Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.118$
$S = 1.063$
2351 reflections
175 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0341P)^2 + 0.2969P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.21$ e Å^{-3}
$\Delta\rho_{\min} = -0.25$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected torsion angles (°)*

C7—O2—C2—C3	−26.5 (3)	C10—O6—C6—C5	−9.8 (3)
C7—O3—C3—C2	4.1 (3)	O5—C5—C6—O6	27.4 (3)
O2—C2—C3—O3	13.6 (3)	C2—O2—C7—O3	29.5 (3)
C2—C3—C4—C5	−179.8 (2)	C3—O3—C7—O2	−20.4 (3)
C10—O5—C5—C6	−35.5 (3)	C5—O5—C10—O6	30.1 (3)
C3—C4—C5—C6	178.8 (2)	C6—O6—C10—O5	−11.7 (3)

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1′···O6^{i}	0.86 (3)	1.96 (4)	2.815 (3)	173 (4)
O4—H4′···O1^{ii}	0.85 (3)	1.98 (3)	2.784 (3)	157 (3)

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

H atoms were located from ΔF synthesis and refined with OH free, rigid methyls and other H atoms riding.

Data collection: *DIF*4 (Stoe & Cie, 1992*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1992*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *XP* (Siemens, 1994). Software used to prepare material for publication: *SHELXL*97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1272). Services for accessing these data are described at the back of the journal.

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A Tricyclic Trisiladiene

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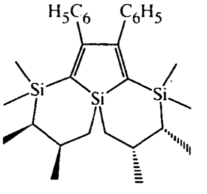
Abstract

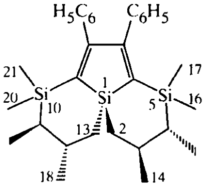
rel-(3*S*,4*R*,11*R*,12*S*)-7,8-Diphenyl-3,4,5,5,10,10,11,12-octamethyl-1,5,10-trisilatricyclo[7.4.0.0^{1,6}]trideca-6,8-diene, $C_{30}H_{42}Si_3$, crystallizes in the space group $P\bar{1}$ and has two independent molecules in the asymmetric unit. The methyl groups in both cyclohexane rings are in quasi-diequatorial positions.

Comment

As part of our interest in Si-containing polycyclic compounds we have investigated the Cp_2Zr-induced cyclizations (Cp is cyclopentadienyl) of diallyl divinyl siloles (Teng *et al.*, 1997). When silole (1), with two allyl substituents at the central Si atom and two vinyl groups at each of the peripheral Si atoms, was treated with a threefold excess of Cp_2ZrCl_2/n-BuLi at a temperature below 273 K, a tricyclic compound was obtained as a single stereoisomer. We had reported that the compound isolated has C_2 symmetry with the vicinal methyl groups *cis* with respect to each other. This stereochemical assignment was based on ^{1}H NMR spectra. In order to verify these conclusions, the X-ray structure analysis of this compound, (2), was carried out.



(1)



(2)

The structure analysis clearly shows that the vicinal methyl groups in both cyclohexane rings are in quasi-diequatorial, *i.e. trans* positions with respect to each other, rather than in axial-equatorial positions (Fig. 1). This is in contrast to the experience that Cp_2Zr-induced reactions of 1,7-octadienes lead preferentially, at low temperature, to *cis*-dimethylcyclohexanes (Rousset *et*

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al., 1989; Taber *et al.*, 1994). The space group $P\bar{1}$ is compatible with the postulated C_2 symmetry of the molecule and a *cis* as well as a *trans* arrangement of the vicinal methyl groups. The structure analysis provides unequivocal evidence for the *trans*-diequatorial arrangement of the vicinal methyl groups in both cyclohexane rings (Fig. 1).

