Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

8-Methoxy-4-(4-methoxyphenyl)isochroman-3-ol

Marcin Palusiak,[a]* Sławomir J. Grabowski,[a] Jan Epsztajn[b] and Justyna A. Kowalska[b]

[a]Department of Crystallography and Crystal Chemistry, University of Łódź, Pomorska 149/153, PL-90236 Łódź, Poland, and [b]Department of Organic Chemistry, University of Łódź, Narutowicza 68, PL-90136 Łódź, Poland

Correspondence e-mail: marcinp@uni.lodz.pl

Received 28 October 2003
Accepted 12 November 2003
Online 29 November 2003

Key indicators

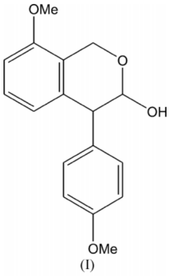
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.006 Å
R factor = 0.069
wR factor = 0.185
Data-to-parameter ratio = 7.2

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

In the title compound, $C_{17}H_{18}O_4$, the heterocyclic ring has a half-chair conformation. The substituents at the 3- and 4-positions are attached equatorially with respect to the heterocyclic ring. The molecules are linked by O—H···O hydrogen bonds, the hydroxyl O atom functioning as both donor and acceptor. This results in the formation of $C(2)$ chains running along the twofold screw axis.

Comment

As a result of their wide range of biological properties, the chemistry of benzo[*c*]pyran derivatives has been investigated (Moore, 1977; Moore & Czerniak, 1981). The title compound, (I), was synthesized as part of a research project on new precursors for the production of this type of antibiotic (Epsztajn *et al.*, 2001). Isochromans have already been investigated by X-ray analysis (Palusiak *et al.*, 2002*a*,*b*, 2003) and the present work is a continuation of these structural studies.



(I)

The structure of (I) is shown in Fig. 1. The heterocyclic ring adopts a half-chair conformation, with the twofold axis passing through the mid-point of the O2—C3 bond. The asymmetry parameter (Nardelli, 1983) Δ_2(O2—C3) is 0.024 (2), and the puckering parameters (Cremer & Pople, 1975), corresponding to the atom sequence C3/O2/C1/C9/C10/C4, are Q = 0.505 (4) Å, φ_2 = −156.7 (6)° and θ_2 = 128.3 (5)°. The substituents at the 3- and 4-positions are in a *trans* configuration, which seems to be characteristic for the previously investigated isochromans. However, in contrast with previous cases, in which the heterocyclic ring adopted the same conformation, these substituents are equatorial in (I).

The aromatic rings are oriented almost perpendicular to each other, the dihedral angle being 77.6 (2)°. As expected, the non-H atoms of the methoxy groups do not deviate significantly from the planes of the attached rings, the maximum deviation being 0.179 (6) Å for atom C440. This arrangement is most advantageous energetically, as has been proved both experimentally and theoretically (Federsel *et al.*, 2001).

DOI: 10.1107/S1600536803026102

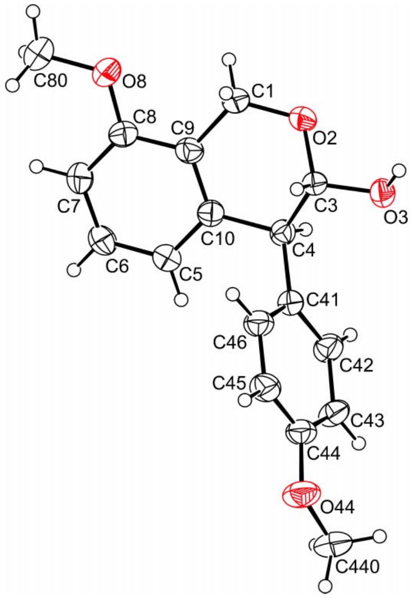


Figure 1
A view of (I) with the atom-labelling scheme. Displacement ellipsoids are drawn at the 40% probability level.

Atom O31 of the hydroxyl group is involved in intermolecular O—H···O hydrogen bonding (Table 2) and functions as both donor and acceptor. Molecules linked in this way form chains running along the *b* direction; these correspond to *C*(2) graph-set motifs (Bernstein *et al.*, 1995).

Experimental

The synthesis of (I) was carried out in the manner proposed by Epsztajn *et al.* (2001) and described in Kowalska (2002). Crystals were obtained by slow evaporation of a methanol solution at room temperature.

