N(2)	1.0013 (1)	0.5056 (3)	0.3375 (3)	0.032 (2)
C(2*A*)	1.0474 (2)	0.4652 (4)	0.3553 (4)	0.034 (2)
C(2*B*)	1.0725 (2)	0.5727 (5)	0.4335 (4)	0.048 (3)
C(2*G*)	1.0524 (2)	0.5866 (6)	0.5511 (5)	0.062 (4)
C(2*D*1)	1.0793 (3)	0.6983 (6)	0.6199 (5)	0.089 (5)
C(2*D*2)	1.0504 (4)	0.4506 (9)	0.6163 (6)	0.136 (7)
C(2)	1.0726 (2)	0.4441 (4)	0.2440 (5)	0.043 (3)
O(2)	1.1008 (1)	0.3464 (3)	0.2331 (3)	0.053 (2)
N(3)	1.0658 (1)	0.5366 (4)	0.1573 (3)	0.042 (2)
C(3*A*)	1.0870 (2)	0.5264 (5)	0.0470 (5)	0.051 (3)
C(3*B*)	1.0538 (2)	0.5597 (5)	−0.0495 (4)	0.051 (3)
O(3*G*1)	1.0312 (1)	0.6890 (4)	−0.0235 (4)	0.071 (3)
C(3*G*2)	1.0188 (2)	0.4404 (7)	−0.0582 (5)	0.070 (4)
C(3)	1.1295 (2)	0.6165 (5)	0.0367 (7)	0.066 (4)
O(3)	1.1469 (1)	0.6221 (5)	−0.0595 (5)	0.100 (4)
O(3*m*)	1.1457 (2)	0.6636 (4)	0.1307 (5)	0.077 (3)
C(3*m*)	1.1899 (3)	0.736 (1)	0.106 (1)	0.150 (8)
C(1*df*)	0.7891 (4)	0.9673 (9)	0.198 (1)	0.126 (8)
O(2*df*)	0.7727 (3)	1.0450 (8)	0.1243 (8)	0.195 (8)
N(3*df*)	0.7649 (2)	0.8986 (7)	0.2784 (7)	0.104 (5)
C(4*df*)	0.7171 (3)	0.911 (2)	0.300 (1)	0.18 (1)
C(5*df*)	0.7880 (3)	0.805 (1)	0.354 (1)	0.142 (8)

Table 2. *Selected torsion angles (°) with e.s.d.'s in parentheses*

	Phe	D-Leu	Thr
φ	−71.0 (4)*	66.8 (4)	−95.6 (5)
ψ	−26.8 (4)	41.4 (4)	14.3 (6)†
ω	178.7 (4)	−178.8 (5)	174.0 (6)‡
χ^1	−71.2 (4)	60.3 (4)	49.9 (4)¶
χ^2	−57.0 (6)	57.9 (5)	

* C(6*b*)—N(1)—C(1*A*)—C(1).
† N(3)—C(3*A*)—C(3)—O(3*m*).
‡ C(2*A*)—C(2)—O(2*m*)—C(2*m*).
¶N(3)—C(3*A*)—C(3*B*)—O(3*G*1).

The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1986). During the last stage of refinement all H atoms were placed at assumed positions with an overall isotropic thermal parameter (U = 0.05 Å^2) and were only included for structure-factor calculations. Refinement of non-H atoms was by full-matrix least-squares methods using *SHELX*76 (Sheldrick, 1976). The molecular conformation was drawn by the *ORTEP* program (Johnson, 1971).

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, bond distances and angles, and torsion angles have been deposited with the british Library Document Supply Centre as Supplementary Publication No. SUP 71108 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1046]

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Structure of 2-(1-Naphthyl)-5-phenyl-1,3-oxazole (α-NPO)

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Abstract

The α-NPO molecule is planar with an average deviation of non-H atoms from the least-squares plane of 0.02 (2) Å.

