

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters with *e.s.d.*'s in parentheses
$$B_{eq} = \frac{4}{3} \sum_i \sum_j \beta_{ij} a_i \cdot a_j$$

	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 Krutošiková, 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

BY C. FOCES-FOCES AND F. H. CANO

UEI de Cristalografía, Instituto del Química-Física Rocasolano, CSIC, Serrano 119, 28006 Madrid, Spain

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Abstract. C₁₈H₁₆N₂O₃, $M_r = 308.34$, orthorhombic, *Pna*2₁, $a = 16.4036$ (7), $b = 11.3924$ (4), $c = 7.9058$ (2) Å, $V = 1477.4$ (1) Å³, $Z = 4$, $D_x = 1.386$ Mg m⁻³, graphite-monochromated Cu $K\alpha$ radiation, $\lambda = 1.5418$ Å, $\mu = 0.741$ mm⁻¹, $F(000) = 648$, $T = 293$ K, $R = 0.033$ 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, 0.52 × 0.51 × 0.83 mm, used for data collection and determination of lattice constants (Cu $K\alpha$, 89 reflexions up to $\theta = 45^\circ$). Philips PW 1100 diffractometer, Cu $K\alpha$, graphite

Table 1. Final atomic coordinates and equivalent isotropic thermal parameters

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* \cos(a_i, a_j)$$

	x	y	z	$U_{eq}(\text{\AA}^2 \times 10^4)$
C1	0.3346 (2)	0.0030 (2)	-0.4015	432 (8)
C2	0.2841 (2)	-0.1074 (2)	-0.3845 (5)	439 (8)
C3	0.2862 (2)	-0.1741 (3)	-0.2277 (5)	524 (9)
C4	0.3374 (2)	-0.1344 (2)	-0.0827 (5)	545 (10)
N5	0.3578 (1)	-0.0096 (2)	-0.0962 (4)	460 (7)
C6	0.3768 (2)	0.0494 (3)	0.0489 (5)	500 (9)
C6a	0.4340 (2)	0.1480 (2)	0.0343 (5)	442 (8)
C7	0.4516 (2)	0.2144 (2)	0.1745 (5)	500 (9)
C7a	0.5090 (2)	0.3067 (2)	0.1646 (5)	495 (9)
C8	0.5277 (2)	0.3781 (3)	0.3072 (6)	624 (11)
C9	0.5825 (2)	0.4672 (3)	0.2938 (6)	680 (12)
C10	0.6214 (2)	0.4900 (3)	0.1389 (6)	698 (13)
C11	0.6054 (2)	0.4236 (3)	-0.0010 (6)	621 (11)
C11a	0.5484 (2)	0.3299 (2)	0.0082 (5)	493 (9)
C12	0.5297 (2)	0.2595 (3)	-0.1334 (5)	502 (9)
C12a	0.4736 (2)	0.1703 (2)	-0.1223 (5)	451 (8)
C13	0.4486 (2)	0.1057 (2)	-0.2746 (5)	473 (8)
N14	0.3909 (1)	0.0204 (2)	-0.2567 (4)	435 (7)
O15	0.3491 (2)	0.0161 (2)	0.1851 (4)	702 (9)
O16	0.4737 (2)	0.1320 (2)	-0.1465 (4)	633 (7)
C17	0.2808 (2)	0.1103 (3)	-0.4250 (6)	630 (11)
C18	0.2610 (2)	-0.1653 (3)	-0.5487 (5)	557 (10)
O19	0.2175 (1)	-0.1002 (2)	-0.2640 (4)	589 (7)

