (18) | 0.25743 (17)  | 0.15815 (9)   | 0.0213 (4)                       |
| C8  | 0.02098 (18)  | 0.36529 (17)  | 0.11797 (9)   | 0.0216 (4)                       |
| C9  | -0.07488 (19) | 0.46221 (18)  | 0.10020 (10)  | 0.0263 (4)                       |
| H6  | -0.0488       | 0.5345        | 0.0725        | 0.032*                           |
| C10 | -0.20760 (19) | 0.4539 (2)    | 0.12252 (10)  | 0.0305 (5)                       |
| H7  | -0.2716       | 0.5213        | 0.1113        | 0.037*                           |
| C11 | -0.24688 (19) | 0.3469 (2)    | 0.16127 (10)  | 0.0311 (5)                       |
| H8  | -0.3385       | 0.3398        | 0.1758        | 0.037*                           |
| C12 | -0.15189 (18) | 0.25012 (19)  | 0.17883 (9)   | 0.0256 (4)                       |
| H9  | -0.1797       | 0.1771        | 0.2055        | 0.031*                           |
| C13 | 0.17620 (17)  | 0.17561 (17)  | 0.02855 (9)   | 0.0223 (4)                       |
| H10 | 0.2334        | 0.1872        | -0.0128       | 0.027*                           |

|     |              |              |              |            |
|-----|--------------|--------------|--------------|------------|
| H11 | 0.0823       | 0.2062       | 0.0153       | 0.027*     |
| C14 | 0.16617 (18) | 0.37943 (17) | 0.09530 (10) | 0.0230 (4) |
| H12 | 0.1655       | 0.4257       | 0.0494       | 0.028*     |
| H13 | 0.2192       | 0.4327       | 0.1306       | 0.028*     |
| N1  | 0.23330 (14) | 0.25313 (14) | 0.08823 (7)  | 0.0196 (3) |
| C15 | 0.38542 (17) | 0.26790 (18) | 0.08917 (10) | 0.0231 (4) |
| H14 | 0.4150       | 0.3025       | 0.1369       | 0.028*     |
| C16 | 0.45915 (18) | 0.13896 (18) | 0.08041 (10) | 0.0267 (4) |
| H15 | 0.4229       | 0.0745       | 0.1128       | 0.040*     |
| H16 | 0.5576       | 0.1508       | 0.0912       | 0.040*     |
| H17 | 0.4445       | 0.1084       | 0.0315       | 0.040*     |
| C17 | 0.4350 (2)   | 0.3648 (2)   | 0.03466 (11) | 0.0331 (5) |
| H18 | 0.4060       | 0.3354       | -0.0130      | 0.050*     |
| H19 | 0.5353       | 0.3705       | 0.0387       | 0.050*     |
| H20 | 0.3954       | 0.4507       | 0.0434       | 0.050*     |
| C18 | 0.25657 (18) | 0.18885 (17) | 0.24348 (9)  | 0.0210 (4) |
| C19 | 0.37831 (18) | 0.11897 (18) | 0.25521 (9)  | 0.0253 (4) |
| H21 | 0.3880       | 0.0367       | 0.2330       | 0.030*     |
| C20 | 0.48570 (19) | 0.1670 (2)   | 0.29869 (10) | 0.0317 (5) |
| H22 | 0.5678       | 0.1178       | 0.3056       | 0.038*     |
| C21 | 0.4733 (2)   | 0.2858 (2)   | 0.33186 (11) | 0.0361 (5) |
| H23 | 0.5466       | 0.3186       | 0.3616       | 0.043*     |
