

Monoclinic

$P2_1/c$
 $a = 8.6086$ (3) Å
 $b = 11.5661$ (4) Å
 $c = 14.7650$ (5) Å
 $\beta = 91.554$ (1)°
 $V = 1469.58$ (9) Å³
 $Z = 4$
 $D_x = 1.246$ Mg m⁻³
 D_m not measured

Cell parameters from 3243 reflections
 $\theta = 1.12$ – 28.35 °
 $\mu = 0.487$ mm⁻¹
 $T = 293$ (2) K
 Block
 $0.28 \times 0.12 \times 0.10$ mm
 Colourless

thanks the Universiti Sains Malaysia for a Visiting Post-doctoral Fellowship.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1290). Services for accessing these data are described at the back of the journal.

Data collection

Siemens SMART CCD area-detector diffractometer
 ω scans
 Absorption correction: empirical (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.876$, $T_{\max} = 0.953$
 11 387 measured reflections
 3622 independent reflections

1757 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.082$
 $\theta_{\text{max}} = 28.32$ °
 $h = -11 \rightarrow 11$
 $k = 0 \rightarrow 15$
 $l = 0 \rightarrow 19$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.062$
 $wR(F^2) = 0.125$
 $S = 1.000$
 3622 reflections
 221 parameters
 All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.042P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.278$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.385$ e Å⁻³
 Extinction correction: none
 Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. Selected geometric parameters (Å, °)

P1—N1	1.629 (2)	P1—C11	2.071 (1)
P1—C1	1.802 (3)	N1—C10	1.492 (4)
P1—S1	1.923 (1)	N1—C7	1.505 (4)
N1—P1—C1	105.1 (1)	N1—P1—C11	106.5 (1)
N1—P1—S1	117.9 (1)	C1—P1—C11	102.5 (1)
C1—P1—S1	113.4 (1)	S1—P1—C11	110.2 (1)

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

C_g is the centroid of the C1—C6 ring.

D—H...A	D—H	H...A	D...A	D—H...A
C2—H2...C11	0.85 (2)	2.70 (3)	3.180 (4)	117 (2)
C11—H11C...C _g 1 ⁱ	0.90 (4)	2.93	3.577	130
C12—H12C...C _g 1 ⁱⁱ	0.93 (4)	3.03	3.897	156

Symmetry codes: (i) $x, \frac{1}{2} - y, z - \frac{1}{2}$; (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: XP in SHELXTL. Software used to prepare material for publication: SHELXTL, PLATON (Spek, 1990) and PARST (Nardelli, 1995).

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9-(4H-1,2,4-Triazol-4-ylimino)-4,5-diazafluorene

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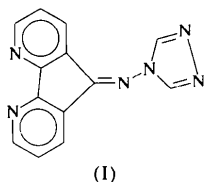
Abstract

The title molecule, C₁₃H₈N₆, is non-planar. The dihedral angle between the planes of the diazafluorene moiety and the triazole ring is 61.0 (1)°. The molecules are arranged as chains running along the *c* axis and along the normal to the *bc* plane through C—H...N hydrogen bonds.

Comment

The aroyl Schiff bases of 4,5-diazafluorene-9-one have received considerable attention over the past few decades

(Wang & Rillema, 1997; Duan *et al.*, 1992). These compounds may function as polypyridyl-bridged ligands (Wang *et al.*, 1998). Schiff base compounds having extensively conjugated π -electron systems have been reported to display second harmonic generation (Houlton *et al.*, 1992), however, the heteroaroyl Schiff bases have rarely been studied. As a continuation of our investigation of the structure of 4,5-diazafluoren-9-one (Fun *et al.*, 1995; Lu *et al.*, 1995, 1996), we have determined the crystal structure of 9-(4*H*-1,2,4-triazol-4-ylimino)-4,5-diazafluorene, (I).