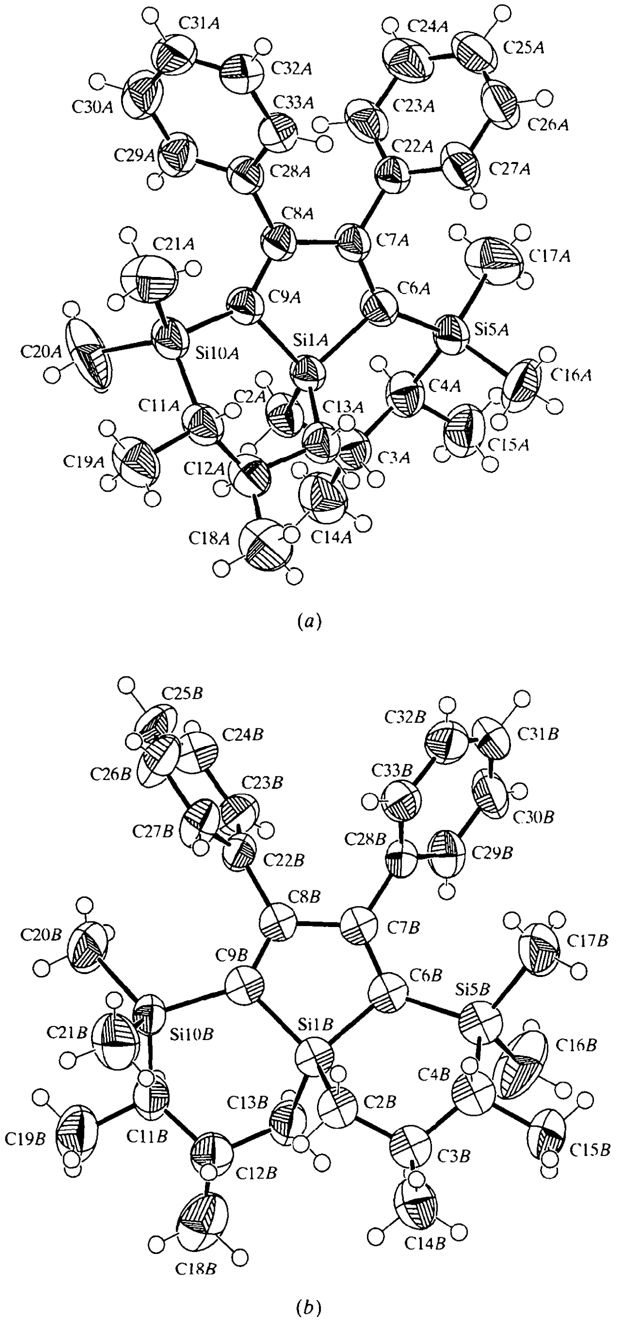

Fig. 1. The molecular structures of (*a*) molecule *A* and (*b*) molecule *B* of (2) (ellipsoids are shown at the 50% probability level).

In the crystalline state, the asymmetric unit contains two independent molecules (*A* and *B*), each with approximate C_2 symmetry. The silacyclopentadiene rings are planar within 0.02 Å in both independent molecules. The two cyclohexane rings have slightly twisted chair conformations in both molecules, with the vicinal methyl groups in quasi-equatorial positions. The best planes are through the groups of atoms C2, C3, Si5, C6 and C9, Si10, C12, C13, which are planar within 0.15 Å in both molecules. Atoms Si1 and C4 are displaced from the former plane by −0.743 and 0.604 Å in molecule *A*, and by −0.695 and 0.539 Å in molecule *B*. Atoms Si1 and C11 are displaced from the latter plane by −0.539 and 0.684 Å in molecule *A* and by −0.602 and 0.741 Å in molecule *B*. The intra-ring bond angle C6—Si1—C9 [94.23 (11)° in *A* and 94.06 (11)° in *B*] is much smaller than those in the cyclohexane rings; C4—Si5—C6 [105.89 (11)° in *A* and 106.92 (11)° in *B*] and C9—Si10—C11 [106.59 (11)° in *A* and 106.02 (11)° in *B*]. The bond angle C2—Si1—C13 is 109.14 (13)° in *A* and 109.33 (13)° in *B*, and thus is larger than its counterpart C6—Si1—C9. Similar relationships were observed for the bond angle C16—Si5—C17 [109.71 (19)° in *A* and 112.2 (2)° in *B*], which is larger than its intracyclic counterpart C9—Si10—C11.

