

Table 3. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²) for (2)

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

	x	y	z	U _{eq}
C(1)	1/2	-0.1899 (5)	1/4	0.095 (4)
C(2)	0.4882 (1)	-0.0968 (3)	0.3497 (3)	0.060 (2)
C(3)	0.4838 (1)	0.0508 (3)	0.2968 (2)	0.042 (1)
C(4)	0.4339 (1)	0.0860 (3)	0.2348 (3)	0.046 (1)
P	0.39587 (2)	0.11679 (7)	0.34781 (7)	0.0428 (3)
C(5)	0.3922 (1)	0.3117 (3)	0.3470 (3)	0.046 (1)
C(6)	0.4394 (1)	0.3787 (3)	0.3894 (4)	0.064 (2)
C(7)	0.4362 (1)	0.5384 (3)	0.3957 (4)	0.074 (2)
C(8)	0.4038 (1)	0.5846 (4)	0.4788 (4)	0.086 (3)
C(9)	0.3566 (2)	0.5216 (4)	0.4356 (7)	0.105 (3)
C(10)	0.3594 (1)	0.3613 (4)	0.4292 (5)	0.085 (3)
C(11)	0.3383 (1)	0.0664 (3)	0.2571 (3)	0.044 (1)
C(12)	0.3247 (1)	0.1266 (3)	0.1265 (3)	0.056 (2)
C(13)	0.2764 (1)	0.0795 (3)	0.0643 (3)	0.061 (2)
C(14)	0.2723 (1)	-0.0782 (3)	0.0612 (3)	0.062 (2)
C(15)	0.2850 (1)	-0.1397 (4)	0.1903 (3)	0.070 (2)
C(16)	0.3334 (1)	-0.0931 (3)	0.2539 (3)	0.059 (2)

Table 4. Selected geometric parameters (Å, °) for (2)

C(1)—C(2)	1.507 (4)	C(2)—C(3)	1.521 (4)
C(3)—C(3 ¹)	1.540 (4)	C(3)—C(4)	1.537 (4)
P—C(4)	1.857 (3)	P—C(5)	1.863 (3)
P—C(11)	1.862 (3)		
C(2)—C(1)—C(2 ¹)	107.8 (2)	C(1)—C(2)—C(3)	106.3 (2)
C(2)—C(3)—C(4)	112.2 (3)	C(2)—C(3)—C(3 ¹)	103.8 (2)
C(4)—C(3)—C(3 ¹)	111.6 (2)	C(3)—C(4)—P	112.9 (1)
C(4)—P—C(5)	101.4 (1)	C(4)—P—C(11)	102.0 (1)
C(5)—P—C(11)	102.0 (1)	P—C(5)—C(6)	111.6 (2)
P—C(5)—C(10)	110.6 (2)	P—C(11)—C(12)	117.1 (2)
P—C(11)—C(16)	110.1 (2)		

Symmetry code: (i) 1 - x, y, ½ - z.

The positional parameters for all non-H atoms were determined by direct methods (Sheldrick, 1985) for both compounds. The refinements were carried out by full-matrix least-squares techniques (Imoto, 1990). The $\Delta f'$ and $\Delta f''$ components of anomalous dispersion were included in the calculation for P and S atoms of compound (1) and for all non-H atoms of compound (2) (Cromer, 1974).

The chosen model for compound (1) has residuals of $R = 0.047$ and $wR = 0.048$, while the other enantiomeric structure gave R and wR values of 0.056 and 0.060, respectively. When the R -factor ratio test (Hamilton, 1965) is applied to wR , the latter enantiomeric model can be rejected.

F_o data were collected at the Faculty of Pharmaceutical Sciences, Osaka University. All calculations were carried out on an NEC ACOS S3700 computer at the Research Center for Protein Engineering, Institute for Protein Research, Osaka University.

This work was partially supported by a Grant-in-Aid for Scientific Research from the Ministry of Education, Science and Culture, Japan.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AS1173). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1995). **C51**, 2076–2078

4,5-Diazafluoren-9-one

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(Received 3 January 1995; accepted 15 March 1995)

Abstract

The 4,5-diazafluoren-9-one molecules, C₁₁H₆N₂O, are planar and lie in parallel planes. The presence of a C—H···N intermolecular hydrogen bond is observed in this structure.

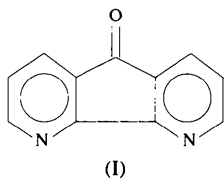
Comment

Extensive studies have been carried out on the synthesis and uses of the title compound, (I) (Eckhard &

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Summers, 1973; Henderson, Fronczek & Cherry, 1984; Grigg, Mongkolaussavaratana, Pounds & Sivagnanam, 1990; Baxter, Connor, Wallis, Povey & Powell, 1992). In general, this compound is synthesized by the oxidation of 1,10-phenanthroline by potassium permanganate in alkaline medium.



