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Crystal structure of (*tert*-butyldimethylsilyl)triphenylgermane, Ph_3Ge-$SiMe_2(t\text{-}Bu)$

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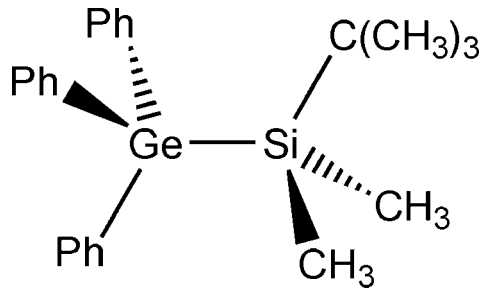
In the title compound, Ph_3Ge-$SiMe_2(t\text{-}Bu)$ or $C_{24}H_{30}GeSi$, the Si and Ge atoms both possess a tetrahedral coordination environment with C—*E*—C (*E* = Si, Ge) angles in the range 104.47 (5)–114.67 (5)°. The molecule adopts an eclipsed conformation, with three torsion angles less than 29.5°. In the crystal, neighbouring molecules are combined to dimers by six T-shaped C—H$\cdots\pi$ interactions, forming sixfold phenyl embraces (6PE).

Keywords: catenated compounds; silagermanes; C—H$\cdots\pi$ interactions; 6PE interactions; crystal structure.

CCDC reference: 1439529

1. Related literature

For general background to the chemistry of Group 14 element catenated compounds, see: Marschner & Hlina (2013); Amadoruge & Weinert (2008); Párkányi *et al.* (1986); Leigh *et al.* (1997). As apart of our studies of the chemistry of oligogermanium compounds (Zaitsev *et al.* 2012, 2013, 2014*a*,*b*), the title compound was obtained and studied. For related crystal structures of silagermanes, see: Zaitsev *et al.* (2015). The 6PE interactions are intensively discussed in Scudder & Dance (2000); Steiner (2000); Churakov *et al.* (2005).



2. Experimental

2.1. Crystal data

$C_{24}H_{30}GeSi$
M_r = 419.16
Monoclinic, *C*2/*c*
a = 13.5332 (6) Å
b = 14.9825 (7) Å
c = 22.7179 (13) Å
β = 106.2048 (10)°
V = 4423.3 (4) Å^3
Z = 8
Mo *K*α radiation
μ = 1.44 mm^{-1}
T = 120 K
0.32 × 0.29 × 0.24 mm

2.2. Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2013)
T_{min} = 0.720, T_{max} = 0.862
32242 measured reflections
7990 independent reflections
6137 reflections with $I > 2\sigma(I)$
R_{int} = 0.043

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.071
S = 1.01
7990 reflections
240 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

Data collection: *APEX2* (Bruker, 2013); cell refinement: *SAINT* (Bruker, 2013); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS2014* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2*.

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: IM2474).

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Acta Cryst. (2015). E**71**, o1015–o1016 [https://doi.org/10.1107/S2056989015022872]

Crystal structure of (*tert*-butyldimethylsilyl)triphenylgermane, Ph_3Ge-$SiMe_2$(*t*-Bu)

Kirill V. Zaitsev, Galina S. Zaitseva, Sergey S. Karlov and Alexander A. Korlyukov

S1. Structural commentary

In the title compound, Ph_3Ge-$SiMe_2$(*t*-Bu), both Si and Ge atoms possess tetrahedral coordination environments with C—E—C angles ranging within 104.47 (5)- 114.67 (5) °. The Ge—Si bond length (2.4026 (4) Å) is slightly longer than in the closely related compound Ph_3Ge-$SiMe_3$ (2.384 (1) Å (Párkányi *et al.*, 1986). The molecule adopts an eclipsed conformation with three torsion angles less than 29.5°.