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

C1—C2	1.512 (4)	C1—N14	1.485 (4)
C1—C17	1.519 (4)	C2—C3	1.454 (5)
C2—C18	1.505 (5)	C2—O19	1.451 (4)
C3—C4	1.492 (5)	C3—O19	1.435 (4)
C4—N5	1.465 (4)	N5—C6	1.366 (5)
N5—N14	1.421 (4)	C6—C6a	1.468 (4)
C6—O15	1.229 (5)	C6a—C12a	1.421 (5)
C7a—C11a	1.420 (5)	C12a—C13	1.469 (5)
C13—N14	1.365 (4)	C13—O16	1.232 (5)
N14—C1—C17	110.4 (2)	C2—C1—C17	111.2 (2)
C2—C1—N14	112.5 (2)	C1—C2—O19	115.1 (2)
C1—C2—O18	115.2 (2)	C1—C2—C3	119.9 (2)
C18—C2—O19	113.7 (3)	C3—C2—O19	59.2 (2)
C3—C2—O18	120.8 (3)	C2—C3—O19	60.3 (2)
C2—C3—C4	120.7 (3)	C4—C3—O19	114.7 (3)
C3—C4—N5	111.5 (3)	C2—O19—C3	60.5 (2)
C4—N5—N14	112.7 (2)	C4—N5—C6	117.9 (3)
C6—N5—N14	123.0 (2)	N5—C6—O15	120.0 (3)
N5—C6—C6a	117.1 (3)	C6a—C6—O15	122.8 (3)
C6—C6a—C12a	119.8 (3)	C6a—C12a—C13	119.9 (3)
C12a—C13—O16	122.1 (3)	C12a—C13—N14	117.7 (3)
N14—C13—O16	120.0 (3)	N5—N14—C13	121.9 (3)
C1—N14—C13	116.5 (3)	C1—N14—N5	114.8 (2)
C2—C1—N14—N5	-39.3 (3)	N14—C1—C2—C3	8.1 (4)
C1—C2—C3—C4	0.3 (5)	C2—C3—C4—N5	20.2 (4)
C3—C4—N5—N14	-51.0 (4)	C4—N5—N14—C1	63.3 (3)
C17—C1—N14—N5	85.6 (3)	N14—C1—C2—O19	75.6 (3)
N14—C1—C2—C18	-149.2 (3)	C17—C1—C2—C18	86.4 (3)

monochromator, bisecting geometry, $\omega/2\theta$ -scan mode, 1236 independent reflexions up to $\theta = 65^\circ$ in the hkl range 0,14; 0,20; 0,10, 1189 considered observed with $I > 3\sigma(I)$. Two standard reflexions were measured every 90 min, no decay observed. The structure was solved by means of the *ORIENT* program (*DIRDIF* system, Beurskens *et al.*, 1984) using a three-ring-system model taken from an analogous compound [(I) in Apreda, Foces-Foces, Cano & García-Blanco

(1982)]. H atoms, from difference synthesis, included isotropically in last cycles of refinement. Empirical weights so as to give no trends in $\langle w\Delta F^2 \rangle$ versus $\langle |F_o| \rangle$ and $\langle (\sin\theta)/\lambda \rangle$. $R(F) = 0.033$, $wR = 0.037$, $S = 1.06$. Max. and average $\Delta/\sigma = 0.90$ and 0.06 . Final $\Delta\rho = \pm 0.15 \text{ e \AA}^{-3}$. No correction for extinction or absorption. Computing with *XRAY76* (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976). Scattering factors from *International Tables for X-ray Crystallography* (1974). Final atomic coordinates for the non-H atoms and the main geometrical parameters are given in Tables 1 and 2,* according to the numbering scheme given in Fig. 1 (Allen *et al.*, 1979). Crystal packing is shown in Fig. 2.

Related literature. The conformation of the pyridazine ring (Fig. 2) is analogous to that in 2,3-epoxy-*trans*-1,3-dimethyl-4a,9a-diaza-1,2,3,4,4a,9,9a,10-octahydroanthracene-9,10-dione (Foces-Foces, Cano & García-Blanco, 1978). The configurational angles of the methyl substituents, with different signs, describe their

* Lists of structure factors, anisotropic thermal factors and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51115 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

