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2,3-Epoxy-13c-methoxy-1,13c-dihydrodibenzo[*a*,*kl*]xanthan-1-one

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.044
wR factor = 0.137
Data-to-parameter ratio = 16.4

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

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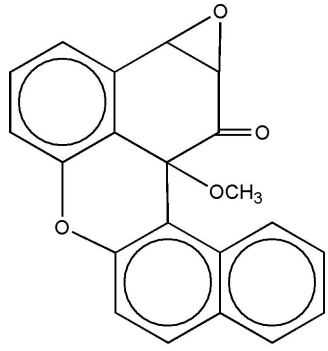
The title compound, $C_{21}H_{14}O_4$, containing three chiral C atoms, is a key intermediate in the design of chiral alcohols. Racemic dimers are generated by C—H···O hydrogen bonds formed between phenyl C—H groups and epoxy O atoms.

Comment

Epoxides are well known as one of the most valuable building blocks which can be used as intermediates and precursors for chemical production (Wynberg & Marsman, 1980; Wang *et al.*, 2003). The title compound, (I), is a key intermediate in the preparation of chiral alcohols, which we are designing for potential use as chiral additives in asymmetric synthesis.



(I)

The structure of (I) provides information on its conformation and the potential stereoselectivity of its ring-opening

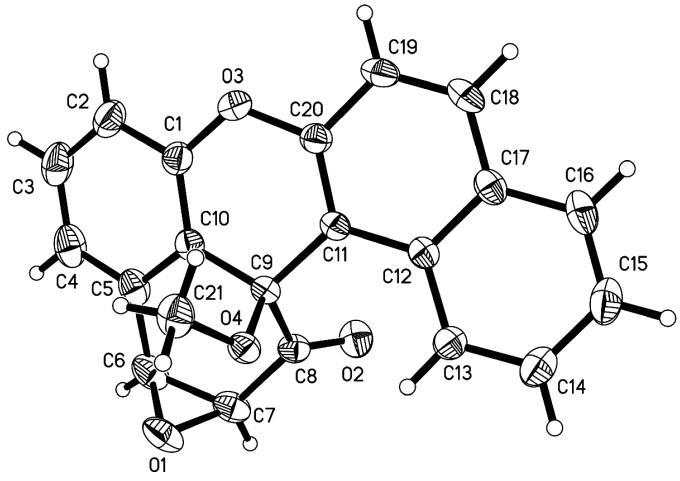


Figure 1
A view of the title molecule, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are represented by circles of arbitrary radii.

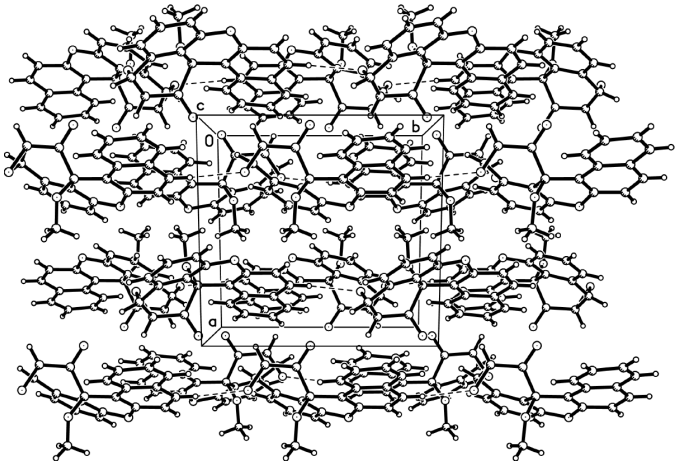


Figure 2
The molecular packing of (I), along the *b* axis. Dashed lines indicate hydrogen bonds.

reactions (Sasidharan *et al.*, 2002; Wang *et al.*, 2003). The structure of (I) was also confirmed by ^{1}H NMR, IR and FAB-MS spectroscopic analysis.

The molecule of (I) contains six fused rings (Fig. 1), with the methoxy group attached to a chiral C atom. The three aromatic rings are almost coplanar and the other six-membered carbocyclic ring adopts a twisted boat conformation. The epoxy group points in the same direction as the methoxy group, adopting a *syn* configuration. The pyran ring is nearly planar, with the C1–C10 and C11–C20 edges slightly folded up along O3–C9 axis.

Weak intermolecular C—H···O hydrogen bonds (H···O 2.47 Å, C···O 3.389 (2) Å and C—H···O 169°) formed between an aromatic C—H and the epoxy O atoms, create racemic dimers which pack along the *b* axis, as shown in Fig. 2.

Experimental

Compound (I) was obtained by epoxidation of 1-oxo-13c-methoxy-1,13c-dihydrodibenzo[*a*,*kl*]xanthene in methanol with aqueous hydrogen peroxide solution (30%) under mild reaction conditions (Tan *et al.*, 2001). Compound (I) was the main product, in a yield of 93%. A single crystal suitable for X-ray analysis was obtained from methanol (m.p. 433–435 K). ν (KBr disk): 3425, 2932, 2359, 1726 (*s*, C=O), 1452, 1247, 1066, 994, 798, 754, 503. ^{1}H NMR (500 MHz in $CDCl_3$/TMS, δ, p.p.m.): 2.80 (*s*, 3H), 4.04 (*d*, J = 4.0 Hz, 1H), 4.34 (*d*, J = 4.0 Hz, 1H), 7.22–7.52 (*m*, 6H), 7.79–7.81 (*m*, 1H), 7.86 (*d*, J = 9.0 Hz, 1H), 7.99–8.00 (*m*, 1H). FAB-MS *m*/*z* (%): 331 (M^++H, 8), 299 ($M^+ - OCH_3$, 25).

Crystal data

$C_{21}H_{14}O_4$	D_x = 1.419 Mg m^{-3}
M_r = 330.32	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 4328 reflections
a = 10.5756 (6) Å	θ = 2.9–25.1°
b = 10.5907 (6) Å	μ = 0.10 mm^{-1}
c = 14.4795 (8) Å	T = 293 (2) K
β = 107.548 (1)°	Block, colourless
V = 1546.28 (15) Å^3	0.47 × 0.45 × 0.23 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	3717 independent reflections
φ and ω scans	2918 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	R_{int} = 0.022
T_{min} = 0.963, T_{max} = 0.987	θ_{max} = 28.0°
10 025 measured reflections	$h = -13 \rightarrow 13$
	$k = -13 \rightarrow 10$
	$l = -19 \rightarrow 19$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0789P)^2 + 0.1717P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.044	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.137	$\Delta\rho_{max}$ = 0.31 e Å^{-3}
S = 1.05	$\Delta\rho_{min}$ = −0.22 e Å^{-3}
3717 reflections	
226 parameters	
Only H-atom displacement parameters refined	

All H atoms were included as riding atoms (C—H = 0.96 Å) and their isotropic displacement parameters were refined.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT* program(s) used to solve structure: *SHELXTL* (Bruker, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

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References

Bruker (1997). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1998). *SMART* (Version 5.625) and *SAINT* (Version 6.02a). Bruker AXS Inc., Madison, Wisconsin, USA.
Sasidharan, M., Wu, P. & Tatsumi, T. (2002). *J. Catal.* **205**, 332–338.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Tan, D. M., Li, H. H. & Wang, B. (2001). *Chin. J. Chem.* **19**, 91–93.
Wang, B., Kang, Y. R., Yang, L. M. & Suo, J. S. (2003). *J. Mol. Catal. A.* **203**, 29–36.
Wynberg, H. & Marsman, J. (1980). *J. Org. Chem.* **45**, 158–161