In the crystal, neighbouring molecules are combined to dimers by six T-shaped C—H···π interactions forming six-fold phenyl embraces (6PE, Steiner, 2000; Churakov *et al.*, 2005). As expected for 6PE-bonded molecules, the C_{ax}—Ge···Ge angle is almost linear - 175.9° (Fig. 2; Scudder & Dance, 2000).

The title compound is isostructural with the corresponding silicon complex Ph_3Si-$SiMe_2$(*t*-Bu) (Leigh *et al.*, 1997).

S2. Synthesis and crystallization

The synthetic procedure leading to the title compound was reported by us earlier (Zaitsev *et al.*, 2014b) to give a white crystalline material in good yield (86 %) by the reaction of Ph_3GeLi (generated *in situ* from equimolar amounts of Ph_3GeH and *n*-BuLi at room temperature in Et_2O) with *t*-$BuMe_2SiCl$ in diethyl ether. Solvent-free crystals of the title compound suitable for X-Ray analysis were obtained after recrystallization from *n*-hexane at room temperature.

S3. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. All non-hydrogen atoms were refined with anisotropic thermal parameters.

All hydrogen atoms were placed in calculated positions and refined using a riding model, with C—H = 0.93–0.96 Å, and with U_{iso}(H) = 1.2 U_{eq}(C) for aromatic hydrogens or 1.5 U_{eq}(C) for methyl H atoms. A rotating model was applied to the methyl groups.

