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3-Cyclopropyl-3-(*cis*-1-cyclopropyl-2-phenylcyclopropyl)-1-phenylcyclopropene

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Key indicators

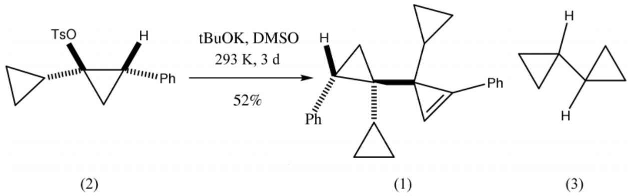
Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.002 Å
R factor = 0.048
wR factor = 0.117
Data-to-parameter ratio = 16.3

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

The structure of the first oligo 1,1-cyclopropane, $C_{24}H_{24}$, is reported. All the three-membered rings are *gauche* with respect to each other. The molecules are connected by a network of C—H···C interactions.

Comment

The title compound, (1), was obtained unexpectedly as the main product (in 52% yield) in an attempted preparation of phenylbicyclopropylidene *via* dehydrotosylation of (*E*)-1-cyclopropyl-2-phenyl-1-*p*-tolylsulfonyloxycaclopropane, (2).



Surprisingly, the structures of oligo 1,1-cyclopropanes are virtually unknown, except the first member of the series, bicyclopropyl, (3) (Nijveldt & Vos, 1988), and its derivatives. Several cyclic 1,1-cyclopropanes, or [*n*]rotanes, are also known (Boese, 1992). Theoretical calculations on bicyclopropyl predict that the two most favourable conformations, with almost equal energy, are *trans* and *gauche* (Yeh & Saebo, 1995). These conformations are defined by H—C—C—H torsion angles of 180 and 60°, respectively. In (1), all three-membered rings are *gauche* with respect to each other. The corresponding torsion angles are listed in Table 1. The 'backbone' of the molecule also has a *gauche* conformation, with a C10—C3—C4—C7 torsion angle of 64.1 (1)°. The mutual orientation of the phenyl groups at C1 and C5 and the three-membered rings is the most efficient one for conjugation of the π-system of the aromatic ring with the cyclopropyl and cyclopropene rings; the orientation is perpendicular in the case of phenyl ring C19–C24 and in the plane of the unsaturated cyclopropene ring for phenyl ring C13–C18 (Shen *et al.*, 2001)

Molecule (1) does not have any functional groups capable of forming strong intermolecular interactions such as hydrogen bonds. However, several types of weaker intermolecular interactions, formed by aromatic rings, are well known (Hunter *et al.*, 2001; Steiner, 2002), as well as the ability of the cyclopropane ring to participate in similar interactions (Allen *et al.*, 1996). Indeed, close C—H···C contacts exist in the crystal structure of (1). The geometrical parameters of these closest intermolecular interactions are listed in Table 2. All types of H atoms (aromatic, secondary and tertiary)

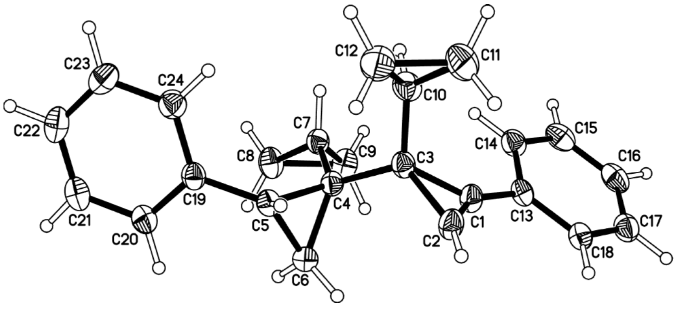


Figure 1
The molecular structure and labelling scheme of (1). Displacement ellipsoids are drawn at the 50% probability level.

