Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

[(2*R*,5*R*)-2,5-Bis(4-nitrophenyl)perhydrofuro-[2,3-*b*]furan-3-yl]triisopropylsilane

Simon J. Coles* and Michael B. Hursthouse

Department of Chemistry, University of Southampton, Southampton SO17 1BJ, England

Correspondence e-mail: s.j.coles@soton.ac.uk

Received 10 January 2002
Accepted 21 October 2002
Online 31 January 2002

The title molecule, $C_{27}H_{36}N_2O_6Si$, contains a pair of fused tetrahydrofuran rings, symmetrically substituted by *p*-nitrophenyl groups at centres of *R* chirality. The crystal structure is composed of dimers interacting through π-stacking to form columns.

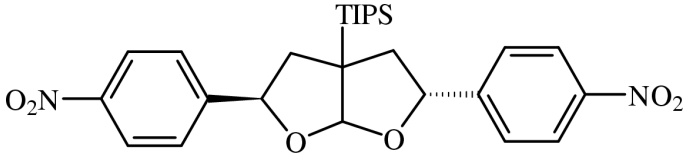
Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.002 Å
R factor = 0.045
wR factor = 0.128
Data-to-parameter ratio = 17.8

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

Comment

Compound (I) was prepared during an ongoing study into the Lewis-acid-mediated additions of silylated methylenecyclopropanes to aldehydes. The structure of (I) is composed of a pair of tetrahydrofuran rings fused at the 2- and 3-positions, both substituted at the 5-position by *p*-nitrophenyl groups. These substituted positions both exhibit *R* chirality. The axial 3-position of the fused ring system is substituted by a triisopropylsilyl group (TIPS). Puckering analysis (Cremer & Pople, 1975) shows that the furan ring containing O1 forms an envelope conformation about C3, whilst the ring containing O2 adopts a twisted conformation. The fused rings form a dihedral angle of 68.91 (6)° with respect to each other. The rings containing O1 and O2 have similar orientations with respect to their nitrophenyl substituents, the angles being 66.37 (5) and 67.64 (6)°, respectively.



(I)

Two molecules associate *via* intermolecular interactions through one of the nitro groups (Table 1) to form dimers. The crystal structure is formed by these dimers interacting by means of π-stacking between phenyl rings to form columns along the *a* axis [ring centroid separations are 4.1305 (6) Å for $Cg1\cdots Cg1^{i}$ and 3.6113 (7) Å for $Cg2\cdots Cg2^{ii}$, where *Cg*1 is the centroid of ring C7–C12 and *Cg*2 is the centroid of ring C13–C18; symmetry codes: (i) $-x, 1-y, 1-z$; (ii) $1-x, 1-y, z$].

Experimental

The title compound was prepared in 16% yield by the $BF_3{\cdot}Et_2O$-mediated addition of triisopropyl(2-methylidenecyclopropyl)silane to 4-nitrobenzaldehyde in dichloromethane.

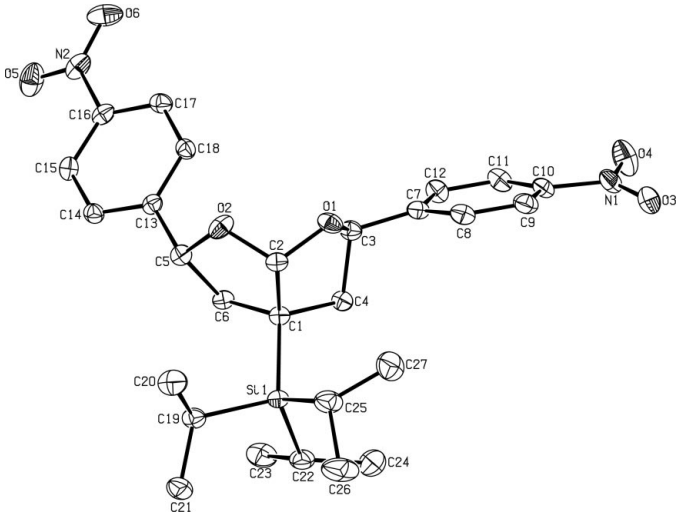


Figure 1
View of (I) with 50% probability displacement ellipsoids.

Crystal data

$C_{27}H_{36}N_2O_6Si$
$M_r = 512.67$
Triclinic, $P\overline{1}$
$a = 7.9703$ (16) Å
$b = 12.128$ (2) Å
$c = 14.121$ (3) Å
$\alpha = 79.14$ (3)°
$\beta = 83.06$ (3)°
$\gamma = 77.29$ (3)°
$V = 1303.2$ (4) Å^3
$Z = 2$
$D_x = 1.307$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 9132 reflections
$\theta = 2.9$–27.5°
$\mu = 0.14$ mm^{-1}
$T = 120$ (2) K
Block, colourless
0.38 × 0.14 × 0.06 mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)
$T_{min} = 0.951$, $T_{max} = 0.992$
19904 measured reflections
5893 independent reflections
4879 reflections with $I > 2\sigma(I)$
$R_{int} = 0.050$
$\theta_{max} = 27.5°$
$h = -10 \rightarrow 10$
$k = -15 \rightarrow 15$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.128$
$S = 1.02$
5893 reflections
332 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0702P)^2 + 0.5256P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.023$
$\Delta\rho_{max} = 0.67$ e Å^{-3}
$\Delta\rho_{min} = -0.37$ e Å^{-3}

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6A···O6^{i}	0.99	2.56	3.481 (2)	154
C12—H12···O5^{i}	0.95	2.50	3.381 (2)	155

Symmetry code: (i) $-x, 1-y, -z$.

H atoms were placed in idealized positions and refined with a riding model.

Data collection: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Hooft, 1998); cell refinement: *DENZO* and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 1990); software used to prepare material for publication: *SHELXL*97 and *PLATON*.

The authors thank the EPSRC for funding of crystallographic facilities.

References

Blessing, R. H. (1997). *J. Appl. Cryst.* **30**, 421–426.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Hooft, R. (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter and R. M. Sweet, pp. 307–326. London: Academic Press.
Sheldrick, G. M. (1997). *SHELXL*97 and *SHELXS*97. University of Göttingen, Germany.
Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.