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A Naphthofuranoquinone

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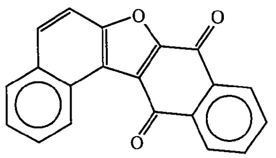
Abstract

The title compound, dinaphtho[2,1-*b*:2′,3′-*d*]furan-8,13-(8*H*,13*H*)-dione, $C_{20}H_{10}O_3$, was prepared in low yield from the reaction of (2-chloroethoxy)ethyl 2-tetrahydropyranyl ether with (*R*)-1,1′-binapth-2,2′-ol. The ring system is virtually planar [the largest deviation from the mean plane of the ring is 0.116 (4) Å for atom C13] despite the O13···H1 short contact of 2.27 Å.

Comment

The title compound, dinaphtho[2,1-*b*:2′,3′-*d*]furan-8,13-(8*H*,13*H*)-dione, (I), was prepared serendipitously in low yield. It is presumably formed *via* oxidative aryl migration and a ring-closure reaction in which a new furan ring is formed.



(I)

Despite the relatively short intramolecular O13···H1 contact distance of 2.27 Å, the whole ring system is virtually planar (Fig. 1). The maximum deviation from the least-squares plane of the ring atoms (*i.e.* all C atoms and O7) is 0.116 (4) Å for atom C13. The O13 atom lies 0.267 (4) Å out of the mean plane, presumably in order to reduce steric interactions with H1. Although compound (I) was prepared from optically pure binaphthol and crystallizes in a non-centrosymmetric space group, it was possible neither to detect any optical activity from (I) nor to determine the absolute configuration. This is not too surprising, since any chirality in (I) must arise from deviations of the ring system from coplanarity. Only one other compound containing the same ring system has been crystallographically characterized, with an *N*-(2-pyridyl)carboxamide group substituted in the 6-position. A similar short intramolecular O···H contact of 2.16 Å was observed and the steric distortions arising from this have been discussed in detail (Goldstein, 1975).

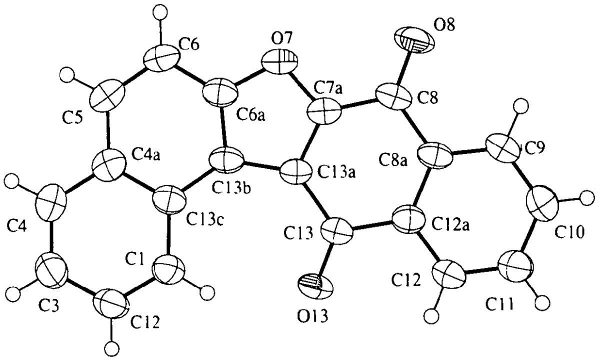


Fig. 1. The structure of (I) showing 50% probability displacement ellipsoids. H atoms are shown as spheres of arbitrary radii.

As expected, the molecules of (I) pack in parallel layers with relatively short intra-ring distances. The distance between the ring mean planes is 3.25 (1) Å. There are no unusually short contacts to the O atoms, indicating no significant hydrogen bonding.

Experimental

A solution of (2-chloroethoxy)ethyl 2-tetrahydropyranyl ether (10 g) in butanol was added to a stirred solution of (*R*)-1,1′-binapth-2,2′-ol (5 g) and sodium hydroxide (0.5 g). After stirring for 10 h and following the procedure of Kyba *et al.* (1977) for the isolation of (*R*)-2,2′-bis(5-hydroxy-3-oxa-1-pentyloxy)-1,1′-binapthyl, a small quantity (*ca* 0.05 g) of golden-yellow crystals of the title compound was isolated. Another synthesis of (I) has been reported previously (Ishikawa, Hinoshita, Tagaki & Ueno, 1988).

Crystal data

$C_{20}H_{10}O_3$
$M_r = 298.28$
Orthorhombic
$P2_12_12_1$
$a = 5.4660 (10)$ Å
$b = 6.1280 (10)$ Å
$c = 39.841 (9)$ Å
$V = 1334.5 (4)$ Å^3
$Z = 4$
$D_x = 1.485$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 21 reflections
$\theta = 8.5–13.7°$
$\mu = 0.100$ mm^{-1}
$T = 292 (2)$ K
Prism
0.35 × 0.30 × 0.20 mm
Golden yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
1430 measured reflections
1430 independent reflections
1068 observed reflections [$I > 2\sigma(I)$]

$\theta_{max} = 24.96°$
$h = -6 \rightarrow 0$
$k = 0 \rightarrow 7$
$l = -47 \rightarrow 0$
3 standard reflections
frequency: 120 min
intensity decay: 1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0428$
$wR(F^2) = 0.1400$
$S = 1.088$
1426 reflections
210 parameters
H atoms riding
$w = 1/[\sigma^2(F_o^2) + (0.0553P)^2 + 0.6156P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.212$ e Å^{-3}
$\Delta\rho_{\min} = -0.221$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0115 (36)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

