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N-(2-Hydroxyethyl)-13c-methoxy-1-oxo-1,13c-dihydrodibenzo[*a*,*kl*]xanthene-2-carboxamide

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Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.002 Å
R factor = 0.037
wR factor = 0.108
Data-to-parameter ratio = 15.4

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

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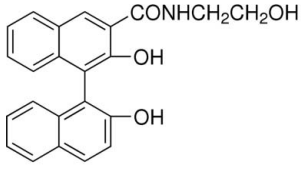
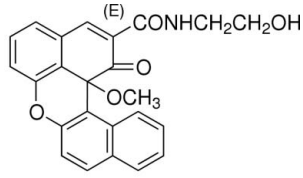
The title compound, $C_{24}H_{19}NO_5$, contains five fused rings, of which three are planar and the other two have twisted and flattened boat forms. The crystal structure is stabilized by intra- and intermolecular N—H···O, O—H···O and C—H···O hydrogen bonds.

Comment

1,1′-Binaphthol and its derivatives are used in a broad field of research. Optically pure binaphthol compounds have been used for various applications (Nishizawa *et al.*, 1981; Noyori *et al.*, 1979; Naruse *et al.*, 1988; Hesemann & Moreau, 2003). It has been reported (Tan *et al.*, 2001) previously that a copper–amine complex led to a domino reaction from binaphthol to yield xanthene.



(I)



(II)

The title compound, (II), was obtained from 1,1′-3-(*N*-2-hydroxyethyl)methaminobinaphthol, (I), oxidized by O_2, under the catalysis of the $CuCl_2$ complex of ethanolamine (1:1) in methanol. The oxidation reaction takes place at a low temperature. Three conversions occurred from (I) to (II): 2′-O—C_8 coupling, –OH oxidation and C_1—OCH_3 coupling.

Fig. 1 shows the molecular structure of (II), with the atomic numbering scheme. It contains five fused rings, *A* (C5–C10), *B* (C1–C4/C9/C10), *C* (C1/C2/O3/ C11–C13), *D* (C12–C17) and *E* (C11/C12/C17–C20), with the carbonyl and methoxy groups attached to atom C19 and the chiral atom C11, respectively. Rings *A*, *B* and *D* are planar. Rings *C* and *E* have total puckering amplitudes of 1.844 (3) and 0.480 (3) Å (Cremer & Pople, 1975) and twisted and flattened boat forms [φ = 150.1 (4)°, θ = 89.4 (5)° and φ = 161.3 (4), θ = 118.2 (5)°, respectively].

The crystal structure is stabilized by intramolecular N—H···O and C—H···O, and intermolecular N—H···O, O—H···O and C—H···O hydrogen bonds (Table 1, and Figs. 1 and 2).

Experimental

Under the catalysis of $CuCl_2$–ethanolamine (1:1) in methanol, (I) was oxidized by O_2 to give (II), which was purified through a short column of Al_2O_3 (eluted with petroleum ether-EtOAc). It was crystallized

from acetone (yield 40%, m.p. 431–433 K). IR (KBr, cm^{-1}): ν 3381 (OH), 3334 (OH), 3062 (Ar), 2931, 2886, 2816, 1708 (C═O), 1635, 1456, 1052, 750; ^{1}H NMR (CD_3OD, p.p.m.): δ 8.06 (1H, *s*), 8.04–8.03 (1H, *m*), 7.98 (1H, *d*, J = 8.0 Hz), 7.86–7.85 (1H, *m*), 7.53 (1H, *t*, J = 7.0 and 8.5 Hz), 7.43–7.41 (2H, *m*), 7.39–7.37 (2H, *m*), 7.27 (1H, *dd*, J = 8.5 and 1.0 Hz), 3.69 (2H, *td*, J = 7.5, 6.5 and 2.0 Hz), 3.54–3.48 (2H, *m*), 2.78 (3H, *s*); FAB-MS, *m*/z** (%): 401 ($[M]^+$, 3), 370 ($[M\text{-}OCH_3]^+$, 23). Analysis calculated for $C_{24}H_{19}NO_5$: C 71.81, H 4.77, N 3.49%; found: C 71.50, H 4.79, N 3.31%.

