

Table 1. Atomic coordinates and equivalent isotropic thermal parameters

$$B_{eq} = (4/3) \sum_i \sum_j \beta_{ij} a_i \cdot a_j$$

	x	y	z	$B_{eq}(\text{Å}^2)$
S(1)	0.6270 (1)	0.0148 (1)	0.935*	3.55 (1)
C(2)	0.6539 (2)	0.0933 (2)	1.1599 (4)	3.63 (4)
N(3)	0.7563 (1)	0.1000 (2)	1.1882 (3)	3.47 (3)
C(4)	0.8191 (2)	0.0498 (2)	1.0459 (4)	3.11 (4)
C(5)	0.7567 (2)	-0.0042 (1)	0.8845 (3)	2.94 (4)
C(6)	0.8018 (2)	-0.0585 (2)	0.7279 (4)	3.04 (4)
C(7)	0.7571 (1)	-0.1175 (2)	0.5565 (4)	2.94 (4)
C(8)	0.8227 (1)	-0.1601 (2)	0.4060 (3)	3.11 (4)
C(9)	0.7862 (2)	-0.2171 (2)	0.2359 (3)	3.18 (4)
C(10)	0.6829 (2)	-0.2339 (2)	0.2152 (3)	2.83 (3)
C(11)	0.6158 (1)	-0.1933 (2)	0.3640 (4)	3.31 (4)
C(12)	0.6531 (2)	-0.1366 (2)	0.5327 (4)	3.36 (4)
C(13)	0.5471 (2)	-0.3155 (2)	0.0225 (4)	4.61 (5)
O(14)	0.6526 (1)	-0.2899 (1)	0.0432 (2)	3.53 (3)
O(15)	0.5901 (1)	0.1330 (2)	1.2687 (3)	5.34 (4)
O(16)	0.9112 (1)	0.0512 (1)	1.0545 (3)	4.18 (3)

* Origin-defining.

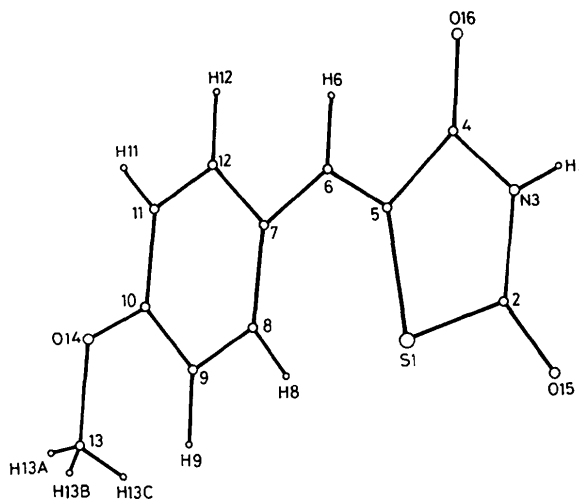


Fig. 1. The structure of the molecule with the atom-numbering scheme (C atoms where no label appears).

Table 2. Bond distances (Å) and bond angles (°)

S(1)—C(5)	1.752 (2)	C(5)—C(6)	1.348 (3)
S(1)—C(2)	1.779 (2)	C(6)—C(7)	1.446 (4)
O(14)—C(10)	1.365 (3)	C(7)—C(8)	1.400 (3)
O(14)—C(13)	1.429 (3)	C(7)—C(12)	1.397 (3)
O(15)—C(2)	1.198 (3)	C(8)—C(9)	1.387 (3)
O(16)—C(4)	1.213 (3)	C(9)—C(10)	1.380 (3)
N(3)—C(2)	1.363 (3)	C(10)—C(11)	1.396 (3)
N(3)—C(4)	1.380 (3)	C(11)—C(12)	1.382 (3)
C(4)—C(5)	1.483 (3)		
C(2)—S(1)—C(5)	91.7 (2)	C(5)—C(6)—C(7)	130.0 (2)
C(2)—N(3)—C(4)	118.4 (2)	C(6)—C(7)—C(8)	117.8 (2)
C(10)—O(14)—C(13)	118.0 (2)	C(6)—C(7)—C(12)	124.4 (2)
S(1)—C(2)—O(15)	124.0 (2)	C(8)—C(7)—C(12)	117.7 (2)
S(1)—C(2)—N(3)	109.9 (2)	C(7)—C(8)—C(9)	121.6 (2)
O(15)—C(2)—N(3)	126.2 (2)	C(8)—C(9)—C(10)	119.6 (2)
O(16)—C(4)—N(3)	124.1 (3)	C(9)—C(10)—C(11)	120.2 (2)
O(16)—C(4)—C(5)	126.3 (3)	C(9)—C(10)—C(14)	116.2 (2)
N(3)—C(4)—C(5)	109.6 (2)	C(11)—C(10)—C(14)	123.7 (2)
S(1)—C(5)—C(4)	110.5 (1)	C(10)—C(11)—C(12)	119.8 (2)
S(1)—C(5)—C(6)	129.3 (2)	C(7)—C(12)—C(11)	121.2 (2)
C(4)—C(5)—C(6)	120.2 (2)		

