Structure of a Furo[3,2-*b*]pyrrole Derivative

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Abstract. Ethyl 4H-furo[3,2-b]pyrrole-5-carboxylate, $C_9H_9NO_3$, $M_r = 179 \cdot 175$, monoclinic, $P2_1/n$, $a = 5 \cdot 356$ (1), $b = 11 \cdot 973$ (3), $c = 13 \cdot 486$ (5) Å, $\beta = 91 \cdot 44$ (3)°, $V = 864 \cdot 55$ Å^3, $Z = 4$, $D_m = 1 \cdot 38$, $D_x = 1 \cdot 38$ Mg m^{-3}, $\lambda(\text{Cu } K\alpha) = 1 \cdot 54178$ Å, $\mu = 0 \cdot 94$ mm^{-1}, $F(000) = 376$, $T = 293$ K. Final $R = 0 \cdot 048$ for 1088 unique reflections. The molecule consists of furan and pyrrole rings which are nearly coplanar, with a dihedral angle between the rings of $1 \cdot 0$ (2)°. The molecules are linked by an N—H···O hydrogen bond.

Experimental. Colourless prismatic crystal with dimensions $0 \cdot 06 \times 0 \cdot 06 \times 0 \cdot 55$ mm; D_m by flotation in n-octane/CCl$_4$; monoclinic space group $P2_1/n$ (No. 14), lattice parameters and Bravais translation lattice found by program *UB* (Sivý, Sivý & Koreň, 1987). Intensities collected with Syntex $P2_1$ diffractometer, $\lambda(\text{Cu } K\alpha) = 1 \cdot 54178$ Å, graphite monochromator, $\theta/2\theta$ scan, $2\theta_{max} = 110°$; time per reflection *ca* 60s, two standard reflections, variation $4 \cdot 3\%$; 25 reflections with $10 \cdot 0 < 2\theta < 35 \cdot 8°$ used for refinement of lattice parameters; minimum and maximum transmission factors $0 \cdot 7143$, $0 \cdot 7246$ (absorption correction not applied); index range $0 \le h \le 5$, $0 \le k \le 12$, $-14 \le l \le 14$; 1338 reflections measured, 1088 unique, $R_{int} = 0 \cdot 03$ (for 276 reflections), 786 reflections observed with $I > 2 \cdot 0\sigma(I)$, 302 unobserved. Data reduction carried out with program *XP21* (Pavelčik, 1987). Furan ring located initially, using a five-membered ring in program *PATSEE* (Egert, 1985). Inclusion of the furan ring in program *SHELXS86* (Sheldrick, 1986) gave positions of all non-H atoms of the structure. Anisotropic refinement by least-squares (full matrix, F values). All H atoms were located from a difference Fourier map and refined isotropically. Scattering factors and f', f'' from *International Tables for X-ray Crystallography* (1974). Maximum positive and maximum negative electron density in final difference Fourier synthesis $0 \cdot 17$ and $-0 \cdot 26$ e Å^{-3}; final $R = 0 \cdot 048$, $wR = 0 \cdot 055$, $w = 1 \cdot 0/[\sigma^2(F_o) + 0 \cdot 0110F_o^2]$; $(\Delta/\sigma)_{max} = 0 \cdot 01$ in final refine-

ment cycle (154 parameters). Calculations performed using an M4030-1 computer, Slovak Technical University, Bratislava, Czechoslovakia, with *SHELX76* (Sheldrick, 1976). Structure and atomic numbering shown in Fig. 1. Projection of crystal structure in Fig. 2. Fractional atomic coordinates for non H-atoms are in Table 1; bond distances and angles are in Table 2.*

The structural units (Fig. 2) are joined to each other by a hydrogen bond between atoms N(8)—H(8)···O(10) with angle at H of $156 \cdot 4$ (6)° and distance N(8)···O(10) $2 \cdot 88$ (4) Å.

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* Lists of structure amplitudes, anisotropic thermal parameters, H-atom parameters and details of least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51081 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
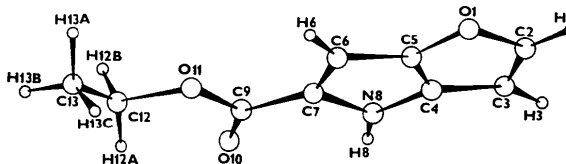

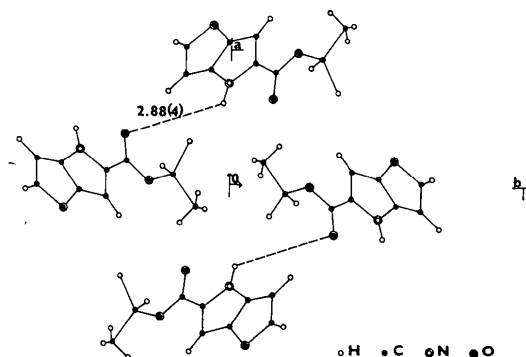
Fig. 1. View of molecule, with atomic numbering.

Fig. 2. Projection of crystal structure down *c* axis (half of unit cell).

