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4,5-Diazafluoren-9-one benzoylhydrazone monohydrate

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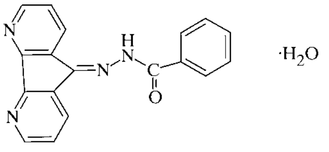
The title compound, $C_{18}H_{12}N_4O{\cdot}H_2O$, adopts the keto tautomeric form and the azomethine C=N double bond is in the *E* configuration. The dihedral angle between the planes of the diazafluorene moiety and the phenyl ring is 11.3 (1)°. In the solid state, the molecules form infinite chain-like structures *via* O—H···N hydrogen bonds involving the water molecules and diazafluorene moieties.

Comment

Aroylhydrazones have continued to attract extensive attention from chemists because of their wide-ranging biological activity and their strong ability to chelate to transition metal ions, lanthanide metal ions and main-group metal ions forming metal complexes (Fun *et al.*, 1999; Lu *et al.*, 1999; Sangeetha *et al.*, 1996; Ainscough *et al.*, 1998). Furthermore, 4,5-diazafluoren-9-one and its derivatives are also strong chelating agents and have been used as ligands to form functional ruthenium complexes (Chao *et al.*, 1999). The combination of the two functional groups yielding ligands capable of forming polymetallic complexes would therefore be of interest for their potential multi-redox, catalytic, electronic and energy-transfer properties. As part of studies on the synthesis and characterization of new aroylhydrazone derivatives, we report here the crystal structure of 4,5-diazafluoren-9-one benzoylhydrazone monohydrate, (I).



(I)

The bond lengths and angles observed in this structure are normal and comparable with those found in the structures of other benzoylhydrazine (Fun *et al.*, 1997) and 4,5-diazafluorene derivatives (Lu *et al.*, 1996). The molecule is in the keto tautomeric form. The dihedral angle between the diazafluorene moiety and the phenyl ring is 11.3 (1)°, and the two fragments make angles of 13.2 (1) and 23.2 (1)°, respectively, with the central hydrazone moiety. In the solid state, the molecules form infinite chain-like structures *via* O—H···N hydrogen bonds involving the water molecules and diazafluorene moieties.

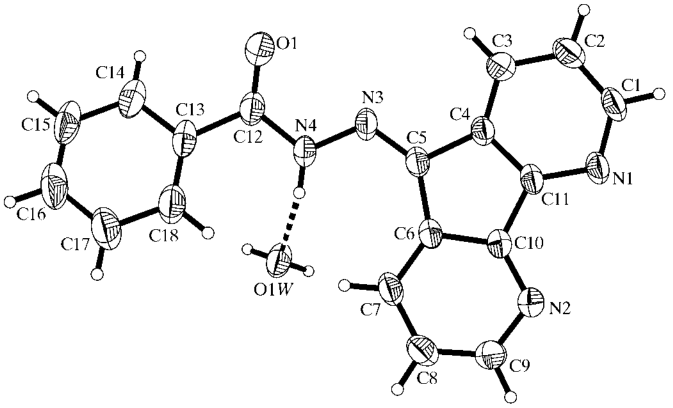


Figure 1
The structure of (I) showing 50% probability displacement ellipsoids and the atom-numbering scheme.

We have observed previously that due to the possibility of different charge concentrations between the two pyridyl N atoms of the diazafluorene moiety, the one with greater charge is involved in the stronger hydrogen bonds, whereas the other may be involved in only a weak interaction (Fun *et al.*, 1995; Lu *et al.*, 1995). However, in this structure, both the N atoms are involved in a similar type of O—H···N hydrogen bond with the water molecule. We also note that the aroylhydrazine derivatives crystallize in hydrated forms only.

Experimental

The synthesis of the title compound was carried out by reaction of 4,5-diazafluoren-9-one and benzoylhydrazine in ethanol solution under reflux for 5 h. Single crystals were obtained by recrystallization from ethanol.

Crystal data

$C_{18}H_{12}N_4O{\cdot}H_2O$
$M_r = 318.33$
Monoclinic, $P2_1/n$
$a = 8.4880$ (2) Å
$b = 22.7523$ (2) Å
$c = 8.7810$ (2) Å
$\beta = 114.929$ (1)°
$V = 1537.80$ (5) Å^3
$Z = 4$

$D_x = 1.375$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3585 reflections
$\theta = 1.79$–28.34°
$\mu = 0.093$ mm^{-1}
$T = 293$ (2) K
Needle, yellow
0.38 × 0.18 × 0.12 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
10723 measured reflections
3729 independent reflections
1870 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.078$
$\theta_{\rm max} = 28.31°$
$h = -9 \rightarrow 11$
$k = -29 \rightarrow 27$
$l = -11 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.061$
$wR(F^2) = 0.121$
$S = 0.930$
3729 reflections
226 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0695P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.29$ e Å^{-3}
$\Delta\rho_{\min} = -0.28$ e Å^{-3}
Extinction correction: *SHELXTL* (Sheldrick, 1997)
Extinction coefficient: 0.014 (2)

Table 1
Selected geometric parameters (Å, °).

N1—C1	1.339 (3)	N3—C5	1.290 (3)
N1—C11	1.340 (3)	N3—N4	1.371 (3)
N2—C10	1.332 (3)	N4—C12	1.378 (3)
N2—C9	1.339 (3)	O1—C12	1.216 (3)
C5—N3—N4	119.4 (2)	O1—C12—C13	121.8 (2)
N3—N4—C12	116.5 (2)	N4—C12—C13	116.1 (2)
O1—C12—N4	122.0 (2)		
C5—N3—N4—C12	170.4 (2)	N3—N4—C12—C13	−177.6 (2)
N3—N4—C12—O1	−0.6 (3)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4A···O1W	0.86	2.21	2.921 (3)	140
O1W—H1W1···N1$^{\rm i}$	0.94 (3)	2.00 (4)	2.886 (3)	157 (3)
O1W—H2W1···N2$^{\rm ii}$	0.89 (5)	2.04 (5)	2.907 (3)	163 (4)

Symmetry codes: (i) $2 - x, 1 - y, -z$; (ii) $x - 1, y, z$.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; 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* and *PLATON* (Spek, 1990).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1396). Services for accessing these data are described at the back of the journal.

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