ular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL*93.

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(±)-6,6′-Dinitro-1,1′-binaphthyl 2,2′-diacetate

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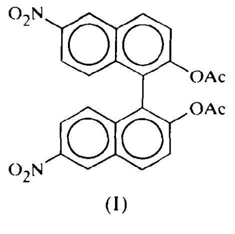
Abstract

The title compound, $C_{24}H_{16}N_2O_8$, is a twofold symmetric ether with two acetyl groups attached to the O atoms of the 1,1′-bi-2-naphthol. The molecular structure confirms that the nitro substituents are at the 6,6′ positions.

Comment

Optically active 1,1′-bi-2-naphthol and its derivatives are excellent ligands for chiral catalysts and have a C_2 symmetry axis (Narasaka, 1991; Mikami & Motoyama, 1995). Metal complexes of these molecules have been used for enantioselective reactions such as aldol reactions, Michael additions (Shibasaki & Sasai, 1996; Keller *et al.*, 1997) and Diels–Alder reactions (Mikami *et al.*, 1994; Markó *et al.*, 1996). Phosphorus amidites derived from 1,1′-bi-2-naphthol are particularly effective in the asymmetric copper-catalyzed conjugate addition of Et_2Zn to enones (Feringa *et al.*, 1997). The title compound, (I), is a derivative of 1,1′-bi-2-naphthol.



(I)

The monoclinic unit cell contains four discrete units of the title compound. Between the molecules, weak hydrogen bonding is observed (Berkovitch-Yellin & Leiserowitz, 1984; Steiner, 1997). The C—H···O distance for C2—H2···O4($\frac{1}{2} + x, -y, 1 + z$) is 3.290 (3) Å [C14—H14···O3($\frac{1}{2} + x, -y, z$) 3.194 (3), C18—H18···O8($x, \frac{1}{2} - y, z - \frac{1}{2}$) 3.244 (2), C19—H19···O5($x - 1, \frac{1}{2} - y, \frac{1}{2} + z$) 3.407 (3), C24—H24″···O1($x - 1, y, z$) 3.146 (3) and C7—H7···O7($x, y, z - 1$) 3.255 (2) Å]. The torsion angle between the two naphthyl fragments is 72.08 (6)°.

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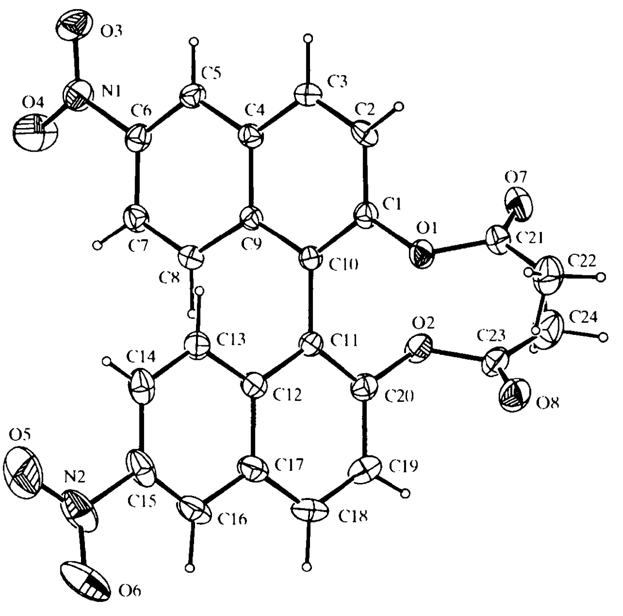

Fig. 1. *ORTEP* (Johnson, 1965) drawing with displacement ellipsoids at the 50% probability level. H atoms are drawn as circles of an arbitrary radius.

Experimental

The title compound was obtained by nitration of racemic 1,1′-bi-2-naphthol in acetic acid at 373 K (Ioffe, 1936) and then subsequent reaction with acetyl chloride in pyridine. The title compound was obtained after crystallization from ethyl acetate.