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Structure of 2,3-Di(2-pyridyl)-6,7-dimethylquinoxaline

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Abstract. $C_{20}H_{16}N_4$, $M_r = 312.37$, monoclinic, $C2/c$, $a = 16.474$ (1), $b = 13.132$ (1), $c = 7.638$ (1) Å, $\beta = 99.24$ (1)°, $V = 1630.9$ (3) Å³, $Z = 4$, $D_x = 1.272$ g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1.54178$ Å, $\mu = 5.78$ cm⁻¹, $F(000) = 656$, $T = 291$ K, $R = 0.0450$ for 1021 unique reflections. The structure consists of molecules oriented about the twofold axes. The angle

between the least-squares planes for the quinoxaline part of the molecule and the pyridyl fragment is 39.65 (5)°.

Experimental. Crystals of the title compound were grown from acetonitrile. A Syntex $P2_1$ diffractometer was used with graphite-monochromatized $\text{Cu } K\alpha$

Table 1. Final fractional coordinates ($\times 10^4$) and equivalent isotropic temperature coefficients ($\text{\AA}^2 \times 10^4$) for non-H atoms

	$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$			
	x	y	z	U_{eq}
N(1)	4173 (1)	3731 (1)	1691 (2)	476 (5)
N(2)	4456 (1)	1170 (1)	815 (2)	485 (6)
C(1)	4576 (1)	2870 (1)	2078 (2)	433 (5)
C(2)	4589 (1)	4625 (1)	2096 (2)	456 (6)
C(3)	4184 (1)	5566 (1)	1674 (2)	499 (7)
C(4)	4582 (1)	6475 (1)	2078 (2)	506 (6)
C(5)	4083 (1)	1935 (1)	1538 (2)	445 (6)
C(6)	3994 (1)	359 (1)	258 (2)	577 (7)
C(7)	3179 (1)	267 (1)	385 (2)	620 (7)
C(8)	2791 (1)	1062 (1)	1108 (2)	603 (7)
C(9)	3255 (1)	1910 (1)	1697 (2)	515 (6)
C(10)	4128 (1)	7461 (1)	1625 (2)	631 (7)

radiation. The crystal size was $0.25 \times 0.30 \times 0.35$ mm. The unit-cell parameters were obtained by a least-squares fit of the setting angles of 16 reflections in the θ range $15 < 2\theta < 28^\circ$. The intensities of 1106 reflections were measured ($\sin\theta/\lambda < 0.547 \text{ \AA}^{-1}$, $0 \leq h \leq 18$, $0 \leq k \leq 14$, $-8 \leq l < 8$, θ - 2θ scan mode). No significant variation ($< 3\%$) was found in the intensities of the intensity control reflections, 100 and $2\bar{2}0$. The data were corrected for Lorentz and polarization effects but no absorption correction was applied. 1021 reflections with $|F| \geq 3\sigma(F)$ were used in the calculations, $R_{int} = 0.020$. The structure was solved with multiresolution direct methods (Sheldrick, 1990) and refined using full-matrix least-squares methods (Sheldrick, 1976), minimizing $\sum w(|F_o| - |F_c|)^2$, $w = \{1/[\sigma^2(F) + 0.00448F^2]\}$. Heavy atoms were refined with anisotropic and H atoms with isotropic temperature factors; 142 parameters were varied. The refinement converged to $R = 0.045$, $wR = 0.062$, $(\Delta/\sigma)_{max} = 0.002$, $(\Delta/\sigma)_{mean} = 0.001$, $\Delta\rho_{max} = +0.12$, $\Delta\rho_{min} = -0.14 \text{ e \AA}^{-3}$. Fractional coordinates and equivalent isotropic temperature coefficients for non-H atoms are given in Table 1.* The molecular geometry is given in Table 2 with atom labelling in Fig. 1. The figure has been drawn with ORTEP (Johnson, 1965). The atomic scattering factors were those given in the least-squares program SHELX76 (Sheldrick, 1976).