Comment

The laser output and lifetime of the title compound has been studied and successfully improved (Konjevic, 1988). Since the structure of the dye plays a significant role in its photophysical properties, the structure of α-NPO was determined as part of our structural laser-dye studies. The naphthyl, oxazole and phenyl rings of the molecule are coplanar with an average deviation of non-H atoms from the least-squares plane of 0.02 (2) Å. The bond lengths and angles in the oxazole ring of this compound are comparable with those of 2-amino-1,3-oxazole (Albinati & Marcon, 1981). The C—C bonds and C—C—C angles within the phenyl and naphthyl rings are typical of such systems, ranging from

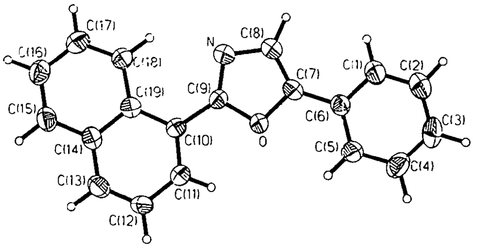

Fig. 1. A view of the molecule showing the labeling of the non-H atoms. Thermal ellipsoids are shown at the 50% probability level.

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1.353 (4) to 1.438 (3) Å and 117.6 (2) to 124.2 (2)°, respectively.

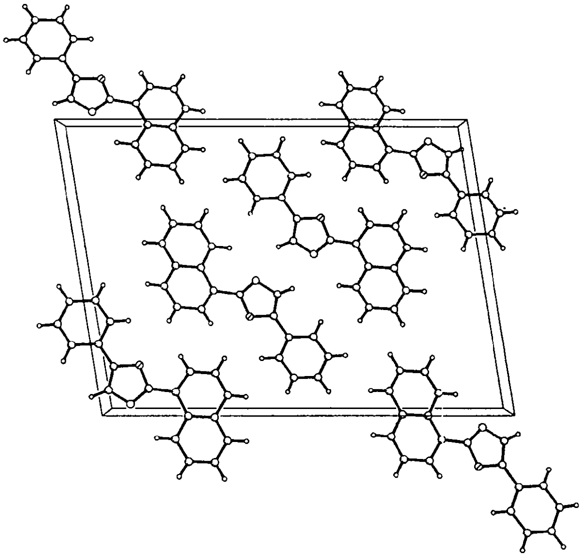

Fig. 2. Packing of the molecules viewed down the *b* axis.

Experimental

Crystal data

$C_{19}H_{13}NO$
M_r = 271.3
Monoclinic
$P2_1/n$
a = 15.722 (3) Å
b = 3.906 (1) Å
c = 21.969 (4) Å
β = 99.44 (2)°
V = 1330.8 (5) Å^3
Z = 4
D_x = 1.354 Mg m^{-3}
D_m = 1.345 Mg m^{-3}
Density measured by flotation in $CHCl_3/CH_2Cl_2$

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 50 reflections
θ = 2.5–17.5°
μ = 0.084 mm^{-1}
T = 298 K
Needle
0.70 × 0.15 × 0.10 mm
Colourless
Crystal source: Sigma Chemical Co. (grown from cyclohexane)

Data collection

Siemens *P*4 diffractometer
$2\theta/\theta$ scans (speed 5.33–29.3° min^{-1} in ω)
Absorption correction: analytical (Ugozzoli, 1987)
$T_{\min}$ = 0.879, $T_{\max}$ = 1.076
3637 measured reflections
3058 independent reflections

1306 observed reflections $[F > 4\sigma(F)]$
$R_{\rm int}$ = 0.0123
$\theta_{\max}$ = 22.5°
$h = 0 \rightarrow 21$
$k = 0 \rightarrow 6$
$l = -29 \rightarrow 29$
2 standard reflections monitored every 200 reflections
intensity variation: none

Refinement

Refinement on F
Final R = 0.0382
wR = 0.0338
S = 1.09
1306 reflections
242 parameters
All H-atom parameters refined

$w = 1/[\sigma^2F + 0.0002F^2]$
$(\Delta/\sigma)_{\max}$ = 0.046
$\Delta\rho_{\max}$ = 0.13 e Å^{-3}
$\Delta\rho_{\min}$ = −0.15 e Å^{-3}
Atomic scattering factors from *SHELXTL/PC* (Siemens Crystallographic Research Systems, 1990)