Crystal data

$C_{24}H_{16}N_2O_8$
$M_r = 460.40$
Monoclinic
Ia
$a = 7.506 (1)$ Å
$b = 29.143 (2)$ Å
$c = 9.832 (1)$ Å
$\beta = 99.04 (1)°$
$V = 2124.0 (4)$ Å^3
$Z = 4$
$D_x = 1.440$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 22 reflections
$\theta = 15.14–21.85°$
$\mu = 0.11$ mm^{-1}
$T = 130$ K
Rectangular block
0.38 × 0.25 × 0.25 mm
Yellow

Data collection

Enraf–Nonius CAD-4F single-crystal diffractometer
$\omega/2\theta$ scans
Absorption correction: none
3625 measured reflections
3625 independent reflections
3319 reflections with $I > 2\sigma(I)$

$\theta_{\max} = 26.48°$
$h = -9 \rightarrow 9$
$k = -36 \rightarrow 36$
$l = -12 \rightarrow 12$
3 standard reflections
frequency: 180 min
intensity decay: 4.0%

Refinement

Refinement on F^2
$R(F) = 0.032$
$wR(F^2) = 0.083$
$S = 1.041$
3625 reflections
371 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0565P)^2 + 0.2418P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.161$ e Å^{-3}
$\Delta\rho_{\min} = -0.154$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

O1—C1	1.400 (2)	O5—N2	1.224 (4)
O1—C21	1.369 (2)	O6—N2	1.210 (3)
O2—C20	1.402 (2)	O7—C21	1.194 (3)
O2—C23	1.374 (2)	O8—C23	1.191 (2)
O3—N1	1.216 (2)	N1—C6	1.469 (2)
O4—N1	1.218 (2)	N2—C15	1.474 (3)
C1—O1—C21	117.62 (14)	N1—C6—C7	118.90 (16)
C20—O2—C23	115.67 (14)	N2—C15—C14	118.2 (2)
O3—N1—O4	123.27 (18)	N2—C15—C16	118.45 (19)
O3—N1—C6	119.20 (17)	O2—C20—C11	118.95 (16)
O4—N1—C6	117.53 (17)	O2—C20—C19	117.67 (19)
O5—N2—O6	124.1 (2)	O1—C21—O7	122.37 (18)
O5—N2—C15	117.2 (2)	O1—C21—C22	110.46 (18)
O6—N2—C15	118.7 (2)	O7—C21—C22	127.2 (2)
O1—C1—C2	118.92 (15)	O2—C23—O8	122.16 (19)
O1—C1—C10	117.55 (15)	O2—C23—C24	111.41 (17)
N1—C6—C5	118.17 (16)	O8—C23—C24	126.4 (2)

The positional and anisotropic displacement parameters for the non-H atoms were refined. A subsequent difference Fourier synthesis resulted in the location of all the H atoms, whose coordinates and isotropic displacement parameters were refined.

The absolute configuration of the structure of the crystal could not be reliably determined: refinement of the Flack parameter (Flack, 1983) gave an ambiguous result [$x = -0.8 (7)$].

Data collection: *CAD*-4 *UNIX Software* (Enraf-Nonius, 1994). Cell refinement: *SET*4 (de Boer & Duisenberg, 1984). Data reduction: *HELENA* (Spek, 1993). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *ORTEP* (Johnson, 1965) and *PLATON* (Spek, 1994). Software used to prepare material for publication: *PLATON* (Spek, 1990).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1240). Services for accessing these data are described at the back of the journal.

