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Structure of 2-(5-Methylthien-2-yl)-2,5-dihydro-3*H*-pyrazolo[4,3-*c*]quinolin-3-one

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Abstract. $C_{15}H_{11}N_3OS$, $M_r = 281{\cdot}33$, monoclinic, $P2_1/c$, $a = 13{\cdot}550\ (4)$, $b = 8{\cdot}723\ (2)$, $c = 14{\cdot}052\ (2)$ Å, $\beta = 130{\cdot}88\ (1)°$, $V = 1255{\cdot}8\ (6)$ Å^3, $Z = 4$, $D_x = 1{\cdot}488$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 2{\cdot}22$ mm^{-1}, $F(000) = 584$, $T = 295$ K, $R = 0{\cdot}076$ for 1474 reflections. The molecule is almost planar. The intermolecular hydrogen bond NH···O, 2·750 (8) Å, is formed between N at position 5 of one pyrazoloquinoline skeleton and C=O at position 3 of another related by a twofold screw axis.

Experimental. Needle-like yellow crystals were obtained from ethanol. Crystal dimensions 0·7 × 0·1 × 0·02 mm. Rigaku AFC-5 diffractometer, graphite-monochromatized Cu $K\alpha$. Unit-cell parameters were refined by 2θ angles for 20 reflections in the range $30 < 2\theta < 40°$. Intensities were measured up to $2\theta = 130°$ in h 0/15, k −10/0 and l −16/12, $R_{int} = 0{\cdot}035$, ω–2θ scans, ω-scan width $(1{\cdot}0 + 0{\cdot}2\tan\theta)°$, three standard reflections monitored every 100 measurements showed no significant change. 2130 unique reflections were measured, 1480 intensities were observed $[F_o > 3\sigma(F_o)]$. Structure solved by *MULTAN*84 (Main, Germain & Woolfson, 1984) and refined by block-diagonal least squares to minimize $\sum(w|\Delta F|^2)$. Absorption corrections by an empirical method (Walker & Stuart, 1983) applied after isotropic refinement (max. and min. transmission factors 1·44 and 0·75). H atoms located on a difference density map. Positional parameters for all atoms and anisotropic thermal parameters for non-H atoms refined. Temperature factor of each H atom set equal to B_{eq} of the bonded atom. $w = [\sigma^2(F_o) + 0{\cdot}00440|F_o|^2]^{-1}$, $w = 0$ for four reflections with $w^{1/2}|\Delta F| \geq 4$ and two very strong ones. Final $R = 0{\cdot}076$, $wR = 0{\cdot}100$ and $S = 1{\cdot}137$. The relatively large R value is assumed to be due to the poor quality of intensity data collected using a very small crystal. Highest peak in the final difference map 0·3 e Å^{-3}. Max. Δ/σ in the final cycle 0·08. Atomic scattering

Table 1. *Atomic coordinates* (× 10^4) *and equivalent isotropic temperature factors* (Å^2 × 10) *with e.s.d.'s in parentheses*

	x	*y*	*z*	B_{eq}*
N(1)	5685 (4)	6725 (5)	803 (4)	27 (2)
N(2)	6644 (4)	5991 (5)	852 (4)	24 (2)
C(3)	6551 (5)	6273 (7)	−171 (5)	27 (2)
C(4)	4977 (5)	8085 (7)	−1988 (5)	27 (2)
N(5)	3991 (4)	9056 (6)	−2457 (4)	29 (2)
C(6)	2423 (5)	10356 (7)	−2474 (5)	29 (2)
C(7)	1878 (5)	10626 (7)	−1932 (5)	32 (3)
C(8)	2344 (6)	9894 (7)	−841 (6)	36 (3)
C(9)	3355 (5)	8860 (7)	−264 (5)	29 (3)
C(10)	3930 (5)	8548 (6)	−791 (4)	24 (2)
C(11)	3436 (5)	9307 (6)	−1911 (5)	26 (2)
C(12)	5006 (5)	7520 (6)	−262 (4)	23 (2)
C(13)	5489 (5)	7308 (6)	−911 (4)	24 (2)
O(14)	7276 (4)	5669 (5)	−338 (4)	34 (2)
C(15)	7566 (5)	5036 (6)	1886 (5)	27 (2)
S(16)	7516 (1)	4763 (2)	3055 (1)	29 (1)
C(17)	8820 (5)	3516 (7)	3826 (5)	32 (3)
C(18)	9278 (6)	3363 (7)	3230 (6)	34 (3)
C(19)	8585 (5)	4233 (7)	2113 (5)	25 (2)
C(20)	9324 (6)	2719 (8)	5021 (6)	42 (3)

*$B_{eq} = \frac{4}{3}\sum_i\sum_j \beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

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Table 2. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

