Table 2. *Selected geometric parameters* (Å, °)

O1—C1	1.194 (2)	C5—C6	1.511 (2)
O2—C21	1.353 (2)	C21—C22	1.493 (3)
O2—C2	1.425 (2)	C31—C32	1.482 (4)
O3—C31	1.363 (2)	C41—C42	1.488 (3)
O3—C3	1.431 (2)	C51—C52	1.487 (3)
O4—C41	1.354 (2)	C61—C62	1.484 (3)
O4—C4	1.439 (2)	O1*a*—C1*a*	1.407 (2)
O5—C51	1.343 (2)	O1*c*—C3*a*	1.343 (2)
O5—C5	1.444 (2)	O1*c*—C1*c*	1.449 (3)
O6—C61	1.341 (2)	O3*a*—C3*a*	1.195 (3)
O6—C6	1.440 (2)	C1*a*—C2*a*	1.330 (3)
O21—C21	1.180 (3)	C1*a*—C1*b*	1.471 (3)
O31—C31	1.197 (3)	C2*a*—C3*a*	1.466 (3)
O41—C41	1.197 (3)	C1*b*—C2*b*	1.375 (3)
O51—C51	1.194 (2)	C1*b*—C6*b*	1.385 (3)
O61—C61	1.189 (3)	C2*b*—C3*b*	1.383 (4)
C1—O1*a*	1.343 (2)	C3*b*—C4*b*	1.367 (4)
C1—C2	1.517 (2)	C4*b*—C5*b*	1.367 (4)
C2—C3	1.518 (2)	C5*b*—C6*b*	1.383 (4)
C3—C4	1.533 (2)	C1*c*—C2*c*	1.487 (4)
C4—C5	1.518 (2)		
C21—O2—C2	116.26 (13)	O41—C41—O4	123.1 (2)
C31—O3—C3	117.8 (2)	O41—C41—C42	125.9 (2)
C41—O4—C4	117.84 (13)	O4—C41—C42	110.9 (2)
C51—O5—C5	116.78 (12)	O51—C51—O5	123.8 (2)
C61—O6—C6	116.52 (14)	O51—C51—C52	125.3 (2)
O1—C1—O1*a*	125.0 (2)	O5—C51—C52	110.87 (14)
O1—C1—C2	122.9 (2)	O61—C61—O6	122.0 (2)
O1*a*—C1—C2	111.88 (15)	O61—C61—C62	125.6 (2)
O2—C2—C1	112.81 (13)	O6—C61—C62	112.4 (2)
O2—C2—C3	109.37 (13)	C1—O1*a*—C1*a*	117.72 (15)
C1—C2—C3	111.53 (13)	C3*a*—O1*c*—C1*c*	115.6 (2)
O3—C3—C2	107.67 (12)	C2*a*—C1*a*—O1*a*	120.7 (2)
O3—C3—C4	107.85 (12)	C2*a*—C1*a*—C1*b*	125.9 (2)
C2—C3—C4	111.78 (12)	O1*a*—C1*a*—C1*b*	113.3 (2)
O4—C4—C5	110.93 (11)	C1*a*—C2*a*—C3*a*	125.2 (2)
O4—C4—C3	105.73 (11)	O3*a*—C3*a*—O1*c*	122.9 (2)
C5—C4—C3	111.98 (12)	O3*a*—C3*a*—C2*a*	127.4 (2)
O5—C5—C6	112.28 (12)	O1*c*—C3*a*—C2*a*	109.7 (2)
O5—C5—C4	106.40 (12)	C2*b*—C1*b*—C6*b*	118.6 (2)
C6—C5—C4	115.88 (13)	C2*b*—C1*b*—C1*a*	120.9 (2)
O6—C6—C5	111.16 (12)	C6*b*—C1*b*—C1*a*	120.5 (2)
O21—C21—O2	123.3 (2)	C1*b*—C2*b*—C3*b*	120.6 (2)
O21—C21—C22	126.3 (2)	C4*b*—C3*b*—C2*b*	120.3 (2)
O2—C21—C22	110.4 (2)	C5*b*—C4*b*—C3*b*	119.7 (2)
O31—C31—O3	122.3 (2)	C4*b*—C5*b*—C6*b*	120.3 (2)
O31—C31—C32	127.2 (2)	C1*b*—C6*b*—C5*b*	120.3 (2)
O3—C31—C32	110.5 (3)	O1*c*—C1*c*—C2*c*	107.2 (2)

