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cis-1,2,2,3-Tetraphenyl-2,3-dihydro-1*H*-2-benzosilole

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The title compound, $C_{32}H_{26}Si$, is a benzosilacyclopentene in which the silacyclopentene ring assumes an envelope conformation, with the Si atom displaced by 0.953 (2) Å from the mean plane of the four C atoms. The Si atom is bonded to two phenyl rings and to two Csp^3 atoms in the silacyclopentene ring; one phenyl ring is attached to each of these two Csp^3 atoms in the *cis* configuration.

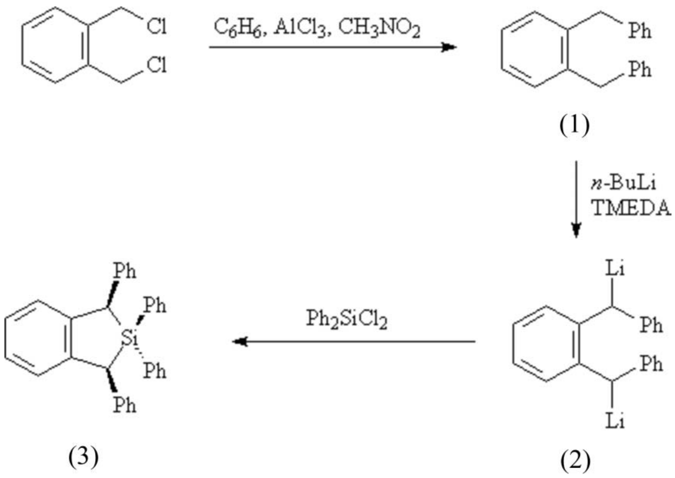
Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.003 Å
R factor = 0.035
wR factor = 0.101
Data-to-parameter ratio = 15.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

In the title compound, (3) (Fig. 1), the Si atom is bonded to two phenyl rings and to two Csp^3 atoms in the silacyclopentene ring; one phenyl ring is attached to each of these two Csp^3 atoms in the *cis* configuration. The average Si—phenyl bond distance is 1.869 (2) Å. The average Si—Csp^3 bond distance is 1.902 (2) Å, and the corresponding C—Si—C angle is 90.68 (7)°. The silacyclopentene ring assumes an envelope conformation, with the Si atom displaced by 0.953 (2) Å from the mean plane of the four C atoms.



The only structure in the Cambridge Structural Database (Version 5.27, ConQuest Version 1.8; Allen, 2002) having the same fused ring system as (3) is 2,2-diphenyl-2-silaindan (Vidal & Falgueirettes, 1973), which has H atoms on the silacyclopentene ring where (3) has phenyl rings. The Si—phenyl distances in 2,2-diphenyl-2-silaindan are nearly identical, at 1.876 and 1.875 Å. The average Si—Csp^3 distance is 1.886 Å, and the corresponding C—Si—C angle is 93.59°. The silacyclopentene ring assumes an envelope conformation, with the Si atom displaced by 0.636 Å from the mean plane of the four C atoms.

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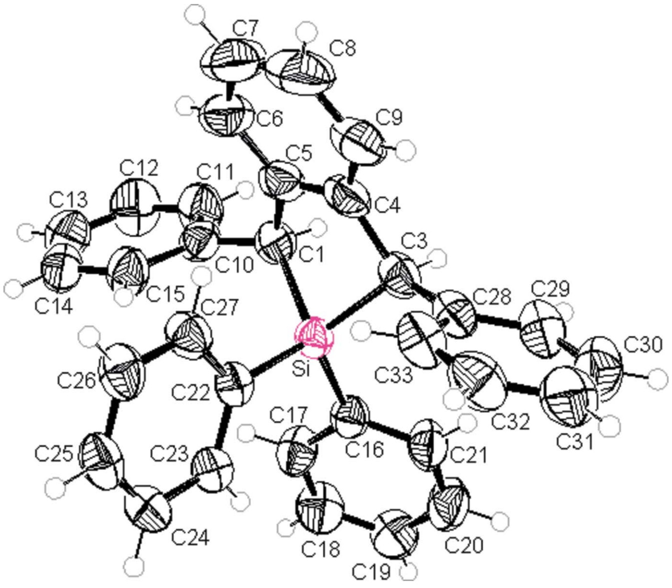

Figure 1
The molecular structure of (3), showing 50% probability displacement ellipsoids.

