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8,9,10,11-Tetrahydrobenz[*c*]acridine

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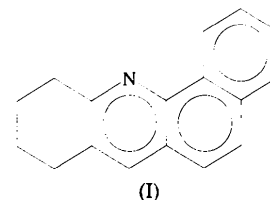
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Abstract

The molecule of the title compound, C₁₇H₁₅N, is planar except for the cyclohexane ring which is in a half-chair conformation.

Comment

Polycyclic aromatic hydrocarbons are useful in carcinogenic studies (Lehr & Jerina, 1983; Kar, Karmakar & Ray, 1989) and are also important for studying the role of 'bay regions' in a molecule towards carcinogenic activity (Kumar, 1985). As part of studies on the synthesis, characterization and bioactivity of polycyclic aromatic hydrocarbon derivatives, the structure of 8,9,10,11-tetrahydrobenz[*c*]acridine, (I), has been determined.



A displacement ellipsoid plot of (I) with the atom-numbering scheme is shown in Fig. 1. The bond lengths and angles observed in this structure are normal, and the cyclohexane ring (C1–C6) is in a half-chair conformation; the asymmetry parameter $\Delta C_2(C1-C6)$ is 0.026 (1) (Nardelli, 1983a). The cyclohexane rings of a related structure, 1,2,3,4,6,7-hexahydro-10-chloronaphth-[3,2-*c*]acridine, were found to undergo conformational flexibility with major and minor disorder components (Ray, Halder, Nigam, Sivakumar & Fun, 1995), but in the present structure, only one conformation is observed. The r.m.s. deviation of the C and N atoms, other than the C2, C3, C4 and C5 atoms, from their mean plane is 0.025 (2) Å and the dihedral angle between

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the planes of the naphthalene moiety and the pyridine ring is 2.61 (3)°. The molecule as a whole is therefore essentially planar, except for the cyclohexane ring, and lies approximately parallel to the *ac* plane. The shortest C...C and N...C contacts are C1...C12ⁱ of 3.352 (2) Å and N1...C12ⁱ of 3.442 (2) Å, respectively [symmetry code: (i) $\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z$], and represent normal van der Waals forces.

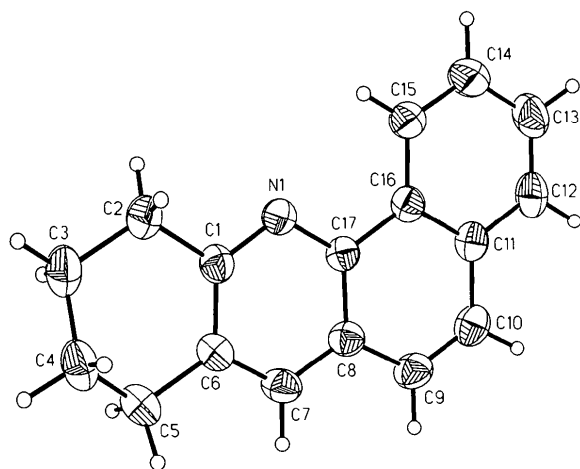


Fig. 1. The molecular structure of (I) showing 50% probability displacement ellipsoids and the atom-numbering scheme.

Experimental

The title compound was prepared from 2-chloro-1-cyclohexenecarbaldehyde by condensation with 1-naphthylamine (Halder, Kar & Ray, 1993). Single crystals were obtained by slow evaporation of a diethyl ether solution of the compound.

Crystal data

C₁₇H₁₅N
M_r = 233.30
 Monoclinic
*P*2₁/*n*
a = 7.4402 (8) Å
b = 14.8767 (13) Å
c = 11.5652 (12) Å
 β = 105.539 (9)°
V = 1233.3 (2) Å³
Z = 4
D_x = 1.257 Mg m⁻³
D_m not measured

Mo *K*α radiation
 λ = 0.71073 Å
 Cell parameters from 25 reflections
 θ = 8–25°
 μ = 0.073 mm⁻¹
T = 293 (2) K
 Rectangular slab
 0.58 × 0.42 × 0.40 mm
 Colourless

Data collection

Siemens *P4* diffractometer
 $\theta/2\theta$ scans
 Absorption correction: none
 3721 measured reflections
 2827 independent reflections
 2078 observed reflections
 $[I > 2\sigma(I)]$
R_{int} = 0.0235

θ_{\max} = 27.50°
 $h = -1 \rightarrow 9$
 $k = -1 \rightarrow 19$
 $l = -15 \rightarrow 14$
 3 standard reflections monitored every 97 reflections
 intensity decay: 3%

Refinement

Refinement on *F*²
R(*F*) = 0.0429
 wR (*F*²) = 0.1356
S = 1.064
 2827 reflections
 224 parameters
 All H-atom parameters refined
 $w = 1/[\sigma^2(F_o^2) + (0.0803P)^2 + 0.0166P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.265 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.144 \text{ e \AA}^{-3}$
 Extinction correction: *SHELXL93*
 Extinction coefficient: 0.029 (4)
 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)