Data collection and cell refinement: Enraf–Nonius CAD-4 software. Data reduction: *CADSHEL* (Kopf, 1987). Program(s) used to solve structure: *SHELXS*90 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL* (Sheldrick, 1994). Molecular graphics: *SCHAKAL*88 (Keller, 1986). Software used to prepare material for publication: *CIF2TEX* (Kopf, 1992*a*); *FCF2FOC* (Kopf, 1992*b*).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1135). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Spiro[3-germabicyclo[3.1.0]hexane-3,9′-[9]germafluorene]

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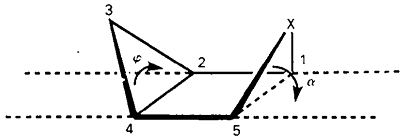
Abstract

The 3-germabicyclo[3.1.0]hexane moiety in the title compound, $C_{17}H_{16}Ge$, adopts a boat form even though the ring bears bulky substituents. This stresses the difference in the structures of germa- *versus* carbocycles.

Comment

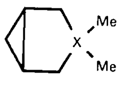
The preference for the boat conformation of the bicyclo[3.1.0]hexane (BCH) ring system, shown in Fig. 1, has been well documented (Mastryukov, Osina, Vikov & Hildebrandt, 1977; Kisiel & Legon, 1978). It was shown that the origin of this rather unexpected conformation is the enhanced eclipsing effect in the chair form, which is larger than the bowsprit–flagpole interaction in the boat form. It is also known, however, that bulky *endo* substituents at C3 will make BCH adopt the chair form (Ahmed & Gabe, 1964).



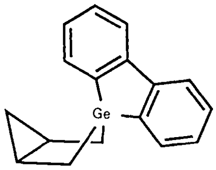
X = C, Ge

Fig. 1. The boat conformation of the bicyclo[3.1.0]hexane ring system. The flap angle α is defined as the angle between the least-squares plane through the atoms C1, C2, C4 and C5 and the plane formed by C1—X—C5. The flap angle φ is defined as the angle between the least-squares plane through the atoms C1, C2, C4 and C5 and the plane formed by C2—C3—C4.

As a part of our extensive investigation of the structures of a variety of germacycles (Takeuchi, 1989), we were interested in the stereochemistry of the 3-germabicyclo[3.1.0]hexane (GBCH) ring system. It is expected that the long C—Ge bond will reduce the bowsprit–flagpole type interaction and that GBCH will remain in a boat form even if bulky substituents are introduced on Ge3. According to our molecular mechanics calculations using the program *MM*2 (Allinger & Yuh, 1977) the boat form is favoured over the chair form by 0.48 kcal mol^{-1} (1 cal = 4.184 J) for 3,3-dimethyl-3-germabicyclo[3.1.0]hexane (2*G*), while the chair form is favoured over the boat form by 1.05 kcal mol^{-1} for 3,3-dimethylbicyclo[3.1.0]hexane (2*C*). This prediction was in accordance with ^{73}Ge NMR spectra for 2*G* and related compounds (Takeuchi, Zicmane, Manuel & Boukherroub, 1993). More definite evidence for the boat form of GBCH being adopted even with bulky substituents attached to the ring system was, however, desirable.



