

Fig. 2. Crystal packing diagram of (1) with C—H atoms of the sugar moiety omitted. Hydrogen bonds are drawn as thin lines. (a) View along the *a* axis illustrating the sandwiching of the cyano group between triazole rings of adjacent molecules along the *z* axis parallel to *a*. Hydrogen bonding also occurs between adjacent molecules along this screw axis. (b) View along the *b* axis showing how the triazole planes are parallel to the *bc* plane. Hydrogen bonding only occurs between molecules along the *a*-parallel screw axes or translated in the *b* direction.

Related literature. The title compound (1) has shown some antiparasitic activity (Kini *et al.*, 1990). The preceding papers report the structures of the carboxamide (Larson, Kini & Robins, 1990) and thio-carboxamide (Larson, Henry, Kini & Robins, 1990) derivatives of (1). In each of these structures the amino group of the sulfamoyl moiety is over the furan ring, regardless of the C5'—O5' orientation. Several other triazole nucleosides have been reported (Prusiner & Sundaralingam, 1976; Haines, Leonard & Wiemer, 1982; Sanghvi, Hanna, Larson, Fujitaki, Willis, Smith, Robins & Revankar, 1988; Hanna, Dimitrijevič, Larson, Robins & Revankar, 1988). A

search of the Cambridge Structural Database (1989) revealed no structures containing the 5-*O*-sulfamoyl-ribose moiety. Conformational parameters follow the conventions of Altona & Sundaralingam (1972).

References

- ALTONA, C. & SUNDARALINGAM, M. (1972). *J. Am. Chem. Soc.* **94**, 8205–8212.
- Cambridge Structural Database (1989). Univ. Chemical Laboratory, Lensfield Road, Cambridge, England.
- CORDES, A. W. (1983). Personal communication.
- FRENZ, B. A. (1985). *Enraf-Nonius SDP-Plus Structure Determination Package*. Version 3.0. Enraf-Nonius, Delft, The Netherlands.
- HAINES, D. R., LEONARD, N. J. & WIEMER, D. F. (1982). *J. Org. Chem.* **47**, 474–482.
- HANNA, N. B., DIMITRIJEVIČ, S. D., LARSON, S. B., ROBINS, R. K. & REVANKAR, G. R. (1988). *J. Heterocycl. Chem.* **25**, 1857–1868.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- JOHNSON, C. K. (1976). ORTEPII. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- KINI, G. D., HENRY, E. M., ROBINS, R. K., LARSON, S. B., MARR, J. J., BERENS, R. L., BACCHI, C. J., NATHAN, H. C. & KEITHLY, J. S. (1990). *J. Med. Chem.* **33**, 121–128.
- LARSON, S. B. (1980). PhD Dissertation, Brigham Young Univ., Provo, Utah, USA.
- LARSON, S. B., HENRