

4,5-Diazafluoren-9-ol

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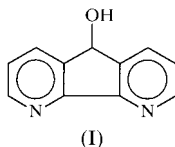
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The title compound, $C_{11}H_8N_2O$, has two crystallographically independent molecules in the crystal. Each molecule is basically planar except for the O atom. The two N atoms in the molecule show different behaviour as hydrogen-bonding acceptors. One of them is involved in intermolecular O—H···N hydrogen bonds which stabilize the crystal packing.

Comment

4,5-Diazafluoren-9-ol, (I), has been synthesized and characterized over the past three decades (Dickeson & Summers, 1970; Gillard & Hill, 1974; Newkome & Roper, 1979). Recently, it has been of interest that some derivatives of (I) are potential drugs for the improvement of cognitive performance in patients with Alzheimer's-type dementia (Wong *et al.*, 1996) and are also potential anticancer agents (Lattmann *et al.*, 1999). Metal modification (metal binding) can have a direct or indirect influence on the activity and metabolism of organic drugs: some metal-organic drug complexes have improved biological activities over the parent organic compounds (Guo & Sadler, 1999a). As part of our research into the design of novel platinum-based anticancer agents and other metal-based drugs (Guo & Sadler, 1999b), and as a continuation of structural investigations of 4,5-diazafluoren-9-one derivatives (Fun *et al.*, 1995; Shanmuga Sundara Raj *et al.*, 1999), we have synthesized compound (I) and report its structure here.



The X-ray analysis of (I) indicated it to have crystallographically independent molecules, namely, molecules 1 and 2, which contain atoms O1 and O2, respectively. Each molecule is basically planar except for the O atom, which has a maximum deviation from the fused rings of 1.073 (1) Å for molecule 1 and 1.092 (1) Å for molecule 2. The dihedral angle between molecules 1 and 2 is 61.0 (3)°.

The bond lengths and angles observed in the structure of (I) are in the normal ranges and are comparable with those found in the structures of other 4,5-diazafluorene derivatives (Lu *et al.*, 1995, 1996). We have reported previously that there is a difference in the behaviour of the two pyridyl N atoms of the diazafluorene moiety, in that one can form strong hydrogen bonds and the other cannot (Fun *et al.*, 1995; Shanmuga Sundara Raj *et al.*, 1999). The same phenomenon is also found in the present structure; atoms N2 of molecule 1 and N3 of molecule 2 are involved in intermolecular O—H···N hydrogen bonds, whereas atoms N1 and N4 are not involved in any hydrogen-bond interaction. The different behaviour of these N atoms with respect to hydrogen bonding may be related to their environments.

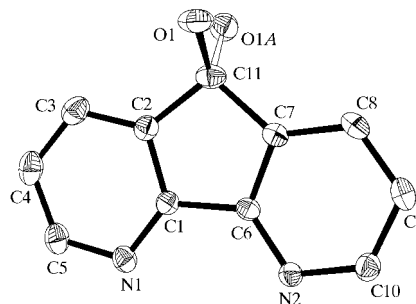


Figure 1

The structure of (I) showing 30% probability displacement ellipsoids and the atom-numbering scheme. H atoms have been omitted for clarity. The hydroxy group is disordered unequally over two sites.

Atom O1 is disordered, with an occupancy of 0.866 (3) for O1 and 0.134 (3) for O1A. Only the hydrogen bond formed by the O1 atom with the highest occupancy factor is considered here. In addition, atom O2 can form a weak intermolecular C—H···O hydrogen bond, whereas O1 cannot form any C—H···O hydrogen bonds. The geometries of these interactions are listed in Table 2.

Experimental

Compound (I) was prepared by the reaction of 4,5-diazafluoren-9-one with an excess of sodium borohydride in 95% ethanol for 30 min. Diffraction quality crystals of (I) were obtained by recrystallization from toluene.

Crystal data

$C_{11}H_8N_2O$
 $M_r = 184.19$
 Monoclinic, $P2_1/n$
 $a = 10.193$ (2) Å
 $b = 14.477$ (3) Å
 $c = 12.092$ (2) Å
 $\beta = 96.29$ (3)°
 $V = 1773.6$ (6) Å³
 $Z = 8$

$D_x = 1.380$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 25 reflections
 $\theta = 2.38$ – 10.24 °
 $\mu = 0.092$ mm⁻¹
 $T = 293$ (2) K
 Block, colourless
 0.28 × 0.24 × 0.22 mm

Data collection

Enraf-Nonius CAD-4 diffractometer
 $2\theta/\omega$ scans
 3295 measured reflections
 3109 independent reflections
 1443 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.033$

$\theta_{max} = 24.97$ °
 $h = 0 \rightarrow 12$
 $k = 0 \rightarrow 17$
 $l = -14 \rightarrow 14$
 3 standard reflections every 97 reflections
 intensity decay: 4.98%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.037$
 $wR(F^2) = 0.085$
 $S = 1.023$
 3109 reflections
 266 parameters

H atoms treated by a mixture of independent and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.025P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.11 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.19 \text{ e } \text{\AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$).

C1—N1	1.334 (2)	C16—N3	1.332 (2)
C5—N1	1.344 (2)	C17—N4	1.346 (3)
C6—N2	1.335 (2)	C21—N4	1.328 (2)
C10—N2	1.345 (2)	C22—O2	1.409 (3)
C11—O1A	1.410 (11)	O1—H1A	0.87 (3)
C11—O1	1.417 (3)	O2—H2A	0.91 (3)
O1A—C11—O1	100.6 (5)	O1—C11—C2	115.3 (2)
O1A—C11—C7	116.4 (5)	O2—C22—C15	114.0 (2)
O1—C11—C7	113.7 (2)	O2—C22—C20	110.9 (2)
O1A—C11—C2	109.7 (5)		

Table 2

Hydrogen-bonding geometry (\AA , $^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1A \cdots N3 ⁱ	0.87 (3)	2.10 (3)	2.936 (3)	160 (2)
O2—H2A \cdots N2 ⁱⁱ	0.91 (3)	1.83 (3)	2.710 (2)	164 (2)
C9—H9A \cdots O2 ⁱⁱⁱ	0.96	2.48	3.292 (3)	142

Symmetry codes: (i) $\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z$; (ii) $\frac{1}{2} - x, y - \frac{1}{2}, \frac{3}{2} - z$; (iii) $x, y, 1 + z$.

H atoms on C atoms were placed in calculated positions with C—H = 0.96 \AA , assigned fixed isotropic displacement parameters and allowed to ride. H atoms on O atoms (except O1A) were located from a difference map and were refined isotropically. Atoms O1 and O1A are disordered and the occupancy factors are 0.866 (3) for O1 and 0.134 (3) for O1A.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *CAD-4 Software*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1481). Services for accessing these data are described at the back of the journal.

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