Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

syn-*exo*-4,7,12,15-Tetrakis(trimethylsilyl)-4,7,12,15-tetrahydro[2.2]paracyclophane

Peter G. Jones,[a]* Peter Bubenitschek,[b] Henning Hopf[b] and Rainer Savinsky[b]

[a]Institut für Anorganische und Analytische Chemie, Technische Universität Braunschweig, Postfach 3329, 38023 Braunschweig, Germany, and [b]Institut für Organische Chemie, Technische Universität Braunschweig, Postfach 3329, 38023 Braunschweig, Germany

Correspondence e-mail: p.jones@tu-bs.de

Key indicators

Single-crystal X-ray study
T = 143 K
Mean σ(Wae) = 0.004 Å
R factor = 0.040
wR factor = 0.114
Data-to-parameter ratio = 14.6

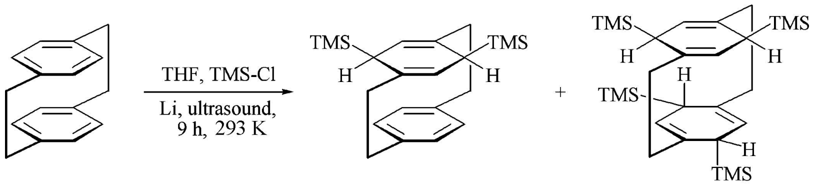
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_{28}H_{52}Si_4$, crystallizes in the uncommon space group $I4_122$ with 222 symmetry. Despite the reduction of the rings, several features of [2.2]paracyclophane strain are still apparent.

Received 7 June 2005
Accepted 9 June 2005
Online 17 June 2005

Comment

In the previous paper (Jones *et al.*, 2005), in which introductory material can be found, we have presented the structure of compound (2) in the scheme. We present here the structure of the doubly reduced compound (3), which was formed in the same reaction.



(1) (2) (53%) (3) (11%)

The structure of (3) is shown in Fig. 1. It crystallizes in the space group $I4_122$, which is uncommon; a search of the Cambridge Structural Database (Version 5.26; Allen, 2002) revealed only 23 other examples. The molecule displays crystallographic symmetry 222, and the numbering of the asymmetric unit therefore cannot conform to the IUPAC numbering of the whole molecule.

The reduced rings display flattened and somewhat distorted twist conformations with significantly non-zero torsion angles about the double bonds C2═C4. The molecule, despite the reduction of both rings, still shows show features of strain that are typical of [2.2]paracyclophane systems, *e.g.* the bridgehead distance C2···C2^{i} = 2.816 (5) Å [symmetry code: (i) $\frac{1}{2}-y, \frac{1}{2}-x, \frac{3}{2}-z$] the lengthened single bonds and widened angles in the bridges.

Experimental

The title compound was prepared as described by Hopf *et al.* (1997). Single crystals were grown by evaporation of a solution in *n*-hexane.

Crystal data

$C_{28}H_{52}Si_4$
M_r = 501.06
Tetragonal, $I4_122$
a = 13.906 (4) Å
c = 16.263 (5) Å
V = 3144.9 (16) Å^3
Z = 4
D_x = 1.058 Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 66 reflections
θ = 10–11.5°
μ = 0.20 mm^{-1}
T = 143 (2) K
Block, colourless
0.6 × 0.6 × 0.5 mm

Data collection

Stoe Stadi-4 diffractometer
ω/θ scans
Absorption correction: none
3911 measured reflections
1068 independent reflections
936 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.031$

$\theta_{\rm max} = 27.5°$
$h = 0 \rightarrow 18$
$k = -18 \rightarrow 0$
$l = -21 \rightarrow 21$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.114$
$S = 1.07$
1068 reflections
73 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0668P)^2 + 1.0612P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.23$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.14$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—C1$^{\rm i}$	1.571 (5)	C2—C3	1.506 (4)
C2—C4	1.326 (4)		
C2—C1—C1$^{\rm i}$	112.36 (15)	C2—C3—C4	109.7 (2)
C4—C2—C3	120.2 (2)	C2—C4—C3	124.8 (2)
C4—C2—C3—C4	28.7 (3)	C2—C3—C4—C2	−18.9 (3)
C3—C2—C4—C3	−10.2 (4)		

Symmetry codes: (i) $-y+\frac{1}{2}, -x+\frac{1}{2}, -z+\frac{3}{2}$.

Methyl H atoms were placed in ideally staggered positions and then refined using a riding model, with C—H 0.98 Å and H—C—H = 109.5°. 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). Refinement with Friedel opposite reflections treated as independent led to a Flack (1983) parameter insignificantly different from 0.5 (indicating inversion twinning) and Friedel opposite reflections were therefore merged for the final refinement. To improve stability of refinement in view of the moderate data/parameter ratio, anisotropic displacement parameters were subject to similarity and rigid-bond restraints.

Data collection: *DIF4* (Stoe & Cie, 1992); cell refinement: *DIF4*; data reduction: *REDU4* (Stoe & Cie, 1992); 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*.

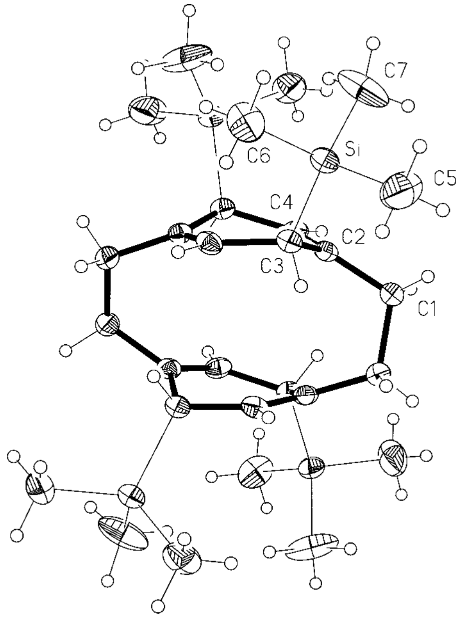


Figure 1
The molecule of compound (3) in the crystal structure. Displacement ellipsoids are drawn at the 30% probability level.

We thank Mr A. Weinkauf for technical assistance.

References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Hopf, H., Savinsky, R., Diesselkämper, B., Daniels, R. G. & de Meijere, A. (1997). *J. Org. Chem.* **62**, 8941–8943.
Jones, P. G., Bubenitschek, P., Hopf, H. & Savinsky, R. (2005). *Acta Cryst.* E**61**, o2175–o2176..
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL97*. Univ. of Göttingen, Germany.
Siemens (1994). *XP*. Version 5.03. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Stoe & Cie (1992). *DIF4* and *REDU4*. Stoe & Cie, Darmstadt, Germany.

o2178 Jones *et al.* • $C_{28}H_{52}Si_4$ *Acta Cryst.* (2005). E**61**, o2177–o2178