An ORTEP (Johnson, 1976) diagram with numbering scheme is shown in Fig. 1. The bond lengths and angles in the structure are normal. The molecule is planar as a whole with a maximum deviation of 0.021 (1) Å for C9. The molecules lie on the (104) plane in an alternating fashion so as to form two sets of parallel layers, each set separated by a normal distance of 3.410 (1) Å. In each layer the molecules are linked by C2—H···N5 intermolecular hydrogen bonds running parallel to the *b* axis. The details of the hydrogen bond are: C2—H2 0.98 (2), C2···N5ⁱ 3.365 (1), H2···N5ⁱ 2.39 (2) Å and C2—H2···N5ⁱ, 174 (1)° [symmetry code: (i) $-x + 1, y - \frac{1}{2}, -z + \frac{1}{2}$]. It is worth mentioning that the formation of a hydrogen bond with an N atom is quite peculiar, particularly when a more electronegative carbonyl O atom (O14) is available in the structure. The structure of the sodium salt of 1*H*-cyclopenta[2,1-*b*:3,4-*b'*]dipyridine-2,5-dione (Baxter *et al.*, 1992) is the only crystal structure found in the literature related to the title compound. The above-mentioned dione compound has a carbonyl substituent at the C4 position and it was concluded in the study of the compound that O14 has less negative charge due to the electron delocalization in the pyridine ring. The same argument may hold here to explain the hydrogen-bond formation with N5 and not with O14.

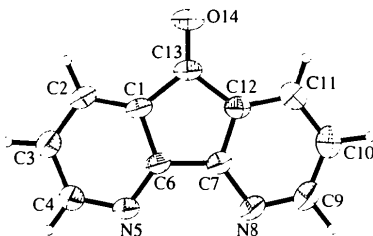


Fig. 1. Structure of the title compound showing 50% probability displacement ellipsoids and atomic numbering scheme.

Experimental

In the process of recrystallizing the complex $\text{CoL}_2(\text{NCS})_2$ (where *L* is the title compound) in ethanol, we unexpectedly obtained single crystals of *L* as yellow prisms. The preparation

of *L* was accomplished by established procedures (Eckhard & Summers, 1973)

Crystal data

$\text{C}_{11}\text{H}_6\text{N}_2\text{O}$
 $M_r = 182.18$
 Monoclinic
 $P2_1/c$
 $a = 10.008 (1) \text{ \AA}$
 $b = 12.409 (1) \text{ \AA}$
 $c = 6.821 (1) \text{ \AA}$
 $\beta = 99.50 (1)^\circ$
 $V = 835.48 (11) \text{ \AA}^3$
 $Z = 4$
 $D_x = 1.448 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation
 $\lambda = 0.71073 \text{ \AA}$
 Cell parameters from 25 reflections
 $\theta = 8-25^\circ$
 $\mu = 0.097 \text{ mm}^{-1}$
 $T = 293 (2) \text{ K}$
 Prism
 $0.58 \times 0.46 \times 0.40 \text{ mm}$
 Yellow

Data collection

Siemens *P4* four-circle diffractometer
 $\theta/2\theta$ scans
 Absorption correction: none
 3088 measured reflections
 2316 independent reflections
 1881 observed reflections
 $[I > 2\sigma(I)]$

$R_{\text{int}} = 0.0131$
 $\theta_{\text{max}} = 30^\circ$
 $h = -14 \rightarrow 14$
 $k = -17 \rightarrow 1$
 $l = -1 \rightarrow 8$
 3 standard reflections monitored every 100 reflections
 intensity decay: < 3%

Refinement

Refinement on F^2
 $R(F) = 0.0439$
 $wR(F^2) = 0.1277$
 $S = 1.140$
 2316 reflections
 152 parameters
 All H-atom parameters refined
 $w = 1/[\sigma^2(F_o^2) + (0.0909P)^2 + 0.0076P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$

$\Delta\rho_{\text{max}} = 0.239 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.263 \text{ e \AA}^{-3}$
 Extinction correction: SHELXL93 (Sheldrick, 1993)
 Extinction coefficient: 0.0713 (90)
 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 (\AA^2)