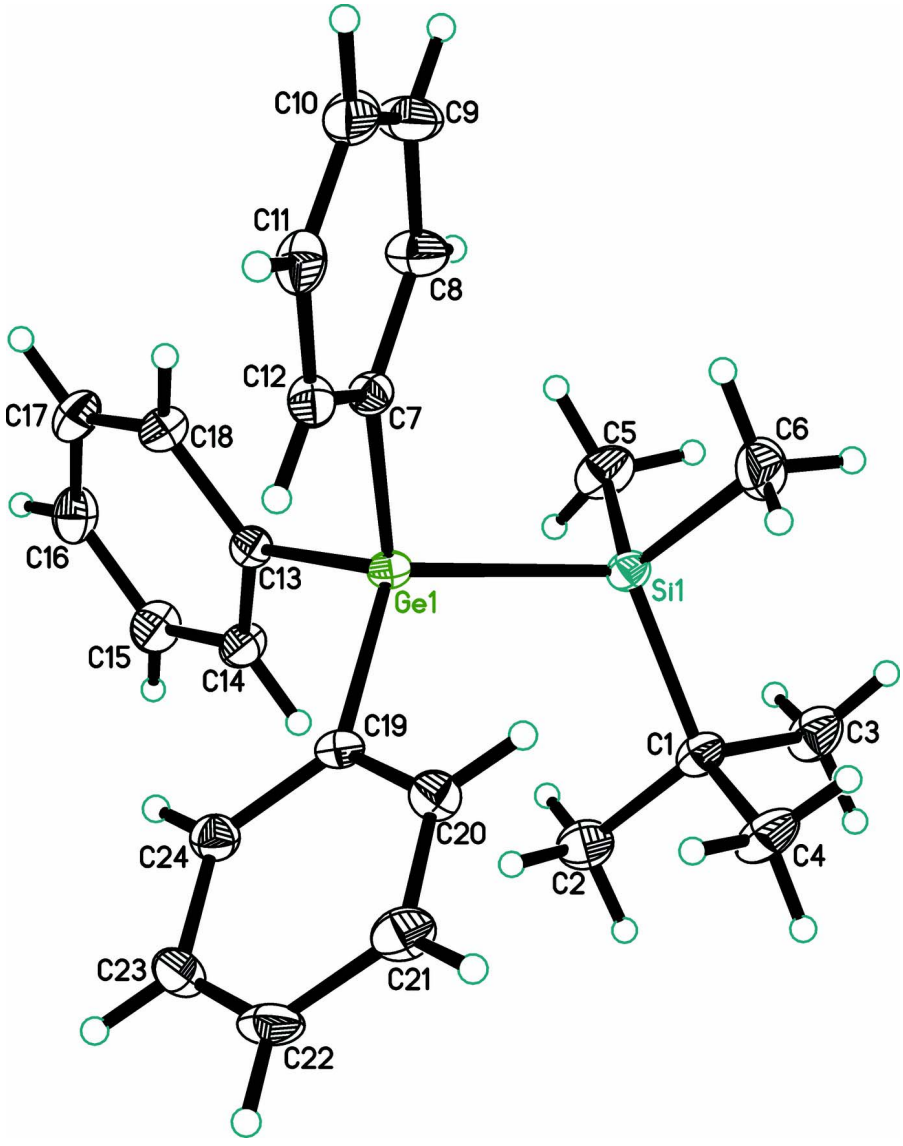

Figure 1

Molecular structure of the title compound, with displacement ellipsoids shown at the 50% probability level.

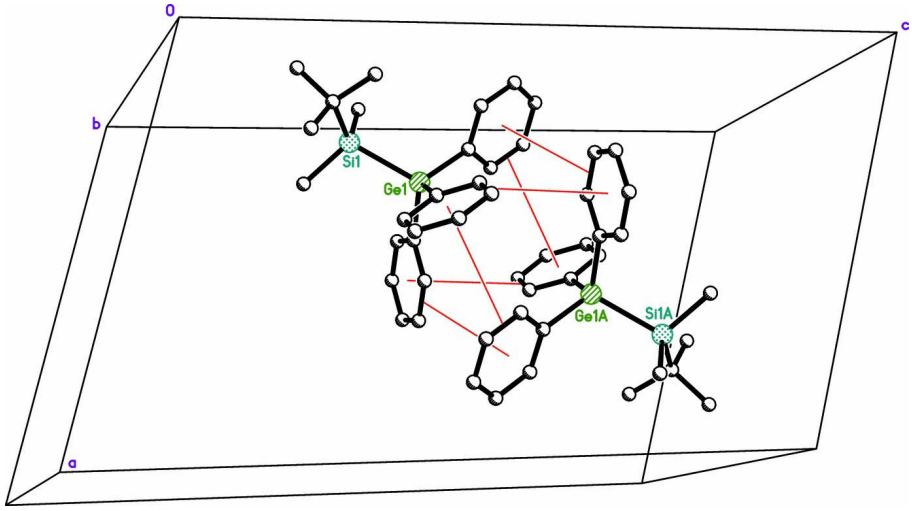

Figure 2

Dimers formed by 6PE interactions between adjacent molecules.

(*tert*-Butyldimethylsilyl)triphenylgermane

Crystal data

$C_{24}H_{30}GeSi$
$M_r = 419.16$
Monoclinic, $C2/c$
$a = 13.5332\ (6)$ Å
$b = 14.9825\ (7)$ Å
$c = 22.7179\ (13)$ Å
$\beta = 106.2048\ (10)°$
$V = 4423.3\ (4)$ Å^3
$Z = 8$
$F(000) = 1760$
$D_x = 1.259$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 8565 reflections
$\theta = 2.5–31.7°$
$\mu = 1.44$ mm^{-1}
$T = 120$ K
Irregular, colourless
0.32 × 0.29 × 0.24 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Radiation source: sealed tube
Graphite monochromator
Detector resolution: 8 pixels mm^{-1}
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2013)
$T_{min} = 0.720$, $T_{max} = 0.862$
32242 measured reflections
7990 independent reflections
6137 reflections with $I > 2\sigma(I)$
$R_{int} = 0.043$
$\theta_{max} = 32.6°$, $\theta_{min} = 1.9°$
$h = -19\rightarrow20$
$k = -22\rightarrow22$
$l = -33\rightarrow34$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.071$
$S = 1.01$
7990 reflections
240 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0303P)^2 + 1.8636P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.43$ e Å^{-3}
$\Delta\rho_{min} = -0.37$ e Å^{-3}

Special details

Experimental. Absorption correctgion: SADABS-2008/1 (Bruker,2008) was used for absorption correction. wR2(int) was 0.0820 before and 0.0431 after correction. The Ratio of minimum to maximum transmission is 0.8344. The $\lambda/2$ correction factor is 0.0015.