Related literature. Crystal structures containing 2,3-disubstituted derivatives of quinoxaline have aroused considerable interest because of the repulsion between the neighbouring substituents (Visser, Vos, de Groot & Wynberg, 1968; Visser & Vos, 1971;

* Lists of the atomic parameters for H atoms, anisotropic thermal parameters for non-H atoms and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53945 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Bond lengths (\AA), valence angles ($^\circ$), selected non-bonded interatomic distances (\AA) and torsion angles ($^\circ$)

C(1)—N(1)	1.320 (2)	C(7)—C(6)	1.367 (2)
C(2)—N(1)	1.370 (2)	C(8)—C(7)	1.384 (2)
C(5)—N(2)	1.342 (2)	C(9)—C(8)	1.384 (2)
C(6)—N(2)	1.338 (2)	C(1)—C(1')	1.442 (2)
C(5)—C(1)	1.493 (2)	C(2)—C(2')	1.395 (2)
C(3)—C(2)	1.417 (2)	C(4)—C(4')	1.424 (2)
C(4)—C(3)	1.373 (2)	C(5)—C(5')	3.140 (2)
C(10)—C(4)	1.507 (2)	N(2)—N(2')	2.891 (2)
C(9)—C(5)	1.389 (2)		
N(1)—C(1)—C(5)	114.2 (1)	C(5)—C(9)—C(8)	119.3 (1)
C(1)—N(1)—C(2)	117.9 (1)	C(6)—C(7)—C(8)	118.9 (1)
N(1)—C(2)—C(3)	119.7 (1)	C(7)—C(8)—C(9)	118.0 (2)
N(2)—C(5)—C(1)	117.7 (1)	C(5)—C(1)—C(1')	124.6 (1)
N(2)—C(5)—C(9)	122.6 (1)	N(1)—C(1)—C(1')	121.1 (1)
C(5)—N(2)—C(6)	116.9 (2)	N(1)—C(2)—C(2')	121.0 (1)
N(2)—C(6)—C(7)	124.3 (1)	C(3)—C(2)—C(2')	119.3 (1)
C(1)—C(5)—C(9)	119.6 (1)	C(3)—C(4)—C(4')	119.6 (1)
C(2)—C(3)—C(4)	121.1 (1)	C(10)—C(4)—C(4')	120.8 (1)
C(3)—C(4)—C(10)	119.6 (1)		
C(9)—C(5)—C(1)—N(1)	-37.6 (2)	C(9)—C(5)—C(1)—C(1')	143.5 (2)
N(2)—C(5)—C(1)—N(1)	138.5 (1)	N(2)—C(5)—C(1)—C(1')	-40.4 (2)

(i) Atom generated by symmetry operation: $1 - x, y, \frac{1}{2} - z$.

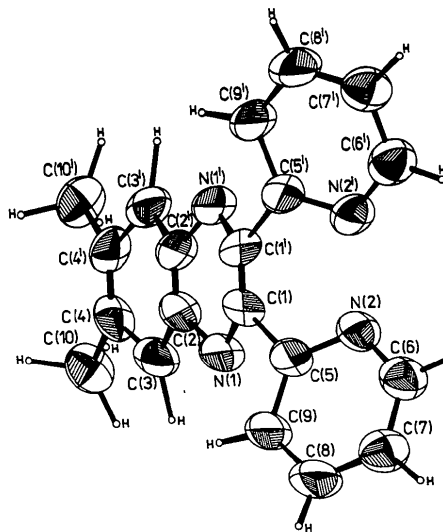


Fig. 1. ORTEP drawing and numbering scheme.

Lipkowski, Herbich & Andreetti, 1985; Krieger, Kocak & Bekaroglu, 1985; Woźniak, Krygowski, Kariuki & Jones, 1990). The N(2)—N(2') distance is equal to 2.891 (2) \AA and it is shorter than two van der Waals radii for N atoms, but the repulsion between the two pyridyl groups still causes significant deformations of valence angles at C(1) and C(1').

