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syn-*exo*-4,7-Bis(trimethylsilyl)-4,7-dihydro[2.2]paracyclophane

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Key indicators

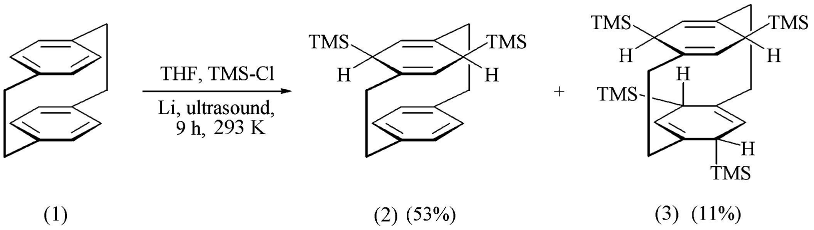
Single-crystal X-ray study
T = 173 K
Mean *σ*(C–C) = 0.005 Å
R factor = 0.055
wR factor = 0.164
Data-to-parameter ratio = 17.3

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

In the title compound, $C_{22}H_{34}Si_2$, the two independent molecules are closely similar. The reduced rings show a flattened twist conformation. Several features of [2.2]paracyclophane strain (*e.g.* lengthened bridge bonds) are apparent; an exception is furnished by the normal *sp*^{2} values of the angles at the bridgehead atoms of the reduced rings.

Comment

In our studies concerning the partial and total hydrogenation of [2.2]paracyclophane, (1), we subjected this layered hydrocarbon to a Birch reduction in the presence of trimethylsilyl chloride and obtained the title compound, (2), and the doubly reduced derivative (3). Whereas we reported the full spectroscopic and analytical data of these products in our original publication (Hopf *et al.*, 1997), their structures have not been described previously; we present the structure of (2) here and that of (3) in the following paper (Jones *et al.*, 2005).



The structure of (2) is shown in Fig. 1. The asymmetric unit contains two independent molecules that are closely similar; a least-squares fit of C atoms 1–16 has an r.m.s. deviation of only 0.034 Å (Fig. 2).

The reduced rings display flattened twist conformations with significantly non-zero torsion angles about the double bonds, the positions of which are clearly established by bond lengths and angles (Table 1) and substitution patterns. The molecules show features of strain that are typical of [2.2]paracyclophane systems, *e.g.* the bridgehead distances C3···C14 = 2.805 (5) and 2.795 (5) Å, and C6···C11 = 2.778 (5) and 2.778 (5) Å, the lengthened single bonds and widened angles in the bridges. In the reduced rings, however, the bond angles at the bridgehead atoms are no longer significantly narrower than the ideal 120°. The non-reduced rings show the typical flattened boat conformation, with the bridgehead atoms C11, C11′, C14 and C14′ lying 0.144 (5), 0.146 (5), 0.152 (5) and 0.141 (5) Å, respectively, out of the planes of the remaining four ring atoms.

A search of the Cambridge Structural Database (Version 5.26; Allen, 2002) revealed only three other structures with the same reduced paracyclophane ring system (Vorontsova *et al.*, 2002). These were obtained by reduction of the corresponding quinone. In contrast to the current structure, the reduced rings displayed boat conformations with the sp^3 atoms out of plane.

Experimental

The title compound was prepared as described by Hopf *et al.* (1997). Single crystals were grown by sublimation.

Crystal data

$C_{22}H_{34}Si_2$
$M_r = 354.67$
Monoclinic, $P2_1/c$
$a = 10.485$ (4) Å
$b = 33.972$ (8) Å
$c = 12.544$ (4) Å
$\beta = 102.12$ (3)°
$V = 4369$ (2) Å^3
$Z = 8$
$D_x = 1.079$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 50 reflections
$\theta = 10$–11°
$\mu = 0.16$ mm^{-1}
$T = 173$ (2) K
Tablet, colourless
0.7 × 0.5 × 0.2 mm