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Ge1	0.36354 (2)	0.03558 (2)	0.37248 (2)	0.01369 (4)
Si1	0.26294 (3)	0.07258 (3)	0.27044 (2)	0.01566 (8)
C1	0.19468 (11)	−0.02626 (9)	0.22333 (7)	0.0193 (3)
C2	0.14760 (13)	−0.08808 (11)	0.26222 (8)	0.0280 (3)
H2A	0.2015	−0.1135	0.2947	0.042*
H2B	0.1099	−0.1350	0.2369	0.042*
H2C	0.1020	−0.0544	0.2794	0.042*
C3	0.10842 (13)	0.00982 (12)	0.16951 (8)	0.0286 (4)
H3A	0.0582	0.0399	0.1849	0.043*
H3B	0.0763	−0.0388	0.1437	0.043*
H3C	0.1368	0.0509	0.1462	0.043*
C4	0.26903 (13)	−0.08070 (11)	0.19725 (8)	0.0293 (4)
H4A	0.2967	−0.0432	0.1715	0.044*
H4B	0.2327	−0.1298	0.1737	0.044*
H4C	0.3241	−0.1032	0.2303	0.044*
C5	0.16501 (12)	0.15374 (11)	0.28177 (8)	0.0288 (4)
H5A	0.1152	0.1229	0.2971	0.043*
H5B	0.1311	0.1814	0.2433	0.043*
H5C	0.1984	0.1986	0.3107	0.043*
C6	0.34687 (14)	0.13018 (13)	0.22957 (8)	0.0340 (4)
H6A	0.3806	0.1798	0.2536	0.051*
H6B	0.3056	0.1512	0.1905	0.051*
H6C	0.3976	0.0891	0.2235	0.051*
C7	0.48101 (10)	0.11767 (9)	0.39511 (6)	0.0160 (3)
C8	0.46614 (12)	0.20997 (10)	0.38666 (7)	0.0244 (3)
H8	0.3997	0.2322	0.3713	0.029*
C9	0.54855 (13)	0.26859 (11)	0.40074 (8)	0.0287 (4)
H9	0.5371	0.3295	0.3948	0.034*
C10	0.64792 (12)	0.23684 (11)	0.42370 (7)	0.0258 (3)
H10	0.7032	0.2763	0.4332	0.031*
C11	0.66436 (11)	0.14632 (11)	0.43231 (7)	0.0223 (3)
H11	0.7310	0.1247	0.4475	0.027*
C12	0.58171 (11)	0.08718 (10)	0.41835 (6)	0.0185 (3)
H12	0.5939	0.0264	0.4246	0.022*
C13	0.28202 (11)	0.05273 (9)	0.43088 (6)	0.0159 (3)
C14	0.19262 (11)	0.00334 (10)	0.42642 (7)	0.0210 (3)