Experimental

Compound (3) was prepared in a model reaction for the preparation of new phosphine ligands of similar structure *via* dicarbanions (Bates *et al.*, 1981). The two-step synthesis is outlined below.

The first step prepares the carbon diacid, (1), by Friedel–Crafts alkylation of benzene with α,α'-dichloro-*o*-xylene (Mataka *et al.*, 1981). The carbon acid is bislithiated using two equivalents of *n*-butyllithium and tetramethylethylenediamine (TMEDA) to give the deep-red dicarbanion, (2) (Sato *et al.*, 2001), which, upon reaction with diphenyldichlorosilane, gives the title compound, (3). The major isomer is the crystalline *cis*-isomer. The compound has been also characterized by both ^{1}H and ^{13}C NMR spectroscopy.

Compound (1), the precursor to the title compound, was prepared by charging a dry 250 ml two-necked round-bottomed flask, equipped with a stirrer bar, a reflux condenser and a gas inlet adapter, with α,α'-dichloro-*o*-xylene (50 mmol, 8.75 g), dry benzene (100 ml) and nitromethane (10 ml) under N_2. Subsequently, $AlCl_3$ (20.0 g, 150 mmol) was added to the flask. The nitrogen line was removed and replaced with a drying tube, and the mixture was allowed to react for 30 min without external heating. External heat was then applied and the mixture was refluxed overnight. The reaction was then quenched with water and the phases separated. The organic layer was dried with Na_2SO_4. After solvent removal under a vacuum, the residue was purified by bulb–bulb distillation (393 K, 1 Pa) Further purification was accomplished by recrystallization from isooctane (11.2 g, 87% yield; m.p. 346–347 K).

For the preparation of (3), compound (1) (0.52 g, 2.0 mmol) was placed in an oven-dried vial equipped with a stirrer bar. After purging with N_2, a septum was attached. Dry degassed Et_2O (15 ml) was introduced *via* a syringe. A positive pressure of N_2 gas was maintained as the vial was cooled to 273 K, and TMEDA (0.51 g, 4.4 mmol) followed by *n*-BuLi (1.61 ml, 2.74 *M*, solvent = hexanes, 4.4 mmol) were introduced *via* a syringe. A deep-red color was observed almost immediately upon addition of the *n*-BuLi. The reaction was quenched after 30 min with dichlorodiphenylsilane (0.56 g, 2.2 mmol). Saturated aqueous NH_4Cl was added 30 min later. The organic layer was separated and dried with Na_2SO_4. It was then reduced under vacuum and purified by bulb–bulb distillation (433 K, 1 Pa) to give 0.82 g of (3) (94% yield; m.p. 408–409 K). X-ray quality crystals of (3) were obtained by recrystallization from ethanol.

Crystal data

$C_{32}H_{26}Si$
$M_r = 438.62$
Triclinic, $P\overline{1}$
$a = 9.877$ (3) Å
$b = 10.633$ (2) Å
$c = 13.693$ (2) Å
$\alpha = 67.620$ (14)°
$\beta = 84.657$ (18)°
$\gamma = 66.77$ (2)°
$V = 1219.2$ (5) Å^3
$Z = 2$
$D_x = 1.195$ Mg m^{-3}
Cu $K\alpha$ radiation
$\mu = 0.96$ mm^{-1}
$T = 295$ (2) K
Prism, colorless
0.50 × 0.30 × 0.25 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
7796 measured reflections
4604 independent reflections
4113 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.013$
$\theta_{\rm max} = 69.9°$
3 standard reflections every 94 reflections intensity decay: 3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.101$
$S = 1.03$
4604 reflections
299 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0506P)^2 + 0.2363P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.23$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.20$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.0250 (10)

Table 1
Selected geometric parameters (Å, °).

Si—C16	1.8611 (15)	Si—C1	1.9007 (15)
Si—C22	1.8777 (15)	Si—C3	1.9038 (15)
C16—Si—C22	109.70 (6)	C16—Si—C3	115.92 (7)
C16—Si—C1	115.01 (7)	C22—Si—C3	112.55 (6)
C22—Si—C1	111.95 (6)	C1—Si—C3	90.68 (7)

H atoms were constrained using a riding model. The aromatic C—H bond lengths were fixed at 0.93 Å and the methine C—H bond lengths at 0.98 Å, with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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