$U_{\rm eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	$U_{\rm eq}$
C1	0.6631 (7)	0.3219 (7)	0.56877 (10)	0.0482 (11)
C2	0.5240 (9)	0.2755 (8)	0.54120 (10)	0.0535 (12)
C3	0.5642 (9)	0.0856 (8)	0.52263 (11)	0.0555 (12)
C4	0.7441 (9)	−0.0548 (8)	0.53222 (11)	0.0537 (12)
C4a	0.8954 (8)	−0.0118 (7)	0.56031 (10)	0.0467 (11)
C5	1.0894 (8)	−0.1563 (7)	0.56915 (11)	0.0537 (12)
C6	1.2432 (8)	−0.1156 (7)	0.59522 (11)	0.0501 (12)
C6a	1.1960 (7)	0.0731 (7)	0.61370 (10)	0.0440 (11)
C7a	1.2304 (7)	0.3241 (7)	0.65208 (10)	0.0407 (10)
C8	1.3280 (7)	0.4370 (7)	0.68167 (10)	0.0442 (11)
C8a	1.1925 (7)	0.6374 (7)	0.69053 (10)	0.0407 (10)
C9	1.2736 (8)	0.7636 (7)	0.71762 (10)	0.0485 (12)
C10	1.1468 (8)	0.9508 (8)	0.72662 (11)	0.0522 (12)
C11	0.9411 (8)	1.0132 (8)	0.70911 (10)	0.0508 (12)
C12	0.8596 (7)	0.8897 (7)	0.68221 (10)	0.0451 (11)
C12a	0.9829 (7)	0.7020 (7)	0.67284 (10)	0.0404 (10)
C13	0.8855 (7)	0.5739 (7)	0.64359 (10)	0.0403 (10)
C13a	1.0319 (7)	0.3836 (6)	0.63334 (10)	0.0376 (9)
C13b	1.0077 (7)	0.2195 (7)	0.60734 (9)	0.0400 (10)
C13c	0.8508 (7)	0.1820 (7)	0.57907 (10)	0.0420 (10)
O7	1.3353 (5)	0.1374 (5)	0.64080 (7)	0.0480 (8)
O8	1.5060 (5)	0.3706 (5)	0.69703 (7)	0.0600 (9)
O13	0.6960 (5)	0.6258 (5)	0.63008 (7)	0.0558 (9)

Table 2. *Selected geometric parameters (Å, °)*

C1—C2	1.366 (6)	C8—O8	1.219 (4)
C1—C13c	1.399 (5)	C8—C8a	1.477 (6)
C2—C3	1.397 (6)	C8a—C9	1.400 (6)
C3—C4	1.361 (6)	C8a—C12a	1.402 (5)
C4—C4a	1.416 (6)	C9—C10	1.387 (6)
C4a—C13c	1.424 (6)	C10—C11	1.377 (6)
C4a—C5	1.426 (6)	C11—C12	1.386 (5)
C5—C6	1.359 (6)	C12—C12a	1.384 (5)
C6—C6a	1.395 (6)	C12a—C13	1.503 (5)
C6a—O7	1.379 (5)	C13—O13	1.210 (4)
C6a—C13b	1.388 (5)	C13—C13a	1.472 (5)
C7a—O7	1.357 (5)	C13a—C13b	1.450 (5)
C7a—C13a	1.367 (5)	C13b—C13c	1.434 (5)
C7a—C8	1.467 (6)		
O7—C6a—C13b	111.6 (4)	O13—C13—C13a	123.4 (4)
O7—C6a—C6	123.2 (4)	O13—C13—C12a	120.7 (4)
C13b—C6a—C6	125.2 (4)	C7a—C13a—C13b	106.1 (4)
O7—C7a—C13a	112.3 (4)	C7a—C13a—C13	119.4 (4)
O7—C7a—C8	120.7 (3)	C13b—C13a—C13	134.3 (3)
C13a—C7a—C8	127.0 (4)	C6a—C13b—C13c	118.9 (4)
O8—C8—C7a	122.4 (4)	C6a—C13b—C13a	104.5 (3)
O8—C8—C8a	123.9 (4)	C13c—C13b—C13a	136.6 (4)

H atoms were included at calculated positions (C—H 0.96 Å) riding on their attached C atoms, with $U(\mathrm{H}) = 1.2U_{\rm eq}(\mathrm{C})$. The refined value of the Flack (1983) absolute-structure parameter [0 (3)] indicates that the absolute configuration could not be determined reliably from the crystallographic data.

Data collection: *CAD*-4 *EXPRESS Software* (Enraf–Nonius, 1992). Cell refinement: *CAD*-4 *EXPRESS Software*. Data reduction: local programs. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: locally modified version of *ORTEP*II (Johnson, 1976; Mallinson & Muir, 1985). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CF1045). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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cis-Perhydro-1,3-dimethyl-4,5-(epoxyethanoxy)purine-2,6,8-trione

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Abstract

The title compound, $C_9H_{12}N_4O_5$, adopts a distinct ring-twisted conformation defined by nearly orthogonal bridgehead torsion angles [C16—C15—C14—N19 −90.1 (2) and C26—C25—C24—N29 −87.8 (2)°]. The antiperiplanar array [C17—O14—C14—N19 −158.0 (2)