H14	0.1719	−0.0391	0.3956	0.025*
C15	0.13399 (12)	0.01638 (11)	0.46710 (7)	0.0254 (3)
H15	0.0750	−0.0174	0.4635	0.031*
C16	0.16361 (12)	0.07993 (11)	0.51311 (7)	0.0252 (3)
H16	0.1242	0.0891	0.5402	0.030*
C17	0.25164 (12)	0.12936 (11)	0.51847 (7)	0.0251 (3)
H17	0.2717	0.1719	0.5494	0.030*
C18	0.31062 (11)	0.11602 (10)	0.47793 (7)	0.0203 (3)
H18	0.3700	0.1497	0.4821	0.024*
C19	0.41975 (10)	−0.08517 (9)	0.38053 (6)	0.0151 (3)
C20	0.47948 (11)	−0.11181 (10)	0.34217 (7)	0.0192 (3)
H20	0.4907	−0.0719	0.3134	0.023*
C21	0.52196 (12)	−0.19630 (10)	0.34629 (7)	0.0238 (3)
H21	0.5611	−0.2128	0.3203	0.029*
C22	0.50633 (12)	−0.25642 (10)	0.38912 (7)	0.0250 (3)
H22	0.5351	−0.3132	0.3921	0.030*
C23	0.44760 (12)	−0.23154 (10)	0.42747 (7)	0.0247 (3)
H23	0.4366	−0.2718	0.4561	0.030*
C24	0.40487 (11)	−0.14636 (10)	0.42328 (7)	0.0194 (3)
H24	0.3659	−0.1302	0.4494	0.023*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ge1	0.01210 (7)	0.01250 (7)	0.01693 (7)	−0.00005 (6)	0.00481 (5)	−0.00168 (6)
Si1	0.01585 (18)	0.01453 (18)	0.01666 (18)	0.00156 (14)	0.00463 (14)	−0.00171 (14)
C1	0.0175 (6)	0.0189 (7)	0.0196 (7)	0.0025 (5)	0.0021 (5)	−0.0043 (5)
C2	0.0260 (8)	0.0266 (8)	0.0293 (8)	−0.0083 (7)	0.0042 (7)	−0.0038 (7)
C3	0.0249 (8)	0.0306 (9)	0.0245 (8)	0.0037 (7)	−0.0025 (6)	−0.0045 (7)
C4	0.0281 (8)	0.0279 (8)	0.0297 (9)	0.0060 (7)	0.0044 (7)	−0.0127 (7)
C5	0.0269 (8)	0.0245 (8)	0.0309 (9)	0.0112 (7)	0.0013 (7)	−0.0060 (7)
C6	0.0410 (10)	0.0391 (10)	0.0243 (8)	−0.0134 (8)	0.0132 (7)	−0.0001 (7)
C7	0.0152 (6)	0.0170 (6)	0.0161 (6)	−0.0019 (5)	0.0050 (5)	−0.0015 (5)
C8	0.0198 (7)	0.0182 (7)	0.0322 (8)	−0.0008 (6)	0.0024 (6)	−0.0008 (6)
C9	0.0291 (8)	0.0182 (7)	0.0356 (9)	−0.0064 (6)	0.0039 (7)	−0.0003 (7)
C10	0.0231 (7)	0.0305 (8)	0.0225 (7)	−0.0123 (6)	0.0040 (6)	−0.0018 (6)
C11	0.0148 (6)	0.0328 (8)	0.0178 (7)	−0.0026 (6)	0.0021 (5)	0.0009 (6)
C12	0.0176 (7)	0.0208 (7)	0.0168 (6)	−0.0004 (5)	0.0044 (5)	0.0014 (5)
C13	0.0151 (6)	0.0158 (6)	0.0167 (6)	0.0016 (5)	0.0044 (5)	−0.0001 (5)
C14	0.0220 (7)	0.0226 (7)	0.0199 (7)	−0.0051 (6)	0.0082 (6)	−0.0044 (6)
C15	0.0220 (7)	0.0318 (9)	0.0251 (8)	−0.0066 (6)	0.0108 (6)	−0.0023 (6)
C16	0.0249 (8)	0.0334 (9)	0.0206 (7)	0.0041 (7)	0.0117 (6)	−0.0005 (6)
C17	0.0277 (8)	0.0277 (8)	0.0200 (7)	0.0009 (6)	0.0069 (6)	−0.0073 (6)
C18	0.0189 (7)	0.0209 (7)	0.0209 (7)	−0.0017 (6)	0.0051 (6)	−0.0037 (6)
C19	0.0135 (6)	0.0123 (6)	0.0189 (6)	−0.0003 (5)	0.0034 (5)	−0.0023 (5)
C20	0.0184 (7)	0.0187 (7)	0.0215 (7)	0.0019 (5)	0.0073 (5)	0.0006 (6)
C21	0.0216 (7)	0.0216 (7)	0.0290 (8)	0.0051 (6)	0.0083 (6)	−0.0045 (6)
C22	0.0256 (8)	0.0124 (6)	0.0327 (8)	0.0033 (6)	0.0010 (6)	−0.0025 (6)