Crystal data

$C_{24}H_{19}NO_5$
M_r = 401.40
Monoclinic, $P2_1/c$
a = 7.275 (2) Å
b = 17.270 (6) Å
c = 15.342 (5) Å
β = 91.475 (6)°
V = 1926.9 (11) Å^3
Z = 4
D_x = 1.384 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from **25** reflections **[quite low - is this correct?]**
θ = 12–18°
μ = 0.10 mm^{-1}
T = 273 (2) K
Block, yellow
0.55 × 0.54 × 0.53 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.948, T_{max} = 0.950
11405 measured reflections
4170 independent reflections
3137 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 27.0°
$h = -8 \rightarrow 9$
$k = -16 \rightarrow 21$
$l = -18 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.108
S = 1.02
4170 reflections
271 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0506P)^2 + 0.3748P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O4	0.86	2.17	2.8317 (17)	133
N1—H1A···O1^{i}	0.86	2.34	3.0197 (19)	136
O1—H1B···O2^{ii}	0.82	1.99	2.8022 (18)	174
C8—H8A···O4	0.93	2.55	2.9246 (19)	104
C8—H8A···O5	0.93	2.54	3.0804 (20)	118
C15—H15A···O2^{iii}	0.93	2.46	3.3468 (18)	159

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

The H atoms were located in a difference map and constrained to ride on their parent atoms at distances of 0.82 (OH), 0.86 (NH), 0.93 (CH), 0.97 (CH_2) and 0.96 Å (CH_3), with U_{iso}(H) values of 1.2 (1.5 for methyl and hydroxy) times U_{eq}(C,N,O).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

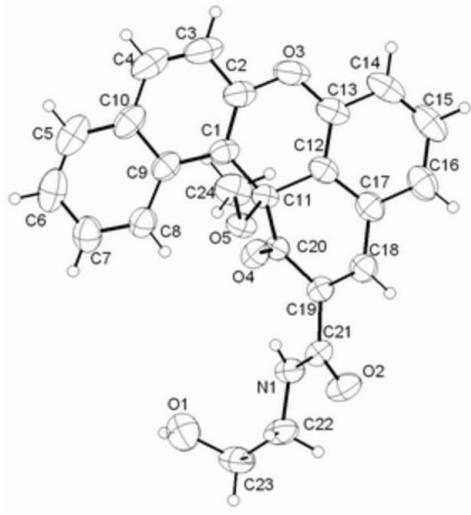


Figure 1
The molecular structure of (II), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

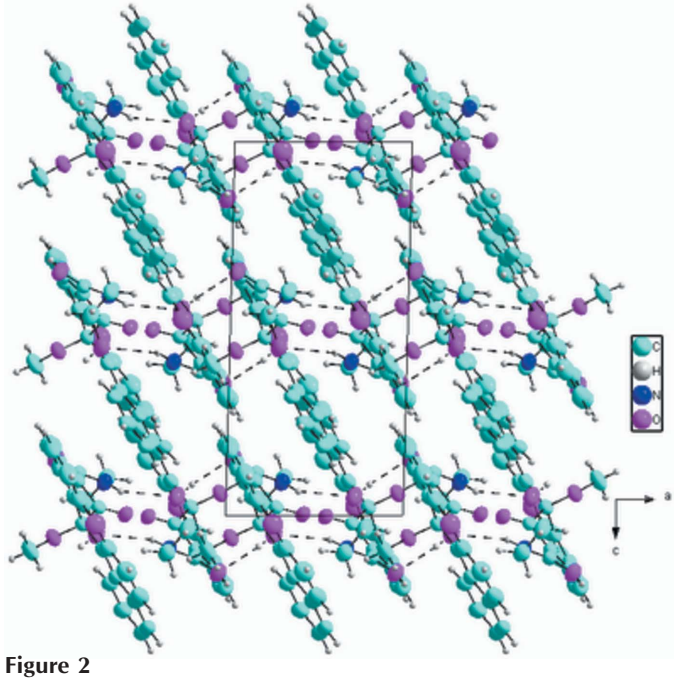


Figure 2
Packing diagram of (II); hydrogen bonds are shown as dashed lines.

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