| C22 | 0.3540 (2)   | 0.3568 (2)   | 0.32163 (11) | 0.0356 (5) |
| H24 | 0.3448       | 0.4386       | 0.3444       | 0.043*     |
| C23 | 0.24691 (19) | 0.30841 (19) | 0.27785 (10) | 0.0281 (4) |
| H25 | 0.1652       | 0.3581       | 0.2712       | 0.034*     |

*Atomic displacement parameters ( $\text{\AA}^2$ )*

|     | $U^{11}$    | $U^{22}$    | $U^{33}$    | $U^{12}$    | $U^{13}$      | $U^{23}$     |
|-----|-------------|-------------|-------------|-------------|---------------|--------------|
| Si1 | 0.0192 (3)  | 0.0210 (3)  | 0.0206 (3)  | -0.0018 (2) | -0.00047 (19) | -0.0006 (2)  |
| C1  | 0.0149 (8)  | 0.0215 (9)  | 0.0243 (10) | -0.0014 (7) | -0.0028 (7)   | -0.0001 (8)  |
| C2  | 0.0130 (8)  | 0.0251 (10) | 0.0246 (10) | -0.0005 (7) | -0.0015 (7)   | -0.0029 (8)  |
| C3  | 0.0195 (9)  | 0.0328 (11) | 0.0256 (10) | -0.0031 (8) | -0.0001 (7)   | -0.0060 (8)  |
| C4  | 0.0201 (9)  | 0.0293 (10) | 0.0361 (12) | -0.0003 (8) | -0.0025 (8)   | -0.0141 (9)  |
| C5  | 0.0238 (10) | 0.0184 (9)  | 0.0460 (13) | -0.0011 (8) | -0.0079 (9)   | -0.0037 (9)  |
| C6  | 0.0212 (9)  | 0.0233 (10) | 0.0268 (10) | -0.0031 (7) | -0.0044 (7)   | 0.0023 (8)   |
| C7  | 0.0201 (9)  | 0.0248 (9)  | 0.0189 (9)  | -0.0010 (7) | -0.0019 (7)   | -0.0079 (8)  |
| C8  | 0.0215 (9)  | 0.0220 (9)  | 0.0207 (9)  | 0.0015 (7)  | -0.0046 (7)   | -0.0063 (8)  |
| C9  | 0.0290 (10) | 0.0235 (10) | 0.0256 (10) | 0.0036 (8)  | -0.0055 (8)   | -0.0070 (8)  |
| C10 | 0.0255 (10) | 0.0353 (11) | 0.0297 (11) | 0.0100 (9)  | -0.0081 (8)   | -0.0127 (9)  |
| C11 | 0.0179 (9)  | 0.0450 (12) | 0.0300 (11) | 0.0035 (9)  | -0.0020 (8)   | -0.0149 (10) |
| C12 | 0.0238 (10) | 0.0327 (11) | 0.0203 (10) | -0.0022 (8) | 0.0007 (7)    | -0.0094 (8)  |
| C13 | 0.0188 (9)  | 0.0274 (10) | 0.0206 (10) | -0.0002 (7) | 0.0007 (7)    | 0.0020 (8)   |
| C14 | 0.0234 (9)  | 0.0183 (9)  | 0.0272 (10) | -0.0002 (7) | -0.0011 (7)   | 0.0025 (8)   |
| N1  | 0.0165 (7)  | 0.0190 (8)  | 0.0232 (8)  | -0.0006 (6) | -0.0006 (6)   | 0.0012 (6)   |
| C15 | 0.0162 (9)  | 0.0270 (10) | 0.0261 (10) | -0.0027 (7) | -0.0007 (7)   | 0.0053 (8)   |
| C16 | 0.0172 (9)  | 0.0296 (10) | 0.0334 (11) | 0.0003 (8)  | 0.0029 (8)    | 0.0043 (9)   |

|     |             |             |             |              |             |              |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| C17 | 0.0226 (10) | 0.0357 (11) | 0.0410 (12) | -0.0047 (9)  | 0.0020 (8)  | 0.0129 (10)  |
| C18 | 0.0220 (9)  | 0.0243 (9)  | 0.0168 (9)  | -0.0031 (7)  | 0.0018 (7)  | 0.0027 (8)   |
| C19 | 0.0262 (10) | 0.0259 (10) | 0.0238 (10) | -0.0007 (8)  | 0.0015 (8)  | 0.0040 (8)   |
