C22—H22*B*···O13^{ii}	0.96	2.42	3.328 (6)	157
C18—H18*B*···O20^{iii}	0.96	2.41	3.168 (4)	135
C7—H7*A*···O13^{iv}	0.93	2.54	3.405 (4)	156

Symmetry codes: (i) $-x, y-\frac{1}{2}, \frac{1}{2}-z$; (ii) $x, y-1, z$; (iii) $x-1, y-1, z$; (iv) $x-\frac{1}{2}, \frac{3}{2}-y, -z$.

The data collection covered over a hemisphere of reciprocal space by a combination of three sets of exposures; each set had a different φ angle (0, 88 and 180°) for the crystal and each exposure of 30 s covered 0.3° in ω. The crystal-to-detector distance was 4 cm and the detector swing angle was −35°. Crystal decay was monitored by repeating 30 initial frames at the end of data collection and analysing the intensity of duplicate reflections, and was found to be negligible.

The structure was solved by direct methods and refined by full-matrix least-squares techniques. After checking the presence of all H atoms in the difference map, the H atoms belonging to the C and hydroxyl O atoms were geometrically fixed and allowed to ride on the parent atoms. Rotating group refinement was used for the methyl and OH groups. In order to allow slightly longer N—H distances upon protonation, the H atoms of N17 were initially isotropically refined, but the N17—H17*A* [1.08 (3) Å] distance was found to be longer than that of N17—H17*B* [0.88 (4) Å]. Hence the N—H distances were restrained by refining them as a free variable (using *DFIX*21 for N17, H17*A*, N17, H17*B*) and the final N—H distance was found to be 1.02 (3) Å. This value is in agreement with those observed (0.97 and 1.01 Å) in pipemidic acid trihydrate (Fonseca *et al.*, 1986).

Data collection: *SMART* (Siemens, 1996). Cell refinement: *SAINT* (Siemens, 1996). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 1990).

The authors would like to thank the Malaysian Government and Universiti Sains Malaysia for research grant R&D No. 190-9609-2801. KC thanks the Universiti Sains Malaysia for a Visiting Post-Doctoral Fellowship.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1259). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1999). C**55**, 768–770

2,2-Bis(2-methoxyphenyl)-4-methyl-8,9-epoxy-7,8,9,10-tetrahydro-2*H*-benzo[*h*]-chromene

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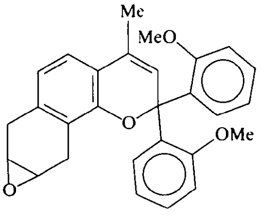
(*Received 24 November 1998; accepted 21 December 1998*)

Abstract

In each independent molecule of the title compound, $C_{28}H_{26}O_4$, the pyran ring adopts a half-chair conformation and the tetrahydrobenzene ring is in a flattened-boat conformation. The phenyl rings are orthogonal to each other; in one of the enantiomers, they form dihedral angles of 74.66 (6) and 54.38 (6)° with the pyran ring, while in the other enantiomer, these angles are 76.42 (6) and 54.15 (5)°.

Comment

Epoxides are versatile reactive intermediates in organic synthesis because they are very susceptible to attack by several nucleophiles (Smith, 1984). Furthermore, the role of arene oxides in biological systems continues to attract attention due to their cytotoxicity, mutagenicity and carcinogenicity (Boyd & Sharma, 1986). The structure determination of the title compound, (I), was carried out in order to elucidate the molecular conformation.



(I)

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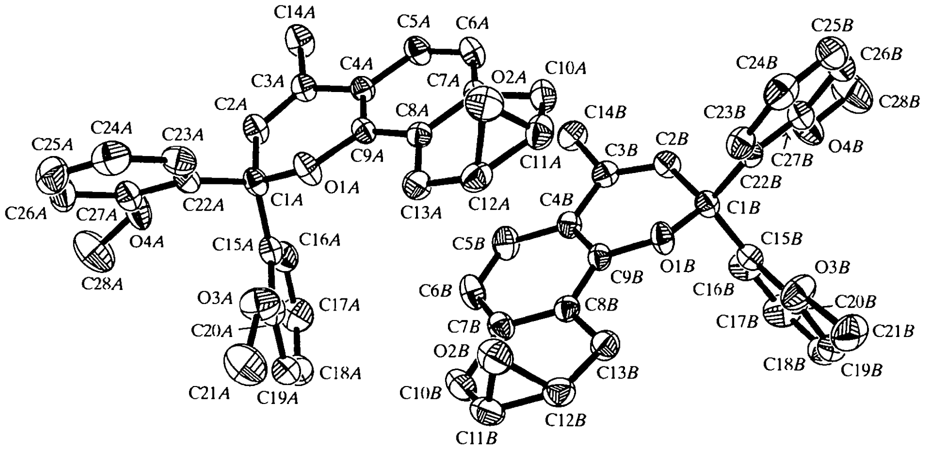