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

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
C1	0.38315 (10)	0.11559 (7)	0.21906 (15)	0.0383 (2)
C2	0.50259 (11)	0.05793 (8)	0.2460 (2)	0.0470 (3)
C3	0.62154 (11)	0.11818 (9)	0.2825 (2)	0.0517 (3)
C4	0.61498 (11)	0.22994 (9)	0.2907 (2)	0.0487 (3)
N5	0.49896 (8)	0.28680 (7)	0.26484 (14)	0.0418 (2)
C6	0.38705 (9)	0.22819 (7)	0.22966 (13)	0.0346 (2)
C7	0.24602 (9)	0.26975 (7)	0.19569 (13)	0.0344 (2)
N8	0.20891 (9)	0.37276 (6)	0.19653 (13)	0.0423 (2)
C9	0.07372 (12)	0.38811 (9)	0.1645 (2)	0.0480 (3)
C10	-0.02192 (12)	0.30710 (10)	0.1286 (2)	0.0509 (3)
C11	0.01945 (11)	0.20016 (10)	0.1265 (2)	0.0473 (3)
C12	0.15737 (10)	0.18216 (7)	0.1635 (2)	0.0379 (2)
C13	0.23863 (11)	0.08050 (8)	0.1769 (2)	0.0420 (2)
O14	0.19506 (9)	-0.01077 (6)	0.1582 (2)	0.0635 (3)

Table 2. Selected geometric parameters (\AA , $^\circ$)

C1—C2	1.379 (1)	C7—N8	1.331 (1)
C1—C6	1.399 (1)	C7—C12	1.397 (1)
C1—C13	1.492 (1)	N8—C9	1.348 (2)

C2—C3	1.393 (2)	C9—C10	1.382 (2)
C3—C4	1.390 (2)	C10—C11	1.391 (2)
C4—N5	1.345 (1)	C11—C12	1.380 (2)
N5—C6	1.324 (1)	C12—C13	1.496 (1)
C6—C7	1.485 (1)	C13—O14	1.213 (1)
C2—C1—C6	119.67 (9)	C12—C7—C6	108.48 (8)
C2—C1—C13	131.72 (9)	C7—N8—C9	114.09 (9)
C6—C1—C13	108.61 (8)	N8—C9—C10	124.98 (10)
C1—C2—C3	116.21 (10)	C9—C10—C11	119.82 (10)
C4—C3—C2	119.88 (10)	C12—C11—C10	116.30 (10)
N5—C4—C3	124.30 (10)	C11—C12—C7	119.53 (9)
C6—N5—C4	114.95 (9)	C11—C12—C13	131.70 (9)
N5—C6—C1	124.99 (9)	C7—C12—C13	108.77 (8)
N5—C6—C7	126.27 (8)	O14—C13—C1	127.81 (10)
C1—C6—C7	108.73 (8)	O14—C13—C12	126.79 (10)
N8—C7—C12	125.25 (9)	C1—C13—C12	105.40 (7)
N8—C7—C6	126.27 (8)		

All the H atoms were located from difference Fourier maps and refined isotropically.

Data collection: XSCANS (Siemens, 1994). Cell refinement: XSCANS. Data reduction: XSCANS. Program(s) used to solve structure: SHELXS86 (Sheldrick, 1985). Program(s) used to refine structure: SHELXL93 (Sheldrick, 1993). Molecular graphics: ORTEPII (Johnson, 1976), SHELXTL/PC (Sheldrick, 1990). Software used to prepare material for publication: SHELXL93. Geometric calculations: PARST (Nardelli, 1983).

The authors would like to thank the Malaysian Government and the Universiti Sains Malaysia for research grant R&D No. 123-3417-2201, and the State Science and Technology Commission, National Nature Science Foundation of China, for a grant for a Key Research Project. One of the authors (KS) thanks the Universiti Sains Malaysia for a Visiting Postdoctoral Research Fellowship.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AS1169). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1995). **C51**, 2078–2080

p-Dimethylaminobenzaldehyde 4,5-Diaza-9-fluorenylidenehydrazone Monohydrate

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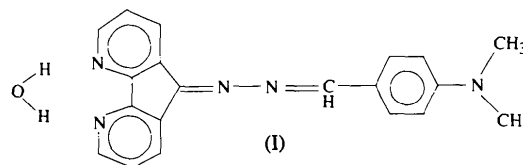
(Received 2 February 1995; accepted 27 March 1995)

Abstract

The X-ray analysis reveals that the title compound, C₂₀H₁₇N₅.H₂O, is an electron-delocalized planar molecule. The molecules are stacked as non-bonding dimeric pairs about the crystallographic inversion centre in the crystal lattice. The lone water molecule present in the unit cell is hydrogen bonded to both N atoms in the diazafluorene moiety.

Comment

Recently, nonlinear optical organic materials have aroused considerable interest as a result of their wide application in science and technology (Chemla & Zyss, 1987). Schiff base compounds having extensively conjugated π -electron systems have been reported to display second harmonic generations (Houlton *et al.*, 1992; Velavan, Sivakumar & Anbu, 1995). The title compound, (C₁₀H₆N₂)C=N—N=CH(C₆H₄)N(CH₃)₂.H₂O, (I), is a Schiff base derivative; this crystal structure determination forms part of our search for and study of new materials for nonlinear optical applications.



An ORTEPII (Johnson, 1976) plot of the molecule with atom-numbering scheme is shown in Fig. 1. The bond lengths and angles observed in this structure show

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