| C20 | 0.0211 (10) | 0.0394 (12) | 0.0341 (12) | -0.0002 (8)  | -0.0043 (8) | 0.0106 (10)  |
| C21 | 0.0276 (11) | 0.0470 (13) | 0.0328 (12) | -0.0103 (10) | -0.0081 (9) | 0.0000 (10)  |
| C22 | 0.0334 (11) | 0.0378 (12) | 0.0352 (12) | -0.0057 (9)  | -0.0035 (9) | -0.0114 (10) |
| C23 | 0.0233 (10) | 0.0324 (11) | 0.0285 (11) | 0.0012 (8)   | -0.0007 (8) | -0.0047 (9)  |

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

|            |             |             |             |
|------------|-------------|-------------|-------------|
| Si1—C1     | 1.876 (2)   | C13—H10     | 0.9900      |
| Si1—C18    | 1.876 (2)   | C13—H11     | 0.9900      |
| Si1—C7     | 1.880 (2)   | C14—N1      | 1.462 (2)   |
| Si1—H1     | 1.438 (18)  | C14—H12     | 0.9900      |
| C1—C6      | 1.396 (3)   | C14—H13     | 0.9900      |
| C1—C2      | 1.405 (3)   | N1—C15      | 1.491 (2)   |
| C2—C3      | 1.385 (3)   | C15—C16     | 1.520 (3)   |
| C2—C13     | 1.512 (3)   | C15—C17     | 1.528 (3)   |
| C3—C4      | 1.384 (3)   | C15—H14     | 1.0000      |
| C3—H2      | 0.9500      | C16—H15     | 0.9800      |
| C4—C5      | 1.384 (3)   | C16—H16     | 0.9800      |
| C4—H3      | 0.9500      | C16—H17     | 0.9800      |
| C5—C6      | 1.388 (3)   | C17—H18     | 0.9800      |
| C5—H4      | 0.9500      | C17—H19     | 0.9800      |
| C6—H5      | 0.9500      | C17—H20     | 0.9800      |
| C7—C8      | 1.400 (3)   | C18—C23     | 1.394 (3)   |
| C7—C12     | 1.403 (3)   | C18—C19     | 1.396 (3)   |
| C8—C9      | 1.396 (3)   | C19—C20     | 1.391 (3)   |
| C8—C14     | 1.506 (3)   | C19—H21     | 0.9500      |
| C9—C10     | 1.384 (3)   | C20—C21     | 1.380 (3)   |
| C9—H6      | 0.9500      | C20—H22     | 0.9500      |
| C10—C11    | 1.385 (3)   | C21—C22     | 1.380 (3)   |
| C10—H7     | 0.9500      | C21—H23     | 0.9500      |
| C11—C12    | 1.388 (3)   | C22—C23     | 1.393 (3)   |
| C11—H8     | 0.9500      | C22—H24     | 0.9500      |
| C12—H9     | 0.9500      | C23—H25     | 0.9500      |
| C13—N1     | 1.469 (2)   |             |             |
| Si1···N1   | 2.6294 (18) |             |             |
| C1—Si1—C18 | 117.89 (8)  | C2—C13—H11  | 109.6       |
| C1—Si1—C7  | 115.86 (8)  | H10—C13—H11 | 108.1       |
| C18—Si1—C7 | 112.52 (9)  | N1—C14—C8   | 111.83 (14) |
| C1—Si1—N1  | 75.14 (8)   | N1—C14—H12  | 109.2       |
| C18—Si1—N1 | 81.91 (8)   | C8—C14—H12  | 109.2       |
| C7—Si1—N1  | 75.47 (8)   | N1—C14—H13  | 109.2       |
| C1—Si1—H1  | 101.4 (7)   | C8—C14—H13  | 109.2       |
| C18—Si1—H1 | 104.2 (7)   | H12—C14—H13 | 107.9       |

|               |              |               |              |
|---------------|--------------|---------------|--------------|