$$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{eq}</i>
N1	0.24170 (13)	0.39584 (7)	0.50480 (8)	0.0378 (3)
C1	0.2815 (2)	0.40870 (7)	0.62271 (10)	0.0370 (3)
C2	0.4846 (2)	0.42365 (11)	0.68532 (12)	0.0487 (3)
C3	0.5208 (2)	0.45623 (11)	0.81471 (12)	0.0541 (4)
C4	0.4048 (2)	0.40396 (10)	0.88069 (11)	0.0529 (4)
C5	0.1983 (2)	0.41917 (10)	0.82232 (11)	0.0486 (3)
C6	0.1457 (2)	0.40436 (8)	0.68801 (10)	0.0385 (3)
C7	-0.0336 (2)	0.38547 (9)	0.62510 (11)	0.0423 (3)
C8	-0.0809 (2)	0.36977 (8)	0.50104 (10)	0.0382 (3)
C9	-0.2656 (2)	0.34648 (9)	0.43224 (11)	0.0452 (3)
C10	-0.3021 (2)	0.32843 (9)	0.31385 (11)	0.0447 (3)
C11	-0.1594 (2)	0.33440 (8)	0.25206 (10)	0.0382 (3)
C12	-0.1975 (2)	0.31635 (9)	0.12781 (11)	0.0470 (3)
C13	-0.0605 (2)	0.32280 (11)	0.07005 (12)	0.0565 (4)
C14	0.1201 (2)	0.34747 (11)	0.13325 (12)	0.0603 (4)
C15	0.1619 (2)	0.36503 (10)	0.25416 (11)	0.0488 (3)
C16	0.0238 (2)	0.35876 (8)	0.31588 (10)	0.0364 (3)
C17	0.06394 (15)	0.37605 (7)	0.44404 (9)	0.0338 (3)

Table 2. Selected bond lengths (Å)

N1—C1	1.3294 (14)	C8—C9	1.434 (2)
N1—C17	1.3534 (14)	C9—C10	1.349 (2)
C1—C6	1.416 (2)	C10—C11	1.433 (2)
C1—C2	1.507 (2)	C11—C16	1.412 (2)
C2—C3	1.527 (2)	C11—C12	1.414 (2)
C3—C4	1.511 (2)	C12—C13	1.364 (2)
C4—C5	1.520 (2)	C13—C14	1.395 (2)
C5—C6	1.513 (2)	C14—C15	1.374 (2)
C6—C7	1.367 (2)	C15—C16	1.403 (2)
C7—C8	1.402 (2)	C16—C17	1.4543 (14)
C8—C17	1.409 (2)		

The structure of (I) was solved by direct methods and refined by full-matrix least-squares techniques. The H atoms were located from difference maps and refined isotropically.

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXL93*. Molecular geometry: *PARST* (Nardelli, 1983b).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CF1059). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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5'-(2-Hydroxy-5-hydroxymethyl-3-methoxyphenyl)-6,9-bis(hydroxymethyl)-3',4,11-trimethoxydibenzo[*d,f*][1,3]dioxepine-2-spiro-4'-cyclohexa-2',5'-dienone

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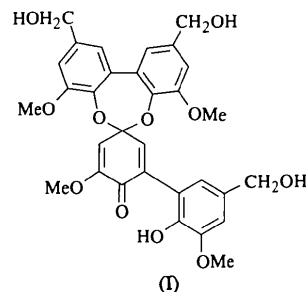
Abstract

The title compound C₃₁H₃₀O₁₁, an oxidation product of dehydrodivanillyl alcohol, contains two *o,o*-dihydroxybiphenyl ring systems joined together with a dioxepin ring structure.

Comment

During our exploration of the oxidative coupling behaviour of *o,o*-dihydroxybiphenyl compounds related to

wood lignins, we have obtained a tetrameric dibenzo[*d,f*][1,3]dioxepin, (I), from dehydrodivanillyl alcohol as bright yellow crystals. The reaction involves a side-chain elimination, a reaction that has been observed previously during dehydrogenation of lignin model compounds with *p*-hydroxybenzyl alcohol structures (Pew & Connors, 1969).



The geometry and numbering scheme of the molecule are shown in Fig. 1. The aromatic ring with a free phenolic group was found to be at an expected angle of 56.4(1)° to the cyclohexadione ring and a hydrogen-bonding interaction can be observed between the phenolic OH (O5) and the cyclohexadienone carbonyl oxygen O4. The benzene rings in the dioxepin moiety are at an angle of 41.7(1)°, which is considerably less than in normal biphenyl structures (57–59°).

The molecules are held together by hydrogen bonds between the terminal OH groups. The hydrogen-bonding details are given in Table 3.

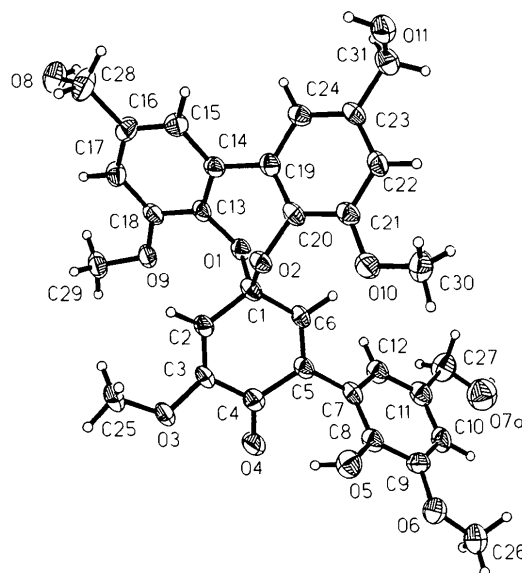


Fig. 1. Molecular geometry and the numbering scheme for the title compound. Displacement ellipsoids are plotted at the 30% probability level. Only one orientation of the disordered hydroxyl group is shown.