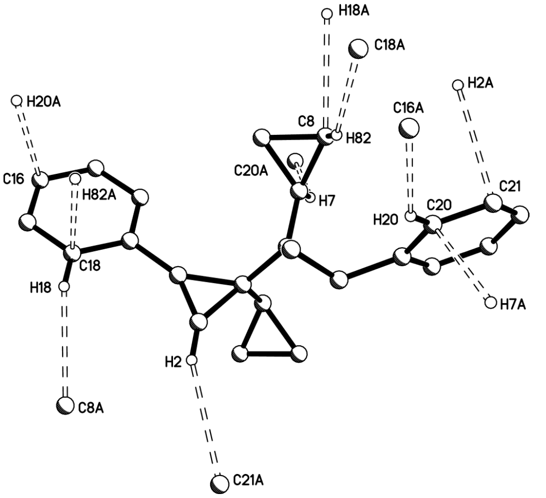


Figure 2
The directionality of the short intermolecular contacts. For details, see Table 2.

participate in the interactions but in most cases an aromatic C atom is the acceptor in these contacts, with the donor C—H bonds almost normal to the plane of the phenyl ring. No π–π stacking is observed in (1). These weak interactions form a three-dimensional network in the crystal structure of (1).

Experimental

Crystals of (1) were obtained by slow evaporation from an Et_2O solution.

Crystal data

$C_{24}H_{24}$
$M_r = 312.43$
Monoclinic, $P2_1/c$
$a = 8.5291$ (2) Å
$b = 15.6301$ (4) Å
$c = 13.6004$ (4) Å
$\beta = 97.562$ (1)°
$V = 1797.31$ (8) Å^3
$Z = 4$

$D_x = 1.155$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6424 reflections
$\theta = 2.4$–29.6°
$\mu = 0.07$ mm^{-1}
$T = 120.0$ (2) K
Plate, colourless
0.35 × 0.26 × 0.05 mm

Data collection

Bruker SMART CCD 1K diffractometer
ω scans
Absorption correction: none
21455 measured reflections
5105 independent reflections

3547 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.062$
$\theta_{\rm max} = 30.4°$
$h = -11 \rightarrow 12$
$k = -21 \rightarrow 21$
$l = -19 \rightarrow 18$