| C7—Si1—H1     | 102.1 (7)    | C14—N1—C13    | 113.33 (14)  |
| N1—Si1—H1     | 173.9 (7)    | C14—N1—C15    | 111.02 (14)  |
| C6—C1—C2      | 118.03 (16)  | C13—N1—C15    | 113.88 (14)  |
| C6—C1—Si1     | 119.71 (14)  | C14—N1—Si1    | 98.76 (11)   |
| C2—C1—Si1     | 122.24 (14)  | C13—N1—Si1    | 95.92 (11)   |
| C3—C2—C1      | 120.09 (17)  | C15—N1—Si1    | 122.57 (10)  |
| C3—C2—C13     | 121.27 (17)  | N1—C15—C16    | 112.71 (15)  |
| C1—C2—C13     | 118.63 (15)  | N1—C15—C17    | 113.89 (14)  |
| C4—C3—C2      | 120.99 (18)  | C16—C15—C17   | 109.11 (16)  |
| C4—C3—H2      | 119.5        | N1—C15—H14    | 106.9        |
| C2—C3—H2      | 119.5        | C16—C15—H14   | 106.9        |
| C3—C4—C5      | 119.69 (18)  | C17—C15—H14   | 106.9        |
| C3—C4—H3      | 120.2        | C15—C16—H15   | 109.5        |
| C5—C4—H3      | 120.2        | C15—C16—H16   | 109.5        |
| C4—C5—C6      | 119.64 (18)  | H15—C16—H16   | 109.5        |
| C4—C5—H4      | 120.2        | C15—C16—H17   | 109.5        |
| C6—C5—H4      | 120.2        | H15—C16—H17   | 109.5        |
| C5—C6—C1      | 121.51 (18)  | H16—C16—H17   | 109.5        |
| C5—C6—H5      | 119.2        | C15—C17—H18   | 109.5        |
| C1—C6—H5      | 119.2        | C15—C17—H19   | 109.5        |
| C8—C7—C12     | 117.67 (17)  | H18—C17—H19   | 109.5        |
| C8—C7—Si1     | 123.33 (14)  | C15—C17—H20   | 109.5        |
| C12—C7—Si1    | 118.98 (14)  | H18—C17—H20   | 109.5        |
| C9—C8—C7      | 120.56 (17)  | H19—C17—H20   | 109.5        |
| C9—C8—C14     | 119.41 (17)  | C23—C18—C19   | 117.03 (16)  |
| C7—C8—C14     | 120.01 (15)  | C23—C18—Si1   | 120.26 (14)  |
| C10—C9—C8     | 120.55 (19)  | C19—C18—Si1   | 122.68 (14)  |
| C10—C9—H6     | 119.7        | C20—C19—C18   | 121.52 (18)  |
| C8—C9—H6      | 119.7        | C20—C19—H21   | 119.2        |
| C9—C10—C11    | 119.83 (18)  | C18—C19—H21   | 119.2        |
| C9—C10—H7     | 120.1        | C21—C20—C19   | 120.14 (18)  |
| C11—C10—H7    | 120.1        | C21—C20—H22   | 119.9        |
| C10—C11—C12   | 119.74 (18)  | C19—C20—H22   | 119.9        |
| C10—C11—H8    | 120.1        | C22—C21—C20   | 119.69 (18)  |
| C12—C11—H8    | 120.1        | C22—C21—H23   | 120.2        |
| C11—C12—C7    | 121.62 (19)  | C20—C21—H23   | 120.2        |
| C11—C12—H9    | 119.2        | C21—C22—C23   | 119.9 (2)    |
| C7—C12—H9     | 119.2        | C21—C22—H24   | 120.1        |
| N1—C13—C2     | 110.32 (14)  | C23—C22—H24   | 120.1        |
| N1—C13—H10    | 109.6        | C22—C23—C18   | 121.72 (18)  |
| C2—C13—H10    | 109.6        | C22—C23—H25   | 119.1        |
| N1—C13—H11    | 109.6        | C18—C23—H25   | 119.1        |