The X-ray analysis of (I) indicated that it is non-planar (Fig. 1). The bond lengths and angles observed in the structure are normal. The dihedral angle between the planes of the diazafluorene moiety and the triazole ring is $61.0(1)^\circ$, whereas that between the diazafluorene moiety and the pyridine ring in 4,5-diaza-9-fluorene isonicotinoyl hydrazone monohydrate (Lu *et al.*, 1996) is $27.74(4)^\circ$. The interplanar angle between rings A and B is $0.7(1)^\circ$, but that between rings B and C is $2.6(1)^\circ$. This increase in the dihedral angle may be due to steric repulsion between C2 and C12 [C2...C12 $3.166(3) \text{ \AA}$].

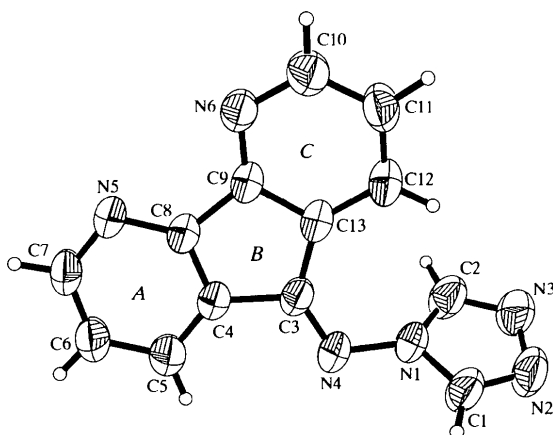


Fig. 1. The structure of title compound showing 50% probability displacement ellipsoids and the atom-numbering scheme.

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 a weak interaction (Fun *et al.*, 1995; Lu *et al.*, 1995). It can be

seen from Fig. 2 that there are C—H...N hydrogen-bonded chains along the normal to the *bc* plane and chains along the *c* axis.

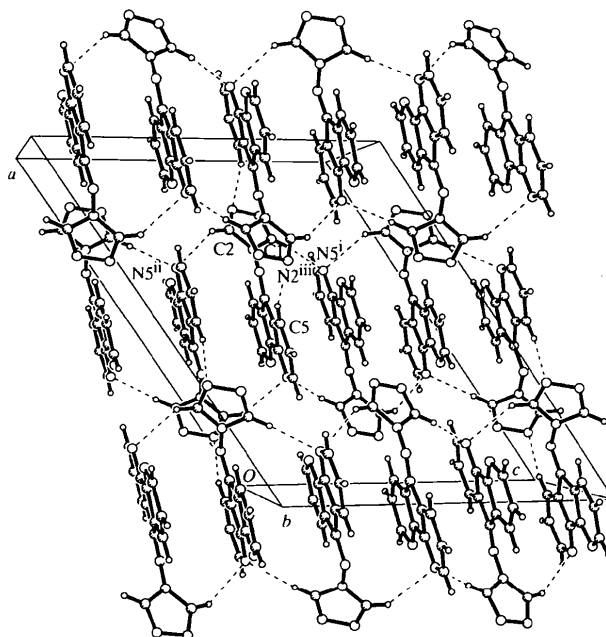


Fig. 2. The packing of molecules of (I) viewed down the *b* axis showing the C—H...N hydrogen bonds

The molecules are stacked along the *c* axis through interactions between ring centroids (*Cg*). The distances between centroids are $Cg1 \cdots Cg1(1-x, y, \frac{1}{2}-z)$ $3.674(4) \text{ \AA}$ and $Cg1 \cdots Cg2(1-x, 1-y, -z)$ $3.512(5) \text{ \AA}$, where *Cg1* and *Cg2* are the centroids of rings B and C, respectively.

Experimental

The title compound was prepared by condensation of 4,5-diazafluoren-9-one and 4-amino-4*H*-1,2,4-triazole in ethanol. X-ray quality crystals were obtained by recrystallization from an *N,N*-dimethylformamide-ether solution at room temperature.