C23	0.0306 (8)	0.0173 (7)	0.0239 (8)	−0.0016 (6)	0.0038 (6)	0.0047 (6)
C24	0.0201 (7)	0.0183 (7)	0.0201 (7)	−0.0017 (6)	0.0060 (5)	−0.0009 (6)

Geometric parameters (Å, º)

Ge1—Si1	2.4026 (4)	C9—H9	0.9300
Ge1—C7	1.9618 (14)	C9—C10	1.384 (2)
Ge1—C13	1.9648 (14)	C10—H10	0.9300
Ge1—C19	1.9512 (13)	C10—C11	1.379 (2)
Si1—C1	1.9078 (15)	C11—H11	0.9300
Si1—C5	1.8687 (15)	C11—C12	1.393 (2)
Si1—C6	1.8670 (17)	C12—H12	0.9300
C1—C2	1.536 (2)	C13—C14	1.398 (2)
C1—C3	1.534 (2)	C13—C18	1.400 (2)
C1—C4	1.537 (2)	C14—H14	0.9300
C2—H2A	0.9600	C14—C15	1.389 (2)
C2—H2B	0.9600	C15—H15	0.9300
C2—H2C	0.9600	C15—C16	1.388 (2)
C3—H3A	0.9600	C16—H16	0.9300
C3—H3B	0.9600	C16—C17	1.379 (2)
C3—H3C	0.9600	C17—H17	0.9300
C4—H4A	0.9600	C17—C18	1.391 (2)
C4—H4B	0.9600	C18—H18	0.9300
C4—H4C	0.9600	C19—C20	1.4019 (19)
C5—H5A	0.9600	C19—C24	1.390 (2)
C5—H5B	0.9600	C20—H20	0.9300
C5—H5C	0.9600	C20—C21	1.383 (2)
C6—H6A	0.9600	C21—H21	0.9300
C6—H6B	0.9600	C21—C22	1.385 (2)
C6—H6C	0.9600	C22—H22	0.9300
C7—C8	1.403 (2)	C22—C23	1.384 (2)
C7—C12	1.3939 (19)	C23—H23	0.9300
C8—H8	0.9300	C23—C24	1.393 (2)
C8—C9	1.385 (2)	C24—H24	0.9300
C7—Ge1—Si1	107.93 (4)	C7—C8—H8	119.4
C7—Ge1—C13	107.92 (6)	C9—C8—C7	121.19 (15)
C13—Ge1—Si1	110.34 (4)	C9—C8—H8	119.4
C19—Ge1—Si1	113.92 (4)	C8—C9—H9	119.9
C19—Ge1—C7	106.89 (6)	C10—C9—C8	120.28 (15)
C19—Ge1—C13	109.61 (6)	C10—C9—H9	119.9
C1—Si1—Ge1	114.67 (5)	C9—C10—H10	120.2
C5—Si1—Ge1	104.47 (5)	C11—C10—C9	119.52 (14)
C5—Si1—C1	109.35 (7)	C11—C10—H10	120.2
C6—Si1—Ge1	109.08 (6)	C10—C11—H11	119.8
C6—Si1—C1	110.26 (7)	C10—C11—C12	120.39 (14)
C6—Si1—C5	108.72 (9)	C12—C11—H11	119.8
C2—C1—Si1	111.05 (10)	C7—C12—H12	119.5