Data collection

Nicolet R3 diffractometer
ω scans
Absorption correction: none
7846 measured reflections
7710 independent reflections
4194 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.031$
$\theta_{\rm max} = 25.1°$
$h = -12 \rightarrow 0$
$k = -40 \rightarrow 0$
$l = -14 \rightarrow 14$
3 standard reflections every 147 reflections intensity decay: 5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.164$
$S = 0.99$
7710 reflections
445 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.065P)^2 + 3.2193P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.34$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.28$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—C2	1.578 (5)	C1′—C2′	1.576 (5)
C3—C8	1.335 (5)	C3′—C8′	1.328 (5)
C5—C6	1.334 (5)	C5′—C6′	1.321 (5)
C9—C10	1.579 (5)	C9′—C10′	1.572 (5)
C14—C1—C2	111.3 (3)	C14′—C1′—C2′	111.0 (3)
C3—C2—C1	113.0 (3)	C3′—C2′—C1′	113.5 (3)
C8—C3—C4	119.8 (3)	C8′—C3′—C4′	120.1 (3)
C3—C4—C5	110.3 (3)	C3′—C4′—C5′	110.0 (3)
C5—C6—C7	120.6 (3)	C5′—C6′—C7′	120.2 (3)
C6—C7—C8	110.0 (3)	C6′—C7′—C8′	109.6 (3)
C6—C9—C10	113.1 (3)	C6′—C9′—C10′	113.9 (3)
C11—C10—C9	110.2 (3)	C11′—C10′—C9′	110.5 (3)
C8—C3—C4—C5	−29.7 (4)	C8′—C3′—C4′—C5′	28.0 (4)
C3—C4—C5—C6	18.4 (5)	C3′—C4′—C5′—C6′	−17.2 (5)
C4—C5—C6—C7	10.3 (5)	C4′—C5′—C6′—C7′	−11.2 (5)
C5—C6—C7—C8	−26.8 (4)	C5′—C6′—C7′—C8′	27.8 (4)
C4—C3—C8—C7	13.3 (5)	C4′—C3′—C8′—C7′	−11.2 (5)
C6—C7—C8—C3	15.3 (5)	C6′—C7′—C8′—C3′	−17.0 (5)

Methyl H atoms were identified in difference syntheses, idealized and then refined using rigid methyl groups (C—H = 0.98 Å and H—C—H = 109.5°) allowed to rotate but not tip. Other H atoms were included using a riding model, with C—H = 0.95 (sp^2), 0.99 (CH_2) or 1.00 Å (methine); $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C).

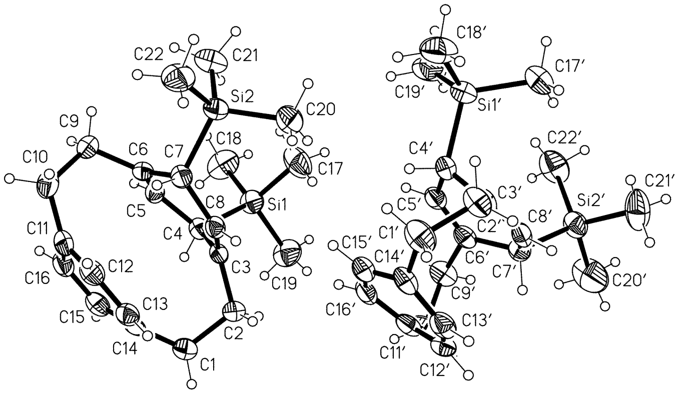


Figure 1
The two independent molecules of compound (2) in the crystal structure. Displacement ellipsoids are drawn at the 50% probability level.

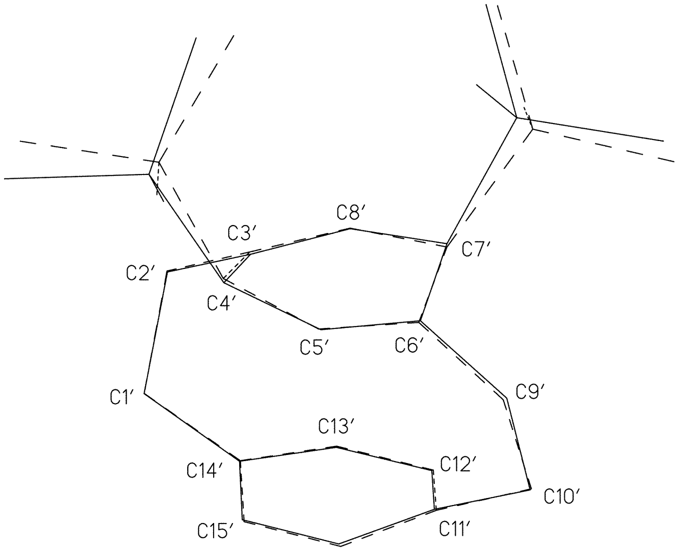


Figure 2
Least-squares fit of the two independent molecules of compound (2).

Data collection: *P3* (Nicolet, 1987); cell refinement: *P3*; data reduction: *XDISK* (Nicolet, 1987); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL97*.

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