Crystal data

$C_{13}H_8N_6$
 $M_r = 248.25$
 Monoclinic
*C*2/*c*
 $a = 18.304(1) \text{ \AA}$
 $b = 10.488(1) \text{ \AA}$
 $c = 14.267(1) \text{ \AA}$
 $\beta = 123.790(1)^\circ$
 $V = 2276.2(3) \text{ \AA}^3$
 $Z = 8$
 $D_x = 1.449 \text{ Mg m}^{-3}$
 D_m not measured

Mo $K\alpha$ radiation
 $\lambda = 0.71073 \text{ \AA}$
 Cell parameters from 2725 reflections
 $\theta = 2.36\text{--}28.32^\circ$
 $\mu = 0.096 \text{ mm}^{-1}$
 $T = 293(2) \text{ K}$
 Block
 $0.36 \times 0.16 \times 0.14 \text{ mm}$
 Yellow

Data collection

Siemens SMART CCD area-detector diffractometer
 ω scans
 Absorption correction: none
 7790 measured reflections
 2817 independent reflections

1553 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.054$
 $\theta_{\text{max}} = 28.29^\circ$
 $h = -24 \rightarrow 22$
 $k = -13 \rightarrow 12$
 $l = -18 \rightarrow 19$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.063$
 $wR(F^2) = 0.197$
 $S = 1.018$
 2817 reflections
 172 parameters
 H atoms constrained
 $w = 1/[\sigma^2(F_o^2) + (0.1040P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.197 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\text{min}} = -0.228 \text{ e } \text{Å}^{-3}$
 Extinction correction: none
 Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. Selected geometric parameters (Å, °)

N1—C1	1.341 (3)	N4—C3	1.280 (3)
N1—C2	1.353 (3)	N5—C8	1.336 (3)
N1—N4	1.410 (3)	N5—C7	1.337 (3)
N2—C1	1.299 (3)	N6—C9	1.321 (3)
N2—N3	1.381 (3)	N6—C10	1.349 (3)
N3—C2	1.289 (3)		
C1—N1—C2	104.5 (2)	C3—N4—N1	116.0 (2)
C1—N1—N4	124.9 (2)	N4—C3—C13	134.6 (2)
C2—N1—N4	129.5 (2)		

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

D—H...A	D—H	H...A	D...A	D—H...A
C1—H1A...N5 ⁱ	0.93	2.57	3.381 (4)	145
C2—H2A...N5 ⁱⁱ	0.93	2.50	3.361 (4)	153
C5—H5A...N2 ⁱⁱⁱ	0.93	2.62	3.513 (4)	162

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

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, PARST (Nardelli, 1995) and PLATON (Spek, 1990).

This work was supported financially by the State Science and Technology Commission and the National Nature Science Foundation of China. The authors would like to thank the Malaysian Government and Universiti Sains Malaysia for research grant R&D No. 190-9609-2801. SSSR thanks the Universiti Sains Malaysia for a Visiting Postdoctoral Fellowship.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1425). Services for accessing these data are described at the back of the journal.

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8-Méthyl-1,3-diphényl-4,5,6,6a,7,8-hexahydrocyclopenta[*b*][1,2,4]triazolo[4,3-*a*]-benzodiazépin-7-one

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Abstract

The structure of the title compound, C₂₆H₂₄N₄O, shows that the dipolar cycloaddition reactions of nitrilamines with benzodiazepines are peri- and regioselective. The central seven-membered ring is fused with a triazolo ring, a five- and a six-membered ring to form the core of the molecule.

Commentaire

La péri- et la régiosélectivité des réactions de cycloaddition dipolaires-1,3 des nitrilamines avec des 1,5- et 1,4-benzodiazépines ont déjà été mises en évidence à plusieurs reprises par notre équipe (Benelbaghdadi *et al.*, 1998; Essaber, Baouid, Hasnaoui, Benharref & Lavergne, 1998; Baouid *et al.*, 1996; Essaber, Baouid, Hasnaoui, Giorgi & Pierrot, 1998; Chiaroni, Riche,