Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(1*S**,4*S**,5*R**,8*R**)-8-(3,4-Dimethoxyphenyl)-4-(3,4,5-trimethoxyphenyl)-3,7-dioxabicyclo[3.3.0]octan-2-one

Mark E. Light* and Michael B. Hursthouse

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

Correspondence e-mail: light@soton.ac.uk

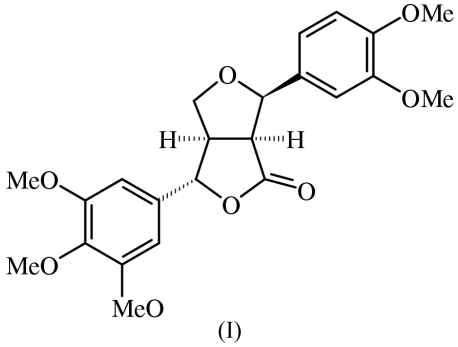
Received 19 April 2001
Accepted 4 May 2001
Online 17 May 2001

The X-ray study of the title compound, $C_{23}H_{26}O_8$, (I), confirmed the *endo,exo*-furofuranone structure of the important intermediate compound in the process of the total synthesis of (±)-epimagnolin A, which employs a new approach involving a highly diastereoselective C—H insertion reaction [Brown *et al.* (2001). *Tetrahedron Lett.* **42**, 473–475].

Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.014 Å
R factor = 0.086
wR factor = 0.334
Data-to-parameter ratio = 10.7

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



(I)

Experimental

The synthesis of the title compound was reported by Brown *et al.* (2001). The crystals were grown from chlorofrom; all of them, however, diffracted very poorly and were barely usable for the X-ray diffraction experiment.

Crystal data

$C_{23}H_{26}O_8$
M_r = 430.44
Monoclinic, $P2_1/n$
a = 11.839 (2) Å
b = 8.0773 (10) Å
c = 22.695 (4) Å
β = 91.84 (3)°
V = 2169.1 (6) Å^3
Z = 4
D_x = 1.318 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2999 reflections
θ = 3.0–23.2°
μ = 0.10 mm^{-1}
T = 150 (2) K
Block, colourless
0.10 × 0.10 × 0.10 mm

Data collection

Nonius Kappa CCD area-detector diffractometer
φ and ω scans to fill Ewald sphere
13 956 measured reflections
2999 independent reflections
947 reflections with $I > 2\sigma(I)$
R_{int} = 0.361
θ_{max} = 23.2°
$h = -13 \rightarrow 13$
$k = -8 \rightarrow 8$
$l = -25 \rightarrow 25$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.086
$wR(F^2)$ = 0.334
S = 0.81
2999 reflections
281 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1905P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.010
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

© 2001 International Union of Crystallography
Printed in Great Britain – all rights reserved

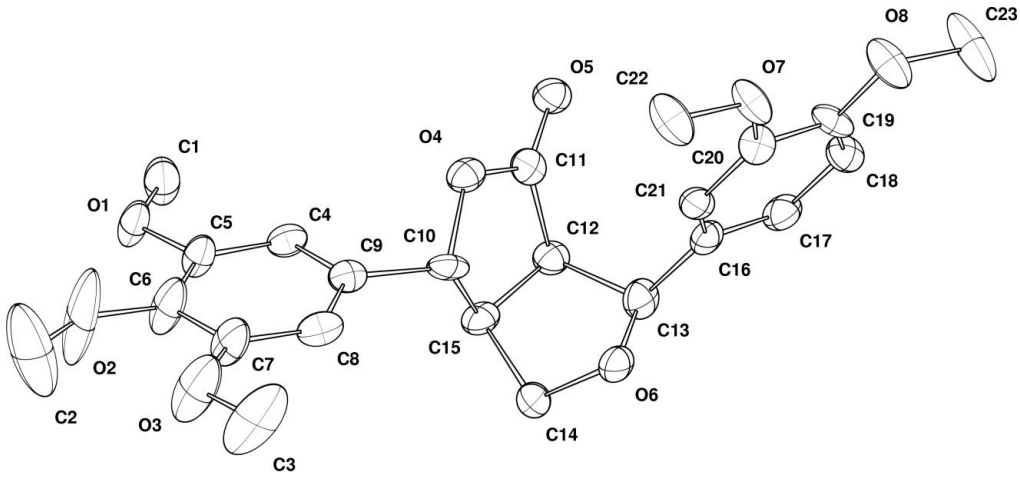


Figure 1
View of (I) showing 50% probability displacement ellipsoids. The H atoms have been omitted for clarity.

The poor diffracting ability of the crystals is most probably due to the thermal disorder of the methoxy groups in both phenyl rings. Attempts to model their disorder, however, did not improve the refinement.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *SCALEPACK* (Otwinoski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* (Otwinoski & Minor, 1997), *COLLECT* and *maXus* (Mackay *et al.*, 1998); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *CAMERON* (Watkin *et al.*, 1993); software used to prepare material for publication: *WinGX* (Farrugia, 1998).

References

Brown, R. C. D., Bataille, C. J. R. & Hinks, J. D. (2001). *Tetrahedron Lett.* **42**, 473–475.
Farrugia, L. J. (1998). *WinGX*. University of Glasgow, Scotland.
Hooft, R. (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
Mackay, S., Gilmore, C. J., Edwards, C., Tremayne, M., Stuart, N. & Shankland, K. (1998). *maXus*. University of Glasgow, Scotland, Nonius BV, Delft, The Netherlands, and MacScience Co. Ltd, Yokohama, Japan.
Otwinoski, Z. & Minor, W. (1997). *Methods Enzymol.* **276**, 307–767.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Watkin, D. M., Pearce, L. & Prout, C. K. (1993). *CAMERON*. Chemical Crystallography Laboratory, University of Oxford, England.