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[(3*R*,4*S*,4a*R*,10a*S*)-5,10-Diacetyl-3,4,4a,5,6,7,10,10a-octahydro-8-methoxy-6-oxo-2*H*-pyrano[3,2-*g*]pteridin-3,4-diyl diacetate†

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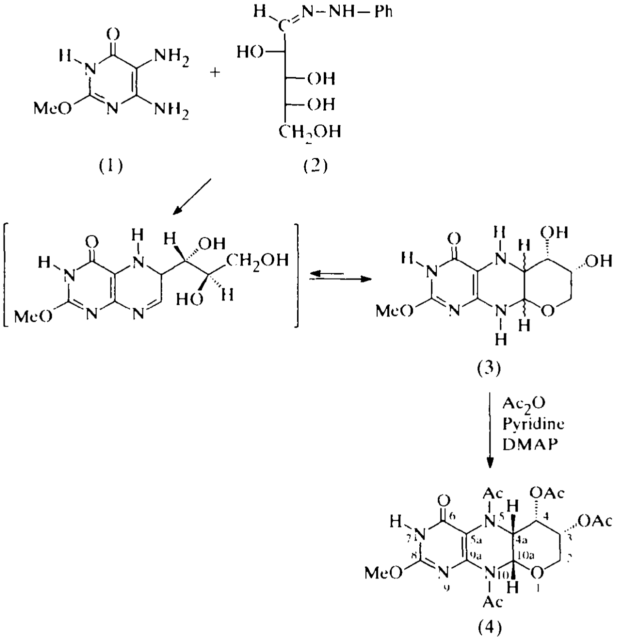
Abstract

The structure and stereochemistry of the title compound, $C_{18}H_{22}N_4O_9$, have been determined. The H atoms at the *A*/*B* ring fusion are in the *cis* position. There is one hydrogen bond in the structure, N7—H7···O51 [N···O 2.886 (4) Å], which links the molecules into infinite chains along the *a* axis.

Comment

In a previous paper (Low *et al.*, 1995), we described the preparation and structure of (3*R*,4*R*,4a*S*,10a*R*)-5-acetyl-3,4,4a,5,6,7,10,10a-octahydro-8-methoxy-7-methyl-6-oxo-2*H*-pyrano[3,2-*g*]pteridin-3,4-diyl diacetate, $C_{17}H_{22}N_4O_8$, (5), which is closely related to the title compound, (4). The present paper thus continues our investigations of the preparation and structural study of compounds formed by the reactions of substituted 5,6-diaminopyrimidines with carbohydrate derivatives.



It was not possible to establish the absolute structure of (4) from the X-ray data, but this was already known from the configuration of the D-arabinose phenylhydrazone used in its synthesis; Fig. 1 and the various data tables all refer to this known configuration. The bond lengths and angles in (4) are in broad agreement with those in (5), and are those expected for this type of molecule (Orpen *et al.*, 1994). Structure (4), like (5), has *cis* H atoms at the *A*/*B* ring fusion and double bonds at C5a=C9a and C8=N9 (Table 1). However, molecules (4) and (5) differ conformationally as expected [in the following discussion the values for (5) are given in square brackets]. The torsion angles N7—C8—N9—C9a, C4—C4a—C10a—O1 and N5—C4a—C10a—N10 are 5.9 (5) [−2.0 (4)], −2.7 (4) [−47.1 (5)] and 2.9 (4)° [−52.0 (5)°], respectively. Ring *A* has a boat conformation (Boeyens, 1978) with the following Cremer & Pople (1975) puckering parameters: Q = 0.755 (3) Å [0.504 (5) Å], θ = 89.8° [6.5 (6)°] and φ = 359.6 (3)° [54 (5)°]. The central *B* ring has a boat conformation, with Q = 0.538 (3) Å [0.481 (5) Å], θ = 86.9 (3)° [125.6 (6)°] and φ = 0.5 (4)° [236.6 (7)°]. The *C* ring is almost planar but is deformed slightly to a skew-boat, with Q = 0.096 (3) Å [0.093 (5) Å], θ =

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† Alternative name: 5,10-diacetyl-3,4,5a,6,7,8,9a,10-octahydro-2-methoxy-4-oxo-5*H*-pyrano[3,2-*g*]pteridin-6,7-diyl diacetate.

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