Fig. 1. A view of the structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

The asymmetric unit consists of an enantiomeric pair of molecules with their centroid at (0.382, 0.195, 0.262). The corresponding bond distances and angles in this enantiomeric pair agree with each other; the C2—C3 bond shows double-bond character. In the tetrahydrobenzene ring, the Csp^2—Csp^3 distances are longer than the Csp^3—Csp^3 distances because of the steric interactions caused by the near planarity of the tetrahydrobenzene ring. The Csp^3—Csp^3 and Csp^3—O lengths in the oxirane ring system agree with the reported mean values of 1.466 and 1.446 Å, respectively (Allen *et al.*, 1987). The C—C lengths in the phenyl rings range from 1.363 (3) to 1.400 (2) Å. In one of the enantiomers, the methoxy groups are twisted away from the phenyl-ring planes [C21*A*—O3*A*—C20*A*—C19*A* −14.4 (3) and C28*A*—O4*A*—C27*A*—C26*A* −30.6 (3)°], while in the other, they are nearly coplanar.

In both molecules, the pyran ring adopts a half-chair conformation with asymmetry parameter ΔC_2(C1*A*—O1*A*) = 0.003 (1) and ΔC_2(C1*B*—O1*B*) = 0.005 (1) (Nardelli, 1983); the tetrahydrobenzene ring is in a flattened-boat conformation, with atoms C10*A*, C13*A*, C10*B* and C13*B* deviating from their best planes by 0.118 (2), 0.098 (2), 0.135 (2) and 0.104 (2) Å, respectively. The oxirane plane in the two molecules forms dihedral angles of 83.53 (9) and 84.12 (9)° with the attached rings. The two phenyl-ring planes are orthogonal to each other and make dihedral angles of 74.66 (6) and 54.38 (6)° [76.42 (6) and 54.15 (5)° in the other molecule] with the best plane through the pyran ring. In one of the enantiomers, the fused benzene ring forms dihedral angles of 2.79 (6) and 5.97 (5)° with the adjacent pyran and tetrahydrobenzene rings, and in the other enantiomer, these angles are 4.23 (6) and 6.59 (6)°.

In the asymmetric unit, the enantiomeric pair is linked by a weak C12*A*—H12*A*···O2*B* hydrogen bond (Table 2).

Experimental

To *o*-anisyl lithium (3 equivalents, 6.57 mmol), prepared by the direct metallation of anisole (0.715 ml, 6.57 mmol) using *n*-butyllithium and tetramethylethylenediamine, was added 4-methyl-7,8,9,10-tetrahydro-8,9-epoxybenzo[*h*]coumarin (0.5 g, 2.19 mmol) at 253 K under a nitrogen atmosphere using dry THF as solvent. After completion of the reaction, it was quenched by addition of saturated sodium bicarbonate (3 ml). The organic layer was separated and chromatographed over silica gel to obtain the title compound in 60–70% yield. Single crystals were grown by slow evaporation from a chloroform–methanol (1:1) solvent system.

Crystal data

$C_{28}H_{26}O_4$
M_r = 426.49
Triclinic
$P\bar{1}$
a = 8.4481 (1) Å
b = 16.4652 (1) Å
c = 16.8140 (2) Å
α = 104.330 (1)°
β = 97.886 (1)°
γ = 92.943 (1)°
V = 2235.72 (4) Å^3
Z = 4
D_x = 1.267 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 8192 reflections
θ = 1.27–28.30°
μ = 0.084 mm^{-1}
T = 293 (2) K
Parallelepiped
0.34 × 0.30 × 0.28 mm
Colourless

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: none
17 735 measured reflections
10 384 independent reflections