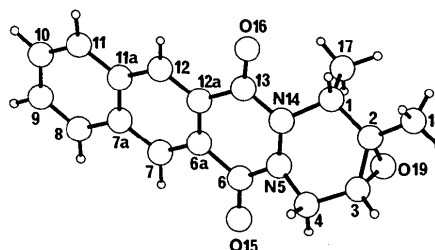
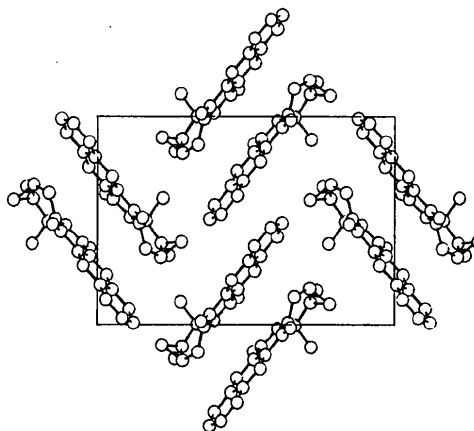


Fig. 1. A view of the molecule showing the atomic numbering.

Fig. 2. A view along the *c* axis of the packing of the title compound showing the zigzag disposition of the molecules.

trans disposition (Foces-Foces, Cano & Garcia-Blanco, 1980).

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Structure of Ethyl 2-Amino-4,5,6,7-tetrahydrobenzo[*b*]thiophene-3-carboxylate at 163 K

BY S. B. LARSON* AND S. H. SIMONSEN

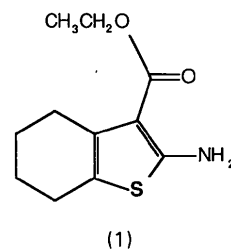
Department of Chemistry, University of Texas at Austin, Austin, TX 78712, USA

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Abstract. C₁₁H₁₅NO₂S, $M_r = 225.31$, monoclinic, $P2_1/c$, $a = 8.2996$ (13), $b = 9.5307$ (12), $c = 14.6009$ (20) Å, $\beta = 105.818$ (13)°, $V = 1111.2$ (3) Å³, $Z = 4$, $D_x(163\text{ K}) = 1.347$, $D_m(295\text{ K}) = 1.310$ (1) g cm⁻³, $\lambda(\text{MoK}\alpha) = 0.71069$ Å, $\mu = 2.68$ cm⁻¹, $F(000) = 480$, $R = 0.0407$ for 2527 reflections [$F_o \geq 4\sigma(F_o)$]. The thiophene ring is planar [max. deviation: C(9), 0.007 (2) Å] as is the carboxyl group, C(3), C(10), O(1), O(2) [max. deviation: C(10), 0.001 (2) Å]; the twist between these planes is only 0.96 (7)°. Planarity of the N, C(2), C(3), C(10), O(1), O(2), C(11) fragment is favored because of the intramolecular hydrogen bond [O(1)··H(N)B, 2.08 (3) Å]; and conjugation which is evident in the short single bonds [C(2)–N and C(3)–C(10)] and long double bonds [C(2)–C(3) and C(10)–O(1)] in this fragment. The N is slightly pyramidal [max. deviation: N, –0.105 (2) Å]. The intermolecular hydrogen bonding [O(1)··H(N)A ($1-x, \frac{1}{2}+y, \frac{1}{2}-z$), 2.01 (2) Å] connects the molecules along the *b* axis. All other intermolecular contacts are not less than the sum of the respective van der Waals radii.

Experimental. Large colorless, transparent prismatic crystals of (1) were obtained by sublimation from a

sample purchased from Aldrich Chemical Co. Density was measured by flotation in aqueous ZnCl₂. Table 1 is a summary of data collection and structural refinement.



The structure was solved by *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), and refined by full-matrix least squares (*SHELX76*, Sheldrick, 1976). Scattering factors and anomalous-dispersion corrections for non-H atoms were taken from *International Tables for X-ray Crystallography* (1974); H from Stewart, Davidson & Simpson (1965). All atomic positional parameters were refined. Non-H atoms were refined anisotropically; H atoms isotropically. The H atoms were located in an electron density difference map as peaks of 0.60–0.94 e Å⁻³. Atom labeling, bond lengths and bond angles are shown in Fig. 1. Fig. 2 illustrates the molecular packing and

* Current address: Nucleic Acid Research Institute, 3300 Hyland Avenue, Costa Mesa, CA 92626, USA.