Crystal data

$C_{17}H_{18}O_4$
$M_r = 286.31$
Monoclinic, $P2_1$
$a = 10.5330$ (18) Å
$b = 5.145$ (3) Å
$c = 13.607$ (3) Å
$\beta = 106.018$ (15)°
$V = 708.8$ (5) Å^3
$Z = 2$
$D_x = 1.342$ Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 45 reflections
$\theta = 45.6$–57.4°
$\mu = 0.78$ mm^{-1}
$T = 293$ (2) K
Prism, colourless
0.5 × 0.3 × 0.3 mm

Data collection

Rigaku AFC-5*S* diffractometer
ω scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.750$, $T_{\max} = 0.793$
4913 measured reflections
1388 independent reflections
1262 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.061$
$\theta_{\max} = 67.5°$
$h = -12 \rightarrow 12$
$k = -6 \rightarrow 6$
$l = -16 \rightarrow 16$
3 standard reflections every 150 reflections intensity decay: <2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.069$
$wR(F^2) = 0.185$
$S = 1.09$
1388 reflections
193 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.153P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.53$ e Å^{-3}
$\Delta\rho_{\min} = -0.24$ e Å^{-3}

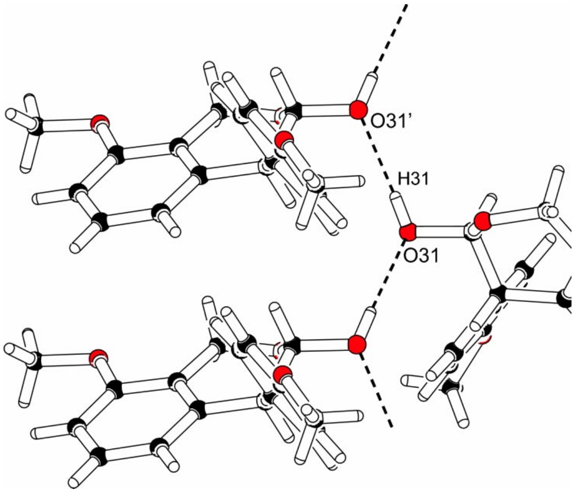


Figure 2
The intermolecular hydrogen bonding, indicated by dashed lines, in the crystal structure of (I).

Table 1
Selected geometric parameters (Å, °).

O2—C3	1.413 (4)	O8—C80	1.435 (5)
O2—C1	1.417 (4)	O44—C44	1.374 (4)
O31—C3	1.396 (4)	O44—C440	1.418 (6)
O8—C8	1.359 (5)		
C3—O2—C1	112.3 (2)	C44—O44—C440	118.1 (4)
C3—O31—H31	109.5	C45—C44—O44	115.1 (4)
C8—O8—C80	117.9 (3)	O44—C44—C43	124.6 (4)
O31—C3—O2	107.4 (3)	O8—C8—C7	124.4 (4)
O31—C3—C4	109.3 (3)	O8—C8—C9	115.0 (3)
O2—C3—C4	109.9 (3)		
C1—O2—C3—O31	−171.7 (3)	C80—O8—C8—C7	6.4 (6)
C1—O2—C3—C4	69.5 (4)	C80—O8—C8—C9	−175.7 (4)
O31—C3—C4—C41	65.9 (4)	C10—C9—C8—O8	177.9 (4)
O31—C3—C4—C10	−168.4 (3)	C1—C9—C8—O8	−2.3 (6)
C4—C10—C9—C1	1.6 (6)	O44—C44—C43—C42	178.9 (4)
C440—O44—C44—C45	174.6 (4)	O8—C8—C7—C6	−180.0 (5)
C440—O44—C44—C43	−4.8 (6)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O31—H31···O31$^{\rm i}$	0.82	2.08	2.899 (4)	178

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

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.93–0.98 Å and the O—H distance fixed at 0.82 Å. For methoxy H atoms, $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$; for all other H atoms, $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$. In the absence of significant anomalous dispersion effects, Friedel pairs were averaged and the absolute configuration cannot be determined from the crystallographic experiment.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1989); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1989); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PARST*97 (Nardelli, 1996).

The authors thank the Rector of the University of Łódź for financial support (University Research Grants).

References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.

Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.

Epsztajn, J., Bieniek, A., Kowalska, J. A. & Malinowski, Z. (2001). *Tetrahedron Lett.* **42**, 9293–9295.

Federsel, D., Herrmann, A., Christen, D., Sander, S., Willner, H. & Oberhammer, H. (2001). *J. Mol. Struct.* **567–568**, 127–136, and references therein.

Kowalska, J. A. (2002). PhD thesis, University of Łódź, Poland.

Molecular Structure Corporation (1989). *MSC/AFC Diffractometer Control Software* and *TEXSAN* (Version 5.0). MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Moore, H. W. (1977). *Science*, **197**, 527–532.

Moore, H. W. & Czerniak, R. (1981). *Med. Res. Rev.* **1**, 249–280.

Nardelli, M. (1983). *Acta Cryst.* C**39**, 1141–1142.

Nardelli, M. (1996). *J. Appl. Cryst.* **29**, 296–300.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Palusiak, M., Małecka, M., Grabowski, S. J., Epsztajn, J., Bieniek, A. & Kowalska J. A. (2002*a*). *Acta Cryst.* E**58**, o620–o622.

Palusiak, M., Małecka, M., Grabowski, S. J., Epsztajn, J., Bieniek, A. & Kowalska J. A. (2002*b*). *Acta Cryst.* E**58**, o1188–o1189.

Palusiak, M., Małecka, M., Rybarczyk-Pirek, A., Epsztajn, J., Bieniek, A. & Kowalska J. A. (2003). *Acta Cryst.* E**59**, o11–o13.

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

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.