References

- JOHNSON, C. K. (1965). ORTEP. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

- KRIEGER, C., KOCAK, A. & BEKAROGLU, O. (1985). *Helv. Chim. Acta*, **68**, 581–583.
- LIPKOWSKI, J., HERBICH, J. & ANDRETTI, G. D. (1985). 9th Eur. Crystallogr. Meet., Turin, Italy. Abstracts, pp. 663–664.
- SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
- SHELDRIK, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
- VISSER, G. J. & VOS, A. (1971). *Acta Cryst.* **B27**, 1793–1801.
- VISSER, G. J., VOS, A., DE GROOT, A. & WYNBERG, H. (1968). *J. Am. Chem. Soc.* **90**, 3253–3254.
- WOŹNIAK, K., KRYGOWSKI, T., KARIUKI, B. & JONES, W. (1990). *Acta Cryst.* **C46**, 1946–1947.

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Structure of a Pyrimidoazepine Derivative

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Abstract. Ethyl 1-oxoperhydropyrrolo[1',2':3,2;2',3'-b]pyrimido[1,2-a]azepin-2-ylideneacetate, $C_{15}H_{20}N_2O_3$, $M_r = 276.3$, monoclinic, $P2_1/c$, $a = 20.472$ (3), $b = 8.869$ (1), $c = 7.806$ (2) Å, $\beta = 99.27$ (1)°, $V = 1398.8$ (5) Å³, $Z = 4$, $D_x = 1.312$ Mg m⁻³, $\lambda(\text{Cu } K\alpha_1) = 1.5405$ Å, $\mu = 0.759$ mm⁻¹, $F(000) = 592$, $T = 293$ K, final $R = 0.056$ for 1533 observed reflections. The seven- and six-membered ring has half-boat puckering with a folded *cis* conformation. The puckering parameter, the angle between the base plane C(2)—C(1)—C(7)—N(6) and C(2)—C(3)—C(4)—C(5), is 56.9 (5)°, and between the base plane C(8)—N(6)—C(7)—N(11)—C(10) and C(10)—C(9)—C(8) is 51.3 (5)°.

Experimental. A reddish needle, 0.15 × 0.05 × 0.40 mm, by recrystallization from CH₂Cl₂. Rigaku

AFC-5 four-circle diffractometer used with the ω -2 θ scan method, ω -scan width (1.3 + 0.41tan θ)° and scan speed 16° min⁻¹. Lattice parameters obtained from least-squares analysis of 20 reflections with 2 θ values ranging from 56 to 61°. Of 2467 reflections scanned [within the index range $h - 22 \rightarrow 22$, $k 0 \rightarrow 9$, $l 0 \rightarrow 8$ up to $\sin\theta/\lambda \leq 0.56$ Å⁻¹ including 182 equivalent reflections ($R_{\text{int}} = 0.03$)], 2076 unique reflections [$F > \sigma(F)$] classified as observed. Three standard reflections measured every 150 reflections, intensity variation < 3%. Intensities corrected for Lorentz and polarization factors, but absorption correction not applied. Structure solved using *SAP85* (Yao, Zheng, Qian, Han, Gu & Fan, 1985), a version of *MULTAN80* (Main, Fiske, Hull, Lessinger,