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{4}{3}\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i\cdot\mathbf{a}_j.$$

	x	y	z	$B_{eq}(\text{Å}^2)$
O(1)	0·1754 (5)	0·5609 (2)	0·8159 (2)	5·2 (1)
C(2)	0·0146 (8)	0·6478 (3)	0·8387 (3)	4·8 (1)
C(3)	−0·1832 (8)	0·6541 (3)	0·7772 (3)	4·5 (1)
C(4)	−0·1531 (7)	0·5661 (3)	0·7088 (2)	3·7 (1)
C(5)	0·0639 (7)	0·5116 (3)	0·7342 (3)	3·8 (1)
C(6)	0·1028 (7)	0·4219 (3)	0·6705 (3)	4·1 (1)
C(7)	−0·1012 (6)	0·4247 (3)	0·6050 (2)	3·5 (1)
N(8)	−0·2545 (6)	0·5121 (2)	0·6285 (2)	3·8 (1)
C(9)	−0·1715 (7)	0·3512 (2)	0·5230 (2)	3·4 (1)
O(10)	−0·3633 (5)	0·3609 (2)	0·4740 (2)	4·8 (1)
O(11)	−0·0073 (4)	0·2695 (2)	0·5092 (2)	4·0 (1)
C(12)	−0·0700 (8)	0·1891 (3)	0·4306 (3)	4·6 (1)
C(13)	0·1565 (10)	0·1202 (5)	0·4143 (4)	5·8 (1)

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

O(1)—C(2)	1·390 (5)	N(8)—C(4)	1·362 (4)
O(1)—C(5)	1·373 (5)	N(8)—C(7)	1·372 (4)
C(3)—C(2)	1·331 (6)	C(9)—C(7)	1·455 (4)
C(3)—C(4)	1·412 (5)	C(9)—O(10)	1·213 (4)
C(5)—C(4)	1·369 (5)	C(9)—O(11)	1·332 (4)
C(5)—C(6)	1·394 (5)	C(12)—O(11)	1·465 (5)
C(6)—C(7)	1·388 (5)	C(12)—C(13)	1·488 (7)
C(2)—O(1)—C(5)	103·8 (3)	C(6)—C(7)—N(8)	109·7 (3)
O(1)—C(2)—C(3)	113·1 (3)	C(6)—C(7)—C(9)	130·8 (3)
C(2)—C(3)—C(4)	105·2 (3)	N(8)—C(7)—C(9)	119·5 (3)
C(3)—C(4)—C(5)	107·6 (3)	C(4)—N(8)—C(7)	108·5 (3)
C(3)—C(4)—N(8)	145·4 (4)	C(7)—C(9)—O(10)	123·7 (3)
C(5)—C(4)—N(8)	107·0 (3)	C(7)—C(9)—O(11)	113·0 (3)
O(1)—C(5)—C(4)	110·2 (3)	O(10)—C(9)—O(11)	123·2 (3)
O(1)—C(5)—C(6)	139·2 (3)	C(9)—O(11)—C(12)	116·3 (3)
C(4)—C(5)—C(6)	110·6 (3)	O(11)—C(12)—C(13)	107·4 (4)
C(5)—C(6)—C(7)	104·3 (3)		

Related literature. Synthesis and chemical behaviour published by Hemetsberger & Knittel (1972) and Krutošíková, Dandárová, Alföldi & Kováč (1987). Intermolecular hydrogen bonds in related crystal structures are discussed by Ringertz (1971) and Harrison, Rettig & Trotter (1972).

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Structure of Diazapolycyclic Compounds. XI. 2,3-Epoxy-*trans*-1,2-dimethyl-1,2,3,4-tetrahydrobenzo[*g*]pyridazino[1,2-*b*]phthalazine-6,13-dione

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Abstract. $C_{18}H_{16}N_2O_3$, $M_r = 308\cdot34$, orthorhombic, $Pna2_1$, $a = 16\cdot4036\,(7)$, $b = 11\cdot3924\,(4)$, $c = 7\cdot9058\,(2)$ Å, $V = 1477\cdot4\,(1)$ Å^3, $Z = 4$, $D_x = 1\cdot386$ Mg m^{-3}, graphite-monochromated Cu $K\alpha$ radiation, $\lambda = 1\cdot5418$ Å, $\mu = 0\cdot741$ mm^{-1}, $F(000) = 648$, $T = 293$ K, $R = 0\cdot033$ for 1189 observed reflexions $[I > 3\sigma(I)]$. The methyl groups are *trans* to each other. The pyridazine ring displays a distorted envelope conformation with the flap at N5 giving rise to some loss of the sp^2 hybridization at N5 and N14 [angles around adding up to 353·6 (3) and 353·2 (3)°]. The molecules pack in zigzag chains along the a and b axes, held together by van der Waals interactions only.

Experimental. Yellow prismatic sample, $0\cdot52 \times 0\cdot51 \times 0\cdot83$ mm, used for data collection and determination of lattice constants (Cu $K\alpha$, 89 reflexions up to $\theta = 45°$). Philips PW 1100 diffractometer, Cu $K\alpha$, graphite