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1-Oxo-13c-propoxy-1,13c-dihydrodibenzo[*a*,*kl*]-xanthene

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.044
wR factor = 0.097
Data-to-parameter ratio = 19.4

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

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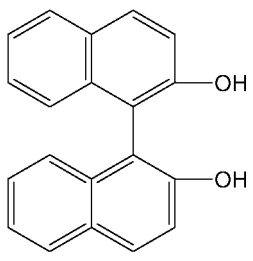
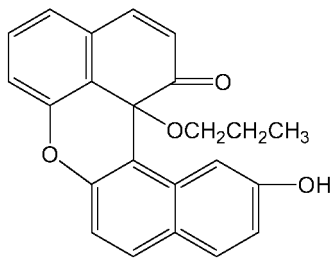
The title compound, $C_{23}H_{18}O_3$, is composed of five rings with one chiral C atom. It crystallizes in the chiral space group $P2_1$. with two molecules per asymmetric unit. These molecules are enantiomeric and exhibit normal geometry. The *n*-propoxy group in one of the pairs is disordered.

Comment

It is well known that most biological reactions are catalysed by enzymes under mild conditions, with high efficiency and selectivity. It is interesting to develop small molecules or complexes as catalysts to mimic some aspect of enzyme function in organic reactions, especially in the synthesis of structurally complicated compounds. Copper–amine complexes have been frequently used to mimic oxidases (Liang *et al.*, 1999; Mahadevan *et al.*, 2000) since many oxidases contain copper in their active sites (McGuirl & Dooley, 1999). Catalytic oxidation using dioxygen as sole oxidant was believed to be environmentally friendly since the resulting product was water (Choudary *et al.*, 2000). Successful application of copper–amine complexes in aerobic oxidative synthesis has been reported (Marko *et al.*, 1996; Nakajima *et al.*, 1999; Tan *et al.*, 2001). In our work, similar to that of Nakajima *et al.* (1999), the complex of $CuCl_2$–ethanolamine (Tan *et al.*, 2001) in *n*-propanol led to a domino reaction from 1,1′-bi-2-naphthol, (I), to yield the title compound, (II), a racemic multicyclic unsaturated ketone containing an *n*-propoxy group derived from the solvent *n*-propanol. The reaction could even take place at room temperature in high yield. Three conversions occurred from (I) to (II), *i.e.* 2′-O–C_8 coupling, 2-OH oxidation and C_1–$OCH_2CH_2CH_3$ coupling. The racemic structure of (II) was confirmed by ^{1}H NMR, ^{13}C NMR, IR, FAB–MS and elemental analysis, further confirmed by this single-crystal X-ray analysis.



(I)



(II)

As shown in Fig. 1, the molecule is composed of five fused rings with the propoxy group attached to a chiral C atom. The

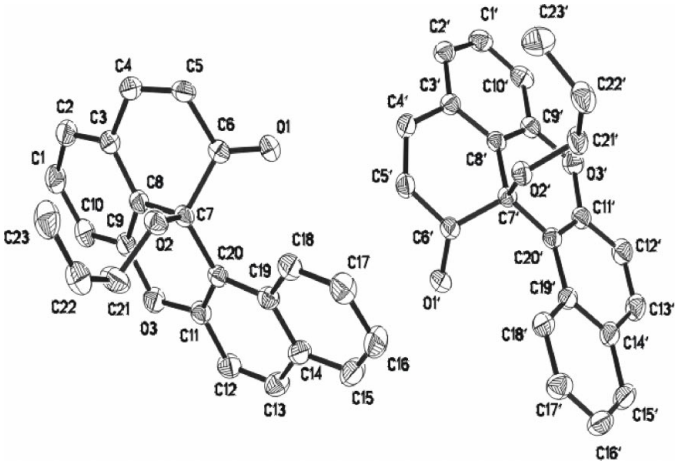


Figure 1
View of the title structure showing the atomic numbering scheme and displacement ellipsoids at the 50% probability level.

geometries of the two molecules show no unexpected features.