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$$U_{\rm eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
N	0.0450 (1)	0.2499 (6)	0.0780 (1)	0.048 (1)
O	0.1702 (1)	−0.0121 (4)	0.1086 (1)	0.038 (1)
C(1)	0.2390 (2)	0.0692 (8)	−0.0435 (1)	0.051 (1)
C(2)	0.3088 (2)	0.0023 (9)	−0.0716 (1)	0.059 (1)
C(3)	0.3826 (2)	−0.1361 (9)	−0.0385 (1)	0.059 (1)
C(4)	0.3858 (2)	−0.2126 (9)	0.0228 (1)	0.056 (1)
C(5)	0.3154 (2)	−0.1468 (8)	0.0515 (1)	0.045 (1)
C(6)	0.2412 (2)	−0.0010 (7)	0.0188 (1)	0.036 (1)
C(7)	0.1672 (2)	0.0799 (7)	0.0479 (1)	0.036 (1)
C(8)	0.0919 (2)	0.2373 (8)	0.0301 (1)	0.047 (1)
C(9)	0.0936 (1)	0.0983 (7)	0.1236 (1)	0.035 (1)
C(10)	0.0806 (1)	0.0293 (7)	0.1869 (1)	0.033 (1)
C(11)	0.1463 (2)	−0.1258 (7)	0.2268 (1)	0.039 (1)
C(12)	0.1394 (2)	−0.1948 (8)	0.2879 (1)	0.043 (1)
C(13)	0.0662 (2)	−0.1160 (8)	0.3098 (1)	0.044 (1)
C(14)	−0.0039 (1)	0.0421 (7)	0.2715 (1)	0.037 (1)
C(15)	−0.0800 (2)	0.1253 (8)	0.2944 (1)	0.046 (1)
C(16)	−0.1472 (2)	0.2783 (8)	0.2580 (1)	0.049 (1)
C(17)	−0.1416 (2)	0.3528 (8)	0.1967 (1)	0.049 (1)
C(18)	−0.0698 (2)	0.2780 (8)	0.1724 (1)	0.043 (1)
C(19)	0.0023 (1)	0.1189 (6)	0.2090 (1)	0.034 (1)

Table 2. *Geometric parameters* (Å, °)

N—C(8)	1.382 (3)	C(4)—C(5)	1.384 (4)
O—C(7)	1.375 (3)	C(5)—C(6)	1.388 (3)
C(1)—C(2)	1.370 (4)	C(6)—C(7)	1.451 (4)
C(7)—C(8)	1.333 (4)	C(10)—C(11)	1.381 (3)
C(9)—C(10)	1.463 (3)	C(11)—C(12)	1.389 (4)
C(10)—C(19)	1.438 (3)	C(12)—C(13)	1.353 (4)
C(14)—C(19)	1.425 (3)	C(13)—C(14)	1.414 (3)
N—C(9)	1.299 (3)	C(14)—C(15)	1.409 (4)
O—C(9)	1.370 (3)	C(15)—C(16)	1.354 (4)
C(1)—C(6)	1.389 (3)	C(16)—C(17)	1.395 (4)
C(2)—C(3)	1.375 (4)	C(17)—C(18)	1.357 (4)
C(3)—C(4)	1.374 (4)	C(18)—C(19)	1.420 (3)
C(8)—N—C(9)	104.5 (2)	C(7)—O—C(9)	105.0 (2)
C(2)—C(1)—C(6)	120.9 (2)	C(1)—C(2)—C(3)	120.4 (3)
C(2)—C(3)—C(4)	119.7 (3)	C(3)—C(4)—C(5)	120.1 (3)
C(4)—C(5)—C(6)	120.6 (2)	C(1)—C(6)—C(5)	118.3 (2)
C(1)—C(6)—C(7)	119.8 (2)	C(5)—C(6)—C(7)	121.9 (2)
O—C(7)—C(6)	117.8 (2)	O—C(7)—C(8)	107.0 (2)
C(6)—C(7)—C(8)	135.2 (2)	N—C(8)—C(7)	110.7 (2)
N—C(9)—O	112.8 (2)	N—C(9)—C(10)	131.3 (2)
O—C(9)—C(10)	115.9 (2)	C(9)—C(10)—C(11)	118.5 (2)
C(9)—C(10)—C(19)	122.5 (2)	C(11)—C(10)—C(19)	119.0 (2)
C(10)—C(11)—C(12)	122.0 (2)	C(11)—C(12)—C(13)	120.2 (2)
C(12)—C(13)—C(14)	121.0 (2)	C(13)—C(14)—C(15)	120.9 (2)
C(13)—C(14)—C(19)	119.6 (2)	C(15)—C(14)—C(19)	119.4 (2)
C(14)—C(15)—C(16)	121.1 (2)	C(15)—C(16)—C(17)	119.7 (3)
C(16)—C(17)—C(18)	121.5 (2)	C(17)—C(18)—C(19)	120.6 (2)
C(10)—C(19)—C(14)	118.2 (2)	C(10)—C(19)—C(18)	124.2 (2)
C(14)—C(19)—C(18)	117.6 (2)		

The structure was solved by direct methods and refined by full-matrix least squares. All H atoms were located from the difference Fourier map. The *SHELXTL/PC* program package was used for all calculations.