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters

	$B_{\text{eq}} = (1/3)\sum_i \sum_j B_{ij} a_i^* a_j^* a_i \cdot a_j$			
	x	y	z	$B_{\text{eq}}(\text{Å}^2)$
C(1)	0.1507 (1)	0.2031 (3)	0.7572 (4)	3.46 (10)
C(2)	0.0802 (1)	0.1746 (5)	0.6770 (6)	4.45 (12)
C(3)	0.0484 (2)	0.0499 (5)	0.7708 (8)	5.88 (17)
C(4)	0.0725 (2)	-0.1096 (5)	0.7399 (8)	6.17 (17)
C(5)	0.1389 (2)	-0.1229 (5)	0.6829 (6)	5.19 (14)
N(6)	0.1939 (1)	-0.0540 (2)	0.8006 (3)	3.72 (9)
C(7)	0.1982 (1)	0.0955 (3)	0.8157 (4)	3.24 (9)
C(8)	0.2484 (2)	-0.1500 (4)	0.8792 (5)	4.74 (12)
C(9)	0.2839 (2)	-0.0772 (4)	1.0434 (5)	4.63 (12)
C(10)	0.3099 (1)	0.0754 (4)	1.0036 (5)	3.81 (11)
N(11)	0.2570 (1)	0.1605 (2)	0.8992 (3)	3.24 (7)
C(12)	0.2480 (1)	0.3141 (3)	0.9000 (4)	3.12 (9)
C(13)	0.1781 (1)	0.3439 (3)	0.8085 (4)	3.49 (10)
O(14)	0.1546 (1)	0.4738 (2)	0.7871 (3)	4.62 (8)
C(15)	0.2891 (1)	0.4259 (4)	0.9631 (4)	3.53 (10)
C(16)	0.3591 (1)	0.4231 (4)	1.0239 (4)	4.01 (11)
O(17)	0.3989 (1)	0.3336 (3)	0.9870 (4)	6.04 (10)
O(18)	0.3776 (1)	0.5435 (3)	1.1267 (3)	5.32 (8)
C(19)	0.4474 (2)	0.5544 (8)	1.1990 (8)	7.30 (20)
C(20)	0.4547 (4)	0.6724 (13)	1.3294 (13)	8.30 (25)

Table 2. Bond lengths (Å) and angles (°)

C(1)—C(2)	1.500 (5)	C(1)—C(7)	1.386 (5)
C(1)—C(13)	1.401 (5)	C(2)—C(3)	1.529 (7)
C(3)—C(4)	1.530 (7)	C(4)—C(5)	1.502 (8)
C(5)—N(6)	1.466 (5)	N(6)—C(7)	1.333 (4)
N(6)—C(8)	1.459 (5)	C(8)—C(9)	1.512 (6)
C(9)—C(10)	1.505 (6)	C(10)—N(11)	1.458 (5)
N(11)—C(7)	1.397 (4)	N(11)—C(12)	1.375 (4)
C(12)—C(13)	1.516 (5)	C(12)—C(15)	1.341 (5)
C(13)—O(14)	1.250 (4)	C(15)—C(16)	1.436 (5)
C(16)—O(17)	1.204 (5)	C(16)—O(18)	1.352 (5)
O(18)—C(19)	1.453 (6)	C(19)—C(20)	1.450 (13)
C(2)—C(1)—C(7)	126.7 (3)	C(2)—C(1)—C(13)	125.6 (3)
C(7)—C(1)—C(13)	107.0 (3)	C(1)—C(2)—C(3)	112.1 (3)
C(2)—C(3)—C(4)	114.8 (5)	C(3)—C(4)—C(5)	116.7 (4)
C(4)—C(5)—C(6)	114.9 (4)	C(5)—N(6)—C(7)	120.1 (3)
C(7)—N(6)—C(8)	118.6 (3)	C(7)—N(6)—C(8)	120.5 (2)
C(5)—N(6)—C(7)	128.5 (3)	C(1)—C(7)—N(11)	112.0 (2)
C(1)—C(7)—N(11)	119.4 (2)	N(6)—C(8)—C(9)	109.7 (3)
N(6)—C(7)—N(11)	110.2 (3)	C(9)—C(10)—N(11)	108.9 (3)
C(8)—C(9)—C(10)	123.6 (2)	C(10)—N(11)—C(12)	126.7 (2)
C(10)—N(11)—C(7)	107.7 (2)	N(11)—C(12)—C(13)	106.6 (2)
C(7)—N(11)—C(12)	107.7 (2)	C(13)—C(12)—C(15)	122.1 (3)
N(11)—C(12)—C(15)	131.1 (3)	C(12)—C(13)—O(14)	122.5 (3)
C(12)—C(13)—C(1)	106.4 (2)	C(12)—C(15)—C(16)	129.9 (3)
C(1)—C(13)—O(14)	130.9 (3)	C(15)—C(16)—O(18)	110.5 (3)
C(15)—C(16)—O(17)	127.5 (3)	C(16)—O(18)—C(19)	116.4 (4)
O(17)—C(16)—O(18)	121.8 (3)		
O(18)—C(19)—C(20)	107.7 (5)		