| <br>          |              |               |              |
| C18—Si1—C1—C6 | 90.22 (15)   | C7—C8—C14—N1  | 27.9 (2)     |
| C7—Si1—C1—C6  | -132.20 (14) | C8—C14—N1—C13 | 68.82 (19)   |
| N1—Si1—C1—C6  | 162.45 (15)  | C8—C14—N1—C15 | -161.54 (14) |
| C18—Si1—C1—C2 | -91.80 (15)  | C8—C14—N1—Si1 | -31.55 (15)  |
| C7—Si1—C1—C2  | 45.78 (17)   | C2—C13—N1—C14 | -142.27 (15) |

|                |              |                 |              |
|----------------|--------------|-----------------|--------------|
| N1—Si1—C1—C2   | -19.57 (13)  | C2—C13—N1—C15   | 89.55 (17)   |
| C6—C1—C2—C3    | -0.2 (2)     | C2—C13—N1—Si1   | -40.07 (14)  |
| Si1—C1—C2—C3   | -178.19 (13) | C1—Si1—N1—C14   | 148.32 (11)  |
| C6—C1—C2—C13   | 179.50 (15)  | C18—Si1—N1—C14  | -89.90 (12)  |
| Si1—C1—C2—C13  | 1.5 (2)      | C7—Si1—N1—C14   | 25.98 (11)   |
| C1—C2—C3—C4    | -1.6 (3)     | C1—Si1—N1—C13   | 33.57 (10)   |
| C13—C2—C3—C4   | 178.74 (16)  | C18—Si1—N1—C13  | 155.34 (11)  |
| C2—C3—C4—C5    | 1.4 (3)      | C7—Si1—N1—C13   | -88.77 (11)  |
| C3—C4—C5—C6    | 0.5 (3)      | C1—Si1—N1—C15   | -89.73 (13)  |
| C4—C5—C6—C1    | -2.4 (3)     | C18—Si1—N1—C15  | 32.04 (13)   |
| C2—C1—C6—C5    | 2.2 (3)      | C7—Si1—N1—C15   | 147.92 (14)  |
| Si1—C1—C6—C5   | -179.79 (13) | C14—N1—C15—C16  | -178.81 (15) |
| C1—Si1—C7—C8   | -79.90 (16)  | C13—N1—C15—C16  | -49.5 (2)    |
| C18—Si1—C7—C8  | 59.91 (17)   | Si1—N1—C15—C16  | 65.15 (18)   |
| N1—Si1—C7—C8   | -14.73 (13)  | C14—N1—C15—C17  | -53.8 (2)    |
| C1—Si1—C7—C12  | 101.44 (15)  | C13—N1—C15—C17  | 75.5 (2)     |
| C18—Si1—C7—C12 | -118.76 (14) | Si1—N1—C15—C17  | -169.85 (12) |
| N1—Si1—C7—C12  | 166.60 (15)  | C1—Si1—C18—C23  | 158.83 (14)  |
| C12—C7—C8—C9   | 0.7 (2)      | C7—Si1—C18—C23  | 19.91 (17)   |
| Si1—C7—C8—C9   | -177.93 (13) | N1—Si1—C18—C23  | 90.44 (15)   |
| C12—C7—C8—C14  | 178.98 (16)  | C1—Si1—C18—C19  | -23.30 (18)  |
| Si1—C7—C8—C14  | 0.3 (2)      | C7—Si1—C18—C19  | -162.22 (14) |
| C7—C8—C9—C10   | 0.6 (3)      | N1—Si1—C18—C19  | -91.69 (15)  |
| C14—C8—C9—C10  | -177.69 (17) | C23—C18—C19—C20 | -0.6 (3)     |
| C8—C9—C10—C11  | -1.7 (3)     | Si1—C18—C19—C20 | -178.53 (14) |
| C9—C10—C11—C12 | 1.4 (3)      | C18—C19—C20—C21 | 0.4 (3)      |
| C10—C11—C12—C7 | -0.1 (3)     | C19—C20—C21—C22 | -0.1 (3)     |
| C8—C7—C12—C11  | -1.0 (3)     | C20—C21—C22—C23 | -0.1 (3)     |
| Si1—C7—C12—C11 | 177.76 (14)  | C21—C22—C23—C18 | 0.0 (3)      |
| C3—C2—C13—N1   | -145.26 (16) | C19—C18—C23—C22 | 0.4 (3)      |
| C1—C2—C13—N1   | 35.1 (2)     | Si1—C18—C23—C22 | 178.38 (15)  |
| C9—C8—C14—N1   | -153.86 (16) |                 |              |