Experimental

Compound (2) was prepared according to Teng *et al.* (1997). The resulting oil dissolved in pentane and a trace of methanol upon warming. On cooling to room temperature, colourless block-like crystals were obtained.

Crystal data

$C_{30}H_{42}Si_3$
$M_r = 486.99$
Triclinic
$P\bar{1}$
$a = 12.6170 (11)$ Å
$b = 13.5333 (15)$ Å
$c = 18.8645 (16)$ Å
$\alpha = 79.129 (12)°$
$\beta = 79.320 (10)°$
$\gamma = 73.004 (11)°$
$V = 2995.9 (5)$ Å^3
$Z = 4$
$D_x = 1.080$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 5000 reflections
$\theta = 2.22$–$25.91°$
$\mu = 0.138$ mm^{-1}
$T = 293 (2)$ K
Block
0.57 × 0.49 × 0.15 mm
Colourless

Data collection

Stoe image-plate diffractometer
φ rotation scans
Absorption correction: none
28 398 measured reflections
10 834 independent reflections

6211 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.035$
$\theta_{\rm max} = 25.91°$
$h = -15 \rightarrow 15$
$k = -16 \rightarrow 16$
$l = -23 \rightarrow 23$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.129$
$S = 0.860$
10 834 reflections
612 parameters
H atoms riding
$w = 1/[\sigma^2(F_o^2) + (0.0807P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.068$
$\Delta\rho_{\max} = 0.547$ e Å^{-3}
$\Delta\rho_{\min} = -0.405$ e Å^{-3}
Extinction correction: *SHELXL*97 (Sheldrick, 1997*b*)
Extinction coefficient: 0.0047 (10)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Si1*A*—C9*A*	1.854 (3)	Si1*B*—C9*B*	1.860 (2)
Si1*A*—C13*A*	1.864 (3)	Si1*B*—C13*B*	1.862 (3)
Si1*A*—C6*A*	1.865 (2)	Si1*B*—C6*B*	1.853 (3)
Si1*A*—C2*A*	1.866 (3)	Si1*B*—C2*B*	1.864 (3)
C2*A*—C3*A*	1.548 (4)	C2*B*—C3*B*	1.549 (3)
C3*A*—C4*A*	1.537 (4)	C3*B*—C4*B*	1.532 (4)
C4*A*—Si5*A*	1.905 (3)	C4*B*—Si5*B*	1.896 (3)
Si5*A*—C6*A*	1.857 (3)	Si5*B*—C6*B*	1.861 (2)
Si5*A*—C16*A*	1.861 (3)	Si5*B*—C16*B*	1.851 (4)
Si5*A*—C17*A*	1.865 (3)	Si5*B*—C17*B*	1.880 (4)
C6*A*—C7*A*	1.355 (4)	C6*B*—C7*B*	1.358 (3)
C7*A*—C8*A*	1.520 (3)	C7*B*—C8*B*	1.518 (3)
C8*A*—C9*A*	1.360 (3)	C8*B*—C9*B*	1.352 (4)
C9*A*—Si10*A*	1.862 (2)	C9*B*—Si10*B*	1.860 (2)
Si10*A*—C20*A*	1.854 (3)	Si10*B*—C20*B*	1.869 (3)
Si10*A*—C21*A*	1.864 (4)	Si10*B*—C21*B*	1.853 (3)
Si10*A*—C11*A*	1.906 (3)	Si10*B*—C11*B*	1.898 (3)
C11*A*—C12*A*	1.532 (4)	C11*B*—C12*B*	1.556 (4)
C12*A*—C13*A*	1.543 (3)	C12*B*—C13*B*	1.549 (4)
C9*A*—Si1*A*—C6*A*	94.23 (11)	C6*B*—Si1*B*—C9*B*	94.06 (11)
C13*A*—Si1*A*—C6*A*	121.76 (11)	C6*B*—Si1*B*—C13*B*	120.20 (12)
C9*A*—Si1*A*—C13*A*	106.88 (12)	C9*B*—Si1*B*—C13*B*	105.71 (11)
C6*A*—Si1*A*—C2*A*	105.45 (11)	C6*B*—Si1*B*—C2*B*	106.24 (11)
C9*A*—Si1*A*—C2*A*	119.63 (11)	C9*B*—Si1*B*—C2*B*	121.55 (11)
C13*A*—Si1*A*—C2*A*	109.14 (13)	C13*B*—Si1*B*—C2*B*	109.33 (13)
C3*A*—C2*A*—Si1*A*	113.24 (16)	C3*B*—C2*B*—Si1*B*	112.82 (18)
C4*A*—C3*A*—C2*A*	113.0 (2)	C4*B*—C3*B*—C2*B*	113.0 (2)
C3*A*—C4*A*—Si5*A*	118.04 (18)	C3*B*—C4*B*—Si5*B*	119.26 (18)
C16*A*—Si5*A*—C17*A*	109.71 (19)	C16*B*—Si5*B*—C17*B*	112.2 (2)
C6*A*—Si5*A*—C4*A*	105.89 (11)	C6*B*—Si5*B*—C4*B*	106.92 (11)
C7*A*—C6*A*—Si1*A*	106.31 (16)	C7*B*—C6*B*—Si1*B*	106.98 (16)
C7*A*—C6*A*—Si5*A*	132.98 (19)	C7*B*—C6*B*—Si5*B*	131.8 (2)
Si5*A*—C6*A*—Si1*A*	117.25 (14)	Si1*B*—C6*B*—Si5*B*	118.13 (14)
C6*A*—C7*A*—C8*A*	116.5 (2)	C6*B*—C7*B*—C8*B*	115.7 (2)
C9*A*—C8*A*—C7*A*	116.1 (2)	C9*B*—C8*B*—C7*B*	116.8 (2)
C8*A*—C9*A*—Si10*A*	132.3 (2)	C8*B*—C9*B*—Si10*B*	133.16 (18)
C8*A*—C9*A*—Si1*A*	106.72 (17)	C8*B*—C9*B*—Si1*B*	106.38 (16)
Si1*A*—C9*A*—Si10*A*	118.12 (14)	Si10*B*—C9*B*—Si1*B*	117.28 (15)
C20*A*—Si10*A*—C21*A*	111.6 (2)	C21*B*—Si10*B*—C20*B*	110.13 (19)
C9*A*—Si10*A*—C11*A*	106.59 (11)	C9*B*—Si10*B*—C11*B*	106.02 (11)
C12*A*—C11*A*—Si10*A*	119.34 (19)	C12*B*—C11*B*—Si10*B*	118.10 (17)
C11*A*—C12*A*—C13*A*	113.9 (2)	C13*B*—C12*B*—C11*B*	112.9 (2)
C12*A*—C13*A*—Si1*A*	113.05 (17)	C12*B*—C13*B*—Si1*B*	113.25 (18)