C2—C1—C4	108.84 (13)	C11—C12—C7	121.06 (14)
C3—C1—Si1	108.38 (10)	C11—C12—H12	119.5
C3—C1—C2	109.00 (13)	C14—C13—Ge1	121.31 (10)
C3—C1—C4	108.31 (13)	C14—C13—C18	117.60 (13)
C4—C1—Si1	111.20 (10)	C18—C13—Ge1	121.09 (11)
C1—C2—H2A	109.5	C13—C14—H14	119.3
C1—C2—H2B	109.5	C15—C14—C13	121.38 (14)
C1—C2—H2C	109.5	C15—C14—H14	119.3
H2A—C2—H2B	109.5	C14—C15—H15	120.0
H2A—C2—H2C	109.5	C16—C15—C14	119.93 (15)
H2B—C2—H2C	109.5	C16—C15—H15	120.0
C1—C3—H3A	109.5	C15—C16—H16	120.1
C1—C3—H3B	109.5	C17—C16—C15	119.72 (14)
C1—C3—H3C	109.5	C17—C16—H16	120.1
H3A—C3—H3B	109.5	C16—C17—H17	119.8
H3A—C3—H3C	109.5	C16—C17—C18	120.36 (14)
H3B—C3—H3C	109.5	C18—C17—H17	119.8
C1—C4—H4A	109.5	C13—C18—H18	119.5
C1—C4—H4B	109.5	C17—C18—C13	121.01 (14)
C1—C4—H4C	109.5	C17—C18—H18	119.5
H4A—C4—H4B	109.5	C20—C19—Ge1	118.88 (10)
H4A—C4—H4C	109.5	C24—C19—Ge1	123.24 (10)
H4B—C4—H4C	109.5	C24—C19—C20	117.87 (13)
Si1—C5—H5A	109.5	C19—C20—H20	119.3
Si1—C5—H5B	109.5	C21—C20—C19	121.30 (14)
Si1—C5—H5C	109.5	C21—C20—H20	119.3
H5A—C5—H5B	109.5	C20—C21—H21	120.0
H5A—C5—H5C	109.5	C20—C21—C22	120.06 (14)
H5B—C5—H5C	109.5	C22—C21—H21	120.0
Si1—C6—H6A	109.5	C21—C22—H22	120.2
Si1—C6—H6B	109.5	C23—C22—C21	119.62 (14)
Si1—C6—H6C	109.5	C23—C22—H22	120.2
H6A—C6—H6B	109.5	C22—C23—H23	119.9
H6A—C6—H6C	109.5	C22—C23—C24	120.25 (14)
H6B—C6—H6C	109.5	C24—C23—H23	119.9
C8—C7—Ge1	120.48 (11)	C19—C24—C23	120.90 (14)
C12—C7—Ge1	121.93 (11)	C19—C24—H24	119.6
C12—C7—C8	117.57 (13)	C23—C24—H24	119.6

Ge1—C7—C8—C9	−178.11 (13)	C13—C14—C15—C16	0.4 (2)
Ge1—C7—C12—C11	177.92 (11)	C14—C13—C18—C17	−0.3 (2)
Ge1—C13—C14—C15	−178.96 (12)	C14—C15—C16—C17	−0.5 (3)
Ge1—C13—C18—C17	178.66 (12)	C15—C16—C17—C18	0.2 (2)
Ge1—C19—C20—C21	179.40 (11)	C16—C17—C18—C13	0.2 (2)
Ge1—C19—C24—C23	−179.38 (11)	C18—C13—C14—C15	0.0 (2)
C7—C8—C9—C10	−0.1 (3)	C19—C20—C21—C22	−0.3 (2)
C8—C7—C12—C11	−0.3 (2)	C20—C19—C24—C23	−0.4 (2)
C8—C9—C10—C11	0.2 (2)	C20—C21—C22—C23	0.3 (2)

C9—C10—C11—C12	−0.3 (2)	C21—C22—C23—C24	−0.3 (2)
C10—C11—C12—C7	0.4 (2)	C22—C23—C24—C19	0.4 (2)
C12—C7—C8—C9	0.2 (2)	C24—C19—C20—C21	0.3 (2)