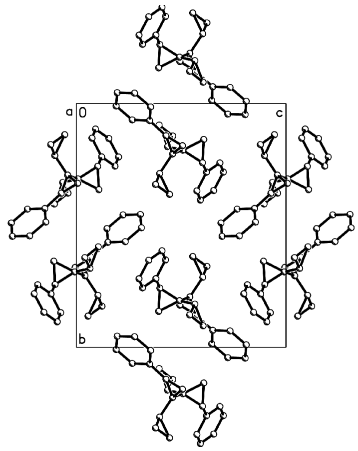


Figure 3
Packing of the molecules of (1) in the crystal structure, viewed along the *a* axis. H atoms have been omitted for clarity.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.117$
$S = 1.02$
5105 reflections
313 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.045P)^2 + 0.45P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.26$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.22$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—C2	1.2989 (18)	C5—C19	1.4999 (17)
C1—C13	1.4559 (17)	C5—C6	1.5126 (18)
C1—C3	1.5107 (18)	C7—C8	1.5036 (18)
C2—C3	1.5156 (17)	C7—C9	1.5105 (18)
C3—C10	1.5109 (18)	C8—C9	1.507 (2)
C3—C4	1.5180 (17)	C10—C11	1.499 (2)
C4—C7	1.5048 (17)	C10—C12	1.507 (2)
C4—C6	1.5074 (17)	C11—C12	1.499 (2)
C4—C5	1.5269 (17)		
C2—C1—C13	150.39 (12)	C3—C4—C5	116.28 (10)
C2—C1—C3	64.78 (9)	C19—C5—C6	122.37 (11)
C13—C1—C3	143.98 (11)	C19—C5—C4	121.55 (11)
C1—C2—C3	64.39 (9)	C6—C5—C4	59.46 (8)
C1—C3—C10	118.24 (11)	C4—C6—C5	60.74 (8)
C1—C3—C2	50.83 (8)	C8—C7—C4	124.29 (11)
C10—C3—C2	119.60 (11)	C8—C7—C9	59.99 (9)
C1—C3—C4	120.47 (11)	C4—C7—C9	120.57 (11)
C10—C3—C4	113.57 (10)	C7—C8—C9	60.23 (9)
C2—C3—C4	120.30 (11)	C8—C9—C7	59.77 (9)
C7—C4—C6	119.63 (11)	C11—C10—C12	59.82 (11)
C7—C4—C3	113.37 (10)	C11—C10—C3	120.96 (13)
C6—C4—C3	118.86 (10)	C12—C10—C3	120.79 (13)
C7—C4—C5	118.85 (10)	C10—C11—C12	60.38 (10)
C6—C4—C5	59.80 (8)	C11—C12—C10	59.80 (10)
C1—C3—C4—C7	−84.76 (14)	C1—C3—C10—C11	−55.46 (18)
C10—C3—C4—C7	64.05 (14)	C2—C3—C10—C11	3.2 (2)
C2—C3—C4—C7	−144.44 (11)	C4—C3—C10—C11	154.98 (13)
C1—C3—C4—C6	63.77 (15)	C1—C3—C10—C12	−126.50 (14)
C10—C3—C4—C6	−147.41 (11)	C2—C3—C10—C12	−67.79 (17)
C2—C3—C4—C6	4.10 (17)	C4—C3—C10—C12	83.94 (15)
C1—C3—C4—C5	132.15 (12)	C2—C1—C13—C18	12.3 (3)
C10—C3—C4—C5	−79.04 (14)	C3—C1—C13—C18	174.67 (16)
C2—C3—C4—C5	72.48 (15)	C2—C1—C13—C14	−165.7 (2)
C6—C4—C7—C8	10.88 (18)	C3—C1—C13—C14	−3.3 (3)
C3—C4—C7—C8	159.15 (12)	C6—C5—C19—C20	28.94 (18)
C5—C4—C7—C8	−58.79 (17)	C4—C5—C19—C20	100.48 (15)
C6—C4—C7—C9	−61.65 (16)	C6—C5—C19—C24	−151.62 (12)
C3—C4—C7—C9	86.62 (14)	C4—C5—C19—C24	−80.09 (16)
C5—C4—C7—C9	−131.32 (13)		

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Table 2
Closest intermolecular approaches (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···C21$^{\rm i}$	0.968 (16)	2.870 (16)	3.7742 (19)	155.9 (12)
C7—H7···C20$^{\rm ii}$	0.994 (14)	2.792 (14)	3.6439 (18)	144.0 (11)
C8—H82···C18$^{\rm iii}$	0.994 (17)	2.890 (17)	3.741 (2)	144.2 (12)
C18—H18···C8$^{\rm i}$	0.982 (16)	2.756 (15)	3.4866 (19)	131.7 (11)
C20—H20···C16$^{\rm iii}$	0.987 (16)	2.776 (16)	3.607 (2)	142.3 (12)

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

Refined C—H distances are in the range 0.967 (16)–1.03 (2) Å.

Data collection: *SMART-NT* (Bruker, 1998); cell refinement: *SAINT-NT* (Bruker, 1998); data reduction: *SAINT-NT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

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References

Allen, F. H., Lommerse, J. P. M., Hoy, V. J., Howard, J. A. K. & Desiraju, G. R. (1996). *Acta Cryst.* B**52**, 734–745.

Boese, R. (1992). *Advances in Strain in Organic Chemistry*, Vol. 2, pp. 191–254. New York: JAI Press.

Bruker (1998). *SMART-NT* (Version 5.0), *SAINT-NT* (Version 5.0) and *SHELXTL* (Verson 6.01). Bruker AXS Inc., Madison, Wisconsin, USA.

Hunter, C. A., Lawson, K. R., Perkins, J. & Urch, C. J. (2001). *J. Chem. Soc. Perkin Trans.* 2, pp. 651–669.

Nijveldt, D. & Vos, A. (1988). *Acta Cryst.* B**44**, 281–289.

Sheldrick, G. M. (1997). *SHELXL*97 and *SHELXS*97. University of Göttingen, Germany.

Shen, Q., Wells, C., Traetteberg, M., Bohn, R. K., Willis, A. & Knee, J. (2001). *J. Org. Chem.* **66**, 5840–5845.

Steiner, T. (2002). *Angew. Chem. Int. Ed. Engl.* **41**, 48–76.

Yeh, J.-Y. & Saebo, S. (1995). *J. Mol. Struct.* (*Theochem*), **334**, 155–163.

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