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Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71044 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: LI1036]

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A Twisted Pentacyclic Diol Derviative

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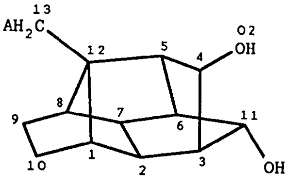
Abstract

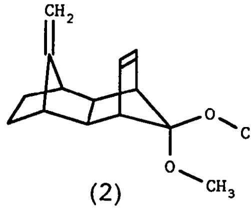
The X-ray crystal structure was determined for (*exo*,*anti*)-9-methylpentacyclo[6.3.1.0^{3,10}.0^{4,12}.0^{5,9}]dodecane-2,11-diol (1). The molecule consists of a twisted pentacyclic hydrocarbon ring system with a methyl substituent and two hydroxyl functional groups. Despite the two hydroxyl groups, there is no evidence of intramolecular hydrogen bonding.

Comment

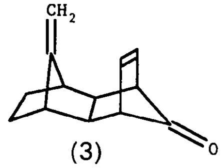
Attempted hydrolysis of the dimethoxyketal in (2) with aqueous strong acids resulted in the formation of mainly ring-closed twisted products (Lloyd, Arif, Allred, Patton & Sharp, 1993) rather than the ketone (3). However, (3) can be isolated as the major product from deuterolysis of (2) with the weakly acidic salt 2,6-lutidinium tosylate-*d* in D_2O. Ketone (3) can be reduced to the *anti*-alcohol (4) whose diene system, like that of (2) and (3), is acid sensitive. Treatment of (4) with strong aqueous acid results in the formation of the racemic ring-closed twisted product (1), the title compound.



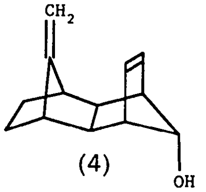
(1) A = H
(1*d*) A = D



(2)



(3)



(4)

There are three intermolecular contacts involving oxygen: O(1$^{\rm i}$)···O(1$^{\rm ii}$), O(1$^{\rm i}$)···O(2$^{\rm ii}$) and O(1$^{\rm i}$)···O(2$^{\rm ii}$), of 2.685 (7), 2.839 (4) and 3.089 (5) Å, respectively, which are near the sum of the van der Waals radii, 2.8 Å (Bondi, 1964). These data and angles O(1$^{\rm i}$)—H(O1$^{\rm i}$)···O(2$^{\rm ii}$) and O(1$^{\rm i}$)···H(O2$^{\rm ii}$)—O(2$^{\rm ii}$) (157.3 and 150.6°, respectively) are consistent with intermolecular hydrogen bonding. There is no evidence for intramolecular hydrogen bonding, as expected with the *exo*- and *anti*-hydroxyl group geometries. There were no other non-hydrogen intermolecular contacts ≤ 3.5 Å.

Bond lengths and bond angles are similar to those of a previously reported derivative having the same pentacyclic ring structure (Lloyd, Arif, Allred, Patton & Sharp, 1993). The two longest bonds, C(2)—C(7) and C(5)—C(6), are also consistent with those of other derivatives (Astin, Fletcher, Mackenzie, Miller, Ratcliffe, Frew & Muir, 1982; Khan, Bauer & Khan, 1972), but the C(6)—C(7) bond is not as short as those found in these structures.

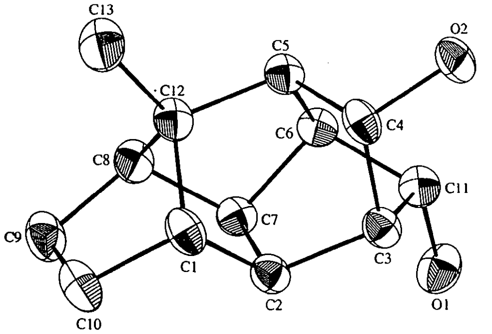


Fig. 1. Drawing of non-H atoms of one of the enantiomers of (1) with thermal ellipsoids at the 40% probability level.

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† Deceased 8 July 1991.

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