Experimental

The title compound, (II), was synthesized from 1,1′-bi-2-naphthol, (I), in *n*-propanol in the presence of dioxygen under the catalysis of the $CuCl_2$–ethanolamine complex. Compound (II) was isolated as the main product, in a yield of 92%. The single crystal used for X-ray analysis was recrystallized from acetone. Elemental analysis calculated for $C_{23}H_{18}O_3$ (%): C 80.68, H 5.30; found: C 80.97, H 5.34; m.p. 422–423 K. ν_{max}: 2959, 2931, 1700 (*s*, C═O), 1454, 1004, 814 cm^{-1}. δ_H (500 MHz in $CDCl_3$/TMS): 0.62 (*t, J* = 7.2 Hz, 3H, $-CH_3$), 1.24–1.38 (*m*, 2H, $-CH_2-$), 2.81 (*t, J* = 6.5 Hz, 2H, $-OCH_2-$), 6.26 (*d, J* = 9.8 Hz, 1H, H2), 7.04 (*d, J* = 7.2 Hz, 1H, H4), 7.07 (*d, J* = 8.5 Hz, 1H, H6), 7.16 (*d, J* = 10.4 Hz, 1H, H3), 7.30 (*d*, *J* = 9.1 Hz, 1H, H9), 7.35 (*t*, $J_{5H\text{-}4H}$ = 7.0 Hz, $J_{6H\text{-}5H}$ = 8.5 Hz, 1H, H5), 7.38–7.42 (*m*, 2H, H10, H13), 7.77–7.80 (*m*, 1H, H11), 7.87 (*d*, *J* = 9.1 Hz, 1H, H8), 8.14–8.17 (*m*, 1H, H12). δ_C (125 MHz): 10.65 (CH_3), 22.93 (CH_2), 66.06 (OCH_2), 75.15 (13*c*-C), 108.31, 116.34, 116.84, 117.20, 123.79, 124.59, 125.65, 126.27, 127.91, 128.10, 130.51, 130.95, 131.74, 132.88, 133.50, 139.00, 151.24, 152.02, 197.91 (C═O). FAB–MS *m*/*z* (%): 343 (M^+ + 1, 12), 342 (M^+, 20), 299 ($M^+-C_3H_7$, 30), 284 (54), 283 ($M^+-OC_3H_7$, 100).

Crystal data

$C_{23}H_{18}O_3$	D_x = 1.316 Mg m^{-3}
M_r = 342.37	Mo $K\alpha$ radiation
Monoclinic, $P2_1$	Cell parameters from 5917 reflections
a = 12.584 (3) Å	θ = 4.2–23.2°
b = 10.083 (2) Å	μ = 0.09 mm^{-1}
c = 13.956 (3) Å	T = 293 (2) K
β = 102.57 (3)°	Block, yellow
V = 1728.4 (7) Å^3	0.41 × 0.30 × 0.25 mm
Z = 4	

Data collection

CCD diffractometer	6047 reflections with $I > 2\sigma(I)$
φ and ω scans	R_{int} = 0.015
Absorption correction: empirical (Blessing, 1995)	θ_{max} = 30.0°
T_{min} = 0.965, T_{max} = 0.979	$h = -17 \rightarrow 17$
12 441 measured reflections	$k = -13 \rightarrow 14$
9327 independent reflections	$l = -7 \rightarrow 19$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0398P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.044	$(\Delta/\sigma)_{max}$ = 0.010
$wR(F^2)$ = 0.097	$\Delta\rho_{max}$ = 0.17 e Å^{-3}
S = 1.05	$\Delta\rho_{min}$ = −0.22 e Å^{-3}
9327 reflections	Absolute structure: Flack (1983)
482 parameters	Flack parameter = −0.4 (7)
H-atoms parameters constrained	

Table 1
Selected geometric parameters (Å, °).

C20′—C7′	1.509 (2)	C8—C7	1.514 (2)
C8′—C7′	1.513 (2)	C7′—C6′	1.539 (2)
O2′—C21′	1.435 (2)	O2—C21	1.430 (2)
O2′—C7′	1.4554 (18)	O2—C7	1.4529 (19)
C20—C7	1.502 (2)	C7—C6	1.543 (2)
C21′—O2′—C7′	113.99 (12)	C21—O2—C7	115.07 (13)
O2′—C7′—C20′	111.09 (13)	O2—C7—C20	111.37 (13)
O2′—C7′—C8′	109.32 (12)	O2—C7—C8	109.55 (13)
C20′—C7′—C8′	111.91 (13)	C20—C7—C8	111.82 (14)
O2′—C7′—C6′	103.99 (12)	O2—C7—C6	103.42 (12)
C20′—C7′—C6′	115.70 (13)	C20—C7—C6	115.60 (14)
C8′—C7′—C6′	104.29 (13)	C8—C7—C6	104.52 (13)

The structure was solved by direct methods and all non-H atoms were refined by the full-matrix least-squares method with anisotropic displacement parameters, except for C22 and C23 which are disordered over two positions. The H atoms were placed in calculated positions and refined using a riding model.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT-Plus* (Bruker, 1999); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

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