(2*C*) X = C
(2*G*) X = Ge



(I)

The X-ray structure analysis described here clearly demonstrates that the GBCH ring system in spiro[3-germabicyclo[3.1.0]hexane-3,9′-[9]germafluorene], (I), adopts a boat form with flap angles α and φ of 14.2 (2) and 70.9 (3)°, respectively, where α and φ are defined in Fig. 1. Each of these values is almost equal to the corresponding value in 2*G* (18.5 and 69.5°, respectively) calculated using the program *MM*2 (Takeuchi, Zicmane, Manuel & Boukherroub, 1993).

The least-squares planes of the germacyclopentane ring and the germafluorene unit are nearly perpendicular [dihedral angle 92.6 (1)°], as expected. The angle between the plane formed by C1—Ge1—C5 and the Ge1—C6 bond [135.2 (1)°] is almost equal to the angle between this plane and the Ge1—C12 bond [135.6 (1)°]. This may suggest that the bowsprit–flagpole interaction between the *endo* H atom on C3 and the H atom on C11 is insignificant.

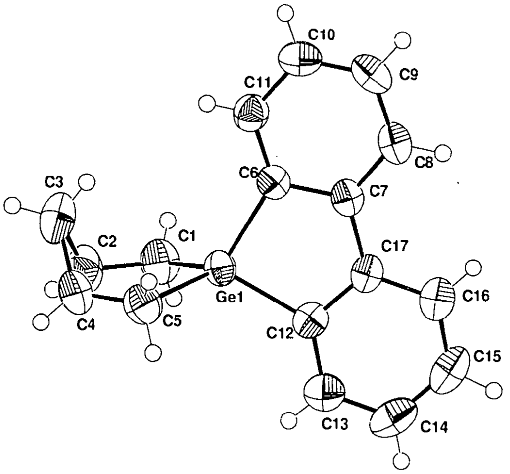


Fig. 2. *ORTEP* (Johnson, 1965) drawing of the title compound. Displacement ellipsoids are shown at the 50% probability level.

Experimental

Crystal data

$C_{17}H_{16}Ge$
M_r = 292.90
Orthorhombic
Pbca
a = 26.191 (2) Å
b = 15.218 (2) Å
c = 6.950 (2) Å
V = 2770 (1) Å^3
Z = 8
D_x = 1.404 Mg m^{-3}

Cu $K\alpha$ radiation
λ = 1.5418 Å
Cell parameters from 25 reflections
θ = 38.26–39.80°
μ = 2.808 mm^{-1}
T = 296.0 K
Prism
0.250 × 0.250 × 0.250 mm
Colourless

Data collection

AFC-6*A* diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical
$T_{\min}$ = 0.923, $T_{\max}$ = 0.973
2403 measured reflections
2047 independent reflections
1628 observed reflections [$I > 3\sigma(I)$]

$R_{\rm int}$ = 0.001
$\theta_{\max}$ = 59.96°
$h = 0 \rightarrow 29$
$k = 0 \rightarrow 0$
$l = -3 \rightarrow 0$
3 standard reflections monitored every 100 reflections intensity variation: −0.23%

Refinement

Refinement on F
R = 0.025
wR = 0.026

$(\Delta/\sigma)_{\max}$ = 0.010
$\Delta\rho_{\max}$ = 0.295 e Å^{-3}
$\Delta\rho_{\min}$ = −0.199 e Å^{-3}

$S = 2.29$
1628 reflections
227 parameters
All H-atom parameters refined
Weighting scheme based on measured e.s.d.'s

Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Ge1	0.62926 (1)	0.50268 (2)	0.86369 (4)	0.0404 (2)
C1	0.6291 (2)	0.3761 (2)	0.8150 (6)	0.058 (3)
C2	0.5974 (2)	0.3383 (2)	0.9738 (6)	0.068 (3)
C3	0.5444 (2)	0.3685 (3)	1.0006 (7)	0.086 (3)
C4	0.5840 (1)	0.3981 (3)	1.1373 (6)	0.068 (2)
C5	0.6022 (1)	0.4915 (2)	1.1242 (5)	0.056 (2)
C6	0.5954 (1)	0.5767 (2)	0.6746 (4)	0.039 (2)
C7	0.6315 (1)	0.6342 (2)	0.5939 (4)	0.041 (2)
C8	0.6156 (1)	0.6927 (2)	0.4535 (5)	0.053 (2)
C9	0.5655 (1)	0.6952 (2)	0.3948 (5)	0.056 (2)
C10	0.5302 (1)	0.6395 (2)	0.4749 (5)	0.053 (2)
C11	0.5454 (1)	0.5802 (2)	0.6137 (5)	0.047 (2)
C12	0.6922 (1)	0.5666 (2)	0.8180 (4)	0.043 (2)
C13	0.7397 (1)	0.5601 (2)	0.9037 (5)	0.057 (2)
C14	0.7791 (1)	0.6136 (3)	0.8455 (6)	0.066 (3)
C15	0.7716 (1)	0.6737 (2)	0.7009 (6)	0.065 (2)
C16	0.7247 (1)	0.6827 (2)	0.6156 (5)	0.055 (2)
C17	0.6843 (1)	0.6288 (2)	0.6732 (4)	0.042 (2)

Table 2. *Selected geometric parameters* (Å, °)

Ge1—C1	1.956 (3)	Ge1—C5	1.952 (3)
Ge1—C6	1.945 (3)	Ge1—C12	1.940 (3)
C1—C2	1.496 (6)	C2—C3	1.474 (7)
C3—C4	1.477 (6)	C2—C4	1.497 (6)
C4—C5	1.502 (5)		
C1—Ge1—C5	94.2 (2)	C6—Ge1—C12	89.2 (1)
Ge1—C1—C2	104.6 (3)	C1—C2—C3	119.7 (4)
C2—C3—C4	61.0 (3)	C3—C4—C5	118.2 (3)
Ge1—C5—C4	104.7 (2)	C1—C2—C4	117.1 (3)
C2—C4—C5	117.0 (3)		
Ge1—C1—C2—C3	57.5 (4)	Ge1—C1—C2—C4	−11.3 (5)
C1—C2—C3—C4	−105.9 (4)	C1—C2—C4—C5	1.9 (5)
C2—C3—C4—C5	106.4 (4)	C2—C4—C5—Ge1	8.7 (4)
C3—C4—C5—Ge1	−59.3 (4)	C4—C5—Ge1—C1	−12.7 (2)
C5—Ge1—C1—C2	13.6 (3)	C1—C2—Ge1—C6	−114.2 (3)
C4—C5—Ge1—C6	112.2 (2)	C1—C2—Ge1—C12	141.2 (3)
C4—C5—Ge1—C12	−138.5 (2)		

Data collection and cell refinement: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1992). Program(s) used to solve structure: *TEXSAN*: *DIRDIF* (Beurskens *et al.*, 1992). Program(s) used to refine structure: *TEXSAN LS*. Software used to prepare material for publication: *TEXSAN FINISH*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AS1113). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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1-(3-Benzoylphenyl)ethanone (I) and 3-Benzoyl-α-methylbenzeneacetamide Methylene Chloride Solvate (2/1) (II)

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Abstract

While the acetyl group in (I) (*m*-benzoylacetophenone, $C_{15}H_{12}O_2$) is nearly coplanar with the phenyl ring [O2—C14—C1—C2 6.8 (5)°] and positioned opposite the C7=O1 group, the 2-propanamido group in (II) [2-(3-benzoylphenyl)propanamide, $C_{16}H_{15}NO_2.0.5CH_2Cl_2$] is almost perpendicular to the corresponding phenyl ring [C16—C14—C1—C2 134.0 (4), C15—C14—C1—C2 −102.4 (5)°] and occupies the same side as the C7=O1 group.

Comment

The compounds 1-(3-benzoylphenyl)ethanone (I) and 3-benzoyl-α-methylbenzeneacetamide (III) are intermediates (Shimizu, Matsumura & Arai, 1988; Cerbelaud &