The two independent molecules (*A* and *B*) in the asymmetric unit not only possess pseudo-twofold symmetry but are also related by a pseudo-twofold screw axis. A thorough search for a suitable monoclinic cell, using the programs *LEPAGE* (Spek, 1988) and *DELOS* (Burzlaff & Zimmerman, 1985), for example, gave no satisfactory result. H atoms were included in calculated positions and treated as riding atoms.

Data collection: *EXPOSE* (Stoe & Cie, 1996). Cell refinement: *CELL* (Stoe & Cie, 1996). Data reduction: *INTEGRATE* (Stoe & Cie, 1996). Program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997*a*). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*b*). Molecular graphics: *Xtal_GX* (Hall & du Boulay, 1997). Software used to prepare material for publication: *SHELXL*97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: AB1538). Services for accessing these data are described at the back of the journal.

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4-Cyano-4′-octylbiphenyl

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Abstract

The title compound, 4′-octylbiphenyl-4-carbonitrile, $C_{21}H_{25}N$, has the phase sequence crystal–smectic A (S_A)–nematic–isotropic. In the crystal structure, infinite chains with a close arrangement of CN groups are found.

Comment

The title compound and its homologues, 4′-alkyl-4-cyanobiphenyl (*n*CB), have been widely studied as key examples of liquid crystalline behaviour, because of the accessibility of their liquid crystalline phases (low melting points), their stable properties and their simple molecular structures (Gray, 1998).

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