Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

6,6,8a-Trimethyl-3a,6,7,8a-tetrahydrobenzo[*b*]furo[3,2-*d*]furan-2,4(3*H*,5*H*)-dione

Basavegowda Nagaraj,[a] Hemmige S. Yathirajan,[a] Padmarajaiah Nagaraja[b] and Daniel E. Lynch[c]*

[a]Department of Studies in Chemistry, University of Mysore, Manasagangotri, Mysore 570 006, India, [b]Department of Studies in Chemistry, University of Mysore, Manasagangotri, Mysore - 570 006, India, and [c]School of Science and the Environment, Coventry University, Coventry CV1 5FB, England

Correspondence e-mail: apx106@coventry.ac.uk

Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.002 Å
R factor = 0.027
wR factor = 0.068
Data-to-parameter ratio = 14.8

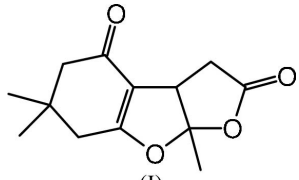
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 9 March 2005
Accepted 14 March 2005
Online 18 March 2005

The structure of the title compound, $C_{13}H_{16}O_4$, comprises a non-planar chiral molecule where the cyclohexene double bond is distinctly shorter [1.335 (2) Å] than the neighbouring C—C single bonds (>1.4 Å).

Comment

The title compound, (I), a perhydrofurobenzofuran, exhibits hypoglycemic properties. A search of the Cambridge Structural Database (Version 5.26; Allen, 2002) for related structures reveals that there are 38 compounds containing a six-membered carbocyclic ring with two linked five-membered furo rings, as in (I). However, in all 38 molecules the C_6 ring is benzene; none are cyclohexane, -ene or -yne variants. The structure of (I) comprises a non-planar chiral molecule where the C5═C10 double bond is distinctly shorter [1.335 (2) Å] than the neighbouring C—C single bonds (>1.4 Å). The two torsion angles that highlight the non-planarity of the molecule are O1—C1—C4—C3 [−127.2 (1)°] and O2—C1—C4—C5 [104.0 (1)°].



(I)

Experimental

The title compound was prepared according to the literature procedure of Nagarajan *et al.* (1988). Crystals were grown from ethanol.

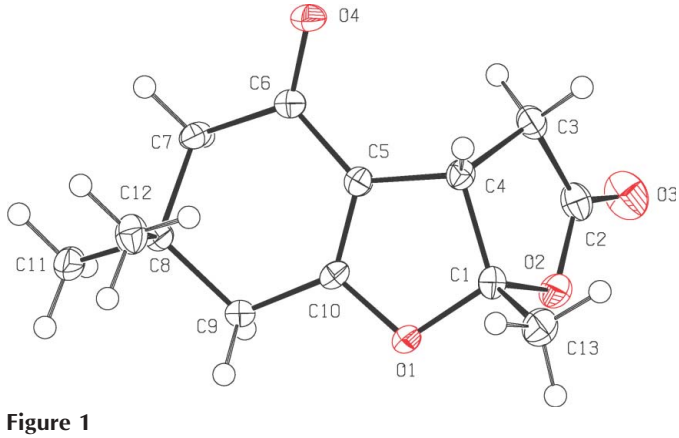


Figure 1
The molecular configuration and atom-numbering scheme for (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are drawn as spheres of arbitrary radius.

Crystal data

$C_{13}H_{16}O_4$	Mo $K\alpha$ radiation
M_r = 236.26	Cell parameters from 1560 reflections
Orthorhombic, $P2_12_12_1$	θ = 2.9–27.5°
a = 9.4853 (3) Å	μ = 0.10 mm^{-1}
b = 10.2904 (2) Å	T = 120 (2) K
c = 12.2872 (4) Å	Prism, colourless
V = 1199.32 (6) Å^3	0.50 × 0.40 × 0.40 mm
Z = 4	
D_x = 1.309 Mg m^{-3}	

Data collection

Nonius KappaCCD diffractometer	2235 reflections with $I > 2\sigma(I)$
φ and ω scans	R_{int} = 0.021
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)	θ_{max} = 26.0°
T_{min} = 0.953, T_{max} = 0.962	$h = -11 \rightarrow 10$
8608 measured reflections	$k = -12 \rightarrow 12$
2343 independent reflections	$l = -14 \rightarrow 15$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0353P)^2 + 0.2043P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.027	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.068	$\Delta\rho_{max}$ = 0.20 e Å^{-3}
S = 1.04	$\Delta\rho_{min}$ = −0.14 e Å^{-3}
2343 reflections	Extinction correction: *SHELXL97*
158 parameters	Extinction coefficient: 0.045 (6)
H-atom parameters constrained	

All H atoms were included in the refinement at calculated positions, in the riding-model approximation, with C—H distances of 0.98 (CH_3), 0.99 (CH_2) and 1.00 Å (CH). The isotropic displacement parameters for all H atoms were set equal to 1.25U_{eq} of the carrier atom. In the absence of significant anomalous scattering effects, the 763 Friedel pairs were merged.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

The authors thank the EPSRC National Crystallography Service (Southampton, England) and acknowledge the use of the EPSRC's Chemical Database Service at Daresbury, England (Fletcher *et al.*, 1996).

References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Fletcher, D. A., McMeeking, R. F. & Parkin, D. J. (1996). *J. Chem. Inf. Comput. Sci.* **36**, 746–749.
Hooft, R. W. W. (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
Nagarajan, K., Talwalker, P. K., Goud, A. N., Shah, R. K., Shenoy, S. J. & Desai, N. D. (1988). *Indian J. Chem. Sect. B*, **27**, 1113–1123.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Sheldrick, G. M. (2003). *SADABS*. Version 2.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.