N(1)—N(2)	1·409 (8)	N(1)—C(12)	1·328 (9)
N(2)—C(3)	1·380 (9)	N(2)—C(15)	1·411 (9)
C(3)—C(13)	1·417 (10)	C(3)—O(14)	1·265 (9)
C(4)—N(5)	1·336 (9)	C(4)—C(13)	1·363 (10)
N(5) C(11)	1·399 (9)	C(6) C(7)	1·385 (10)
C(6)—C(11)	1·388 (10)	C(7)—C(8)	1·375 (11)
C(8)—C(9)	1·376 (11)	C(9)—C(10)	1·409 (10)
C(10)—C(11)	1·409 (10)	C(10)—C(12)	1·437 (10)
C(12)—C(13)	1·441 (10)	C(15)—S(16)	1·705 (7)
C(15)—C(19)	1·386 (10)	S(16)—C(17)	1·724 (7)
C(17)—C(18)	1·336 (11)	C(17)—C(20)	1·506 (11)
C(18)—C(19)	1·411 (11)		
N(2)—N(1)—C(12)	103·2 (5)	N(1)—N(2)—C(3)	114·5 (5)
N(1)—N(2)—C(15)	119·0 (5)	C(3)—N(2)—C(15)	126·4 (6)
N(2)—C(3)—C(13)	103·6 (6)	N(2)—C(3)—O(14)	124·5 (6)
C(13)—C(3)—O(14)	131·9 (7)	N(5)—C(4)—C(13)	118·8 (6)
C(4)—N(5)—C(11)	123·5 (6)	C(7)—C(6)—C(11)	118·9 (6)
C(6)—C(7)—C(8)	120·9 (7)	C(7)—C(8)—C(9)	121·0 (7)
C(8)—C(9)—C(10)	119·8 (7)	C(9)—C(10)—C(11)	118·3 (6)
C(9)—C(10)—C(12)	124·9 (6)	C(11)—C(10)—C(12)	116·8 (6)
N(5)—C(11)—C(6)	118·5 (6)	N(5)—C(11)—C(10)	120·4 (6)
C(6)—C(11)—C(10)	121·1 (6)	N(1)—C(12)—C(10)	129·0 (6)
N(1)—C(12)—C(13)	112·3 (6)	C(10)—C(12)—C(13)	118·7 (6)
C(3)—C(13)—C(4)	131·8 (7)	C(3)—C(13)—C(12)	106·3 (6)
C(4)—C(13)—C(12)	121·8 (6)	N(2)—C(15)—S(16)	120·5 (5)
N(2)—C(15)—C(19)	127·8 (6)	S(16)—C(15)—C(19)	111·7 (5)
C(15)—S(16)—C(17)	91·3 (3)	S(16)—C(17)—C(18)	112·0 (6)
S(16)—C(17)—C(20)	121·5 (5)	C(18)—C(17)—C(20)	126·5 (7)
C(17)—C(18)—C(19)	113·5 (7)	C(15)—C(19)—C(18)	111·4 (7)

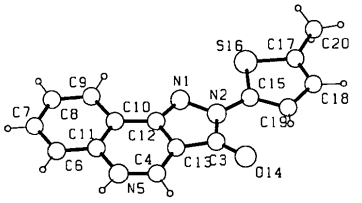

Fig. 1. Perspective view of the title compound, with the atom-numbering system.

factors were calculated by $\sum[a_i\exp(-b_i\lambda^{-2}\sin^2\theta)] + c$ ($i = 1, 2, 3, 4$) (*International Tables for X-ray Crystallography*, 1974). Calculations were performed on a FACOM M340R computer at Shionogi Research Laboratories. Final atomic coordinates and equivalent isotropic temperature factors are given in Table 1. Bond distances and angles are listed in Table 2.* A perspective view of the molecule with the atom-numbering system, drawn using the program *PLUTO* (Motherwell & Clegg, 1978), is presented in Fig. 1.

Related literature. The structure of the title compound has been discussed by Shindo, Takada, Murata, Eigyo & Matsushita (1989).

* Lists of H-atom coordinates, anisotropic temperature factors of the non-H atoms and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52627 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 4-Piperidone Derivatives. I. 3-Methyl-2,6-diphenyl-4-piperidone

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Abstract. $C_{18}H_{19}NO$, $M_r = 265{\cdot}3$, monoclinic, $C2/c$, $a = 19{\cdot}266\ (2)$, $b = 6{\cdot}999\ (2)$, $c = 21{\cdot}653\ (1)$ Å, $\beta = 94{\cdot}42\ (1)°$, $V = 2911{\cdot}07$ Å^3, $Z = 8$, $D_x = 1{\cdot}21$ g cm^{-3}, λ(Cu $K\alpha$) $= 1{\cdot}5418$ Å, $\mu = 5{\cdot}05$ cm^{-1}, $F(000) = 1136$, $T = 295$ K, $R = 0{\cdot}047$ for 1655 unique observed reflections [$I > 3\sigma(I)$]. The 4-piperidone has

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