5133 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.063
$\theta_{\rm max}$ = 28.3°
$h = -11 \rightarrow 11$
$k = -21 \rightarrow 21$
$l = 0 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.138$
$S = 0.884$
10 384 reflections
584 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0513P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.29$ e Å^{-3}
$\Delta\rho_{\min} = -0.26$ e Å^{-3}
Extinction correction: *SHELXTL* (Sheldrick, 1997)
Extinction coefficient: 0.0063 (10)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

O1*A*—C9*A*	1.377 (2)	O1*B*—C9*B*	1.373 (2)
O1*A*—C1*A*	1.450 (2)	O1*B*—C1*B*	1.453 (2)
O2*A*—C12*A*	1.442 (2)	O2*B*—C11*B*	1.440 (2)
O2*A*—C11*A*	1.443 (2)	O2*B*—C12*B*	1.446 (2)
O3*A*—C20*A*	1.368 (2)	O3*B*—C20*B*	1.366 (2)
O3*A*—C21*A*	1.386 (2)	O3*B*—C21*B*	1.408 (2)
O4*A*—C27*A*	1.376 (2)	O4*B*—C27*B*	1.375 (2)
O4*A*—C28*A*	1.397 (3)	O4*B*—C28*B*	1.393 (2)
C2*A*—C3*A*	1.325 (2)	C2*B*—C3*B*	1.325 (2)
C7*A*—C10*A*	1.508 (2)	C7*B*—C10*B*	1.510 (2)
C8*A*—C13*A*	1.501 (2)	C8*B*—C13*B*	1.501 (2)
C10*A*—C11*A*	1.486 (3)	C10*B*—C11*B*	1.490 (3)
C11*A*—C12*A*	1.456 (3)	C11*B*—C12*B*	1.445 (3)
C12*A*—C13*A*	1.483 (3)	C12*B*—C13*B*	1.491 (2)
C12*A*—O2*A*—C11*A*	60.6 (1)	C11*B*—O2*B*—C12*B*	60.1 (1)
C21*A*—O3*A*—C20*A*—C19*A*	−14.4 (3)		
C28*A*—O4*A*—C27*A*—C26*A*	−30.6 (3)		
C21*B*—O3*B*—C20*B*—C19*B*	6.3 (3)		
C28*B*—O4*B*—C27*B*—C26*B*	−1.4 (3)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12*A*—H12*A*···O2*B*	0.98	2.52	3.261 (2)	132

The data collection covered over a hemisphere of reciprocal space by a combination of three sets of exposures; each set had a different φ angle (0, 88 and 180°) for the crystal and each exposure of 30 s covered 0.3° in ω. The crystal-to-detector distance was 3 cm and the detector swing angle was −20°. Crystal decay was monitored by repeating 30 initial frames at the end of data collection and analysing the intensity of duplicate reflections, and was found to be negligible. The structure was solved by direct methods and refined by full-matrix least-squares techniques. After checking their presence in the difference map, all the H atoms were placed at geometrically calculated positions and a riding model was used for their refinement; rotating group refinement was used for the methyl groups.

Data collection: *SMART* (Siemens, 1996). Cell refinement: *SAINT* (Siemens, 1996). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL* and *PARST* (Nardelli, 1995).

The authors would like to thank the Malaysian Government and Universiti Sains Malaysia for research grant R&D No. 190-9609-2801. KC thanks the Universiti Sains Malaysia for a Visiting Post-Doctoral Fellowship.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1398). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1999). C**55**, 770–772

4-(*p*-Methoxyphenyl)-3,5-bis(2-pyridyl)-4*H*-1,2,4-triazole

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(*Received 4 January 1999; accepted 28 January 1999*)

Abstract

In the title molecule, $C_{19}H_{15}N_5O$, the dihedral angle between the phenyl and the triazole rings is 59.30 (7)°. The triazole ring forms dihedral angles of 47.90 (8) and 26.91 (8)° with the two pyridyl rings. In the crystal, the molecules exist as centrosymmetrically C—H···N intermolecular hydrogen-bonded dimers. The glide-related molecules translated a unit along the *b* axis are linked by weak C—H···N hydrogen bonds to form an infinite chain along the *c* axis which is further stabilized by C—H···π interactions.

Comment

Extensive studies have been carried out on the substituted 1,2,4-triazole ligands (Cornelissen *et al.*, 1992; Gupta & Bhargava, 1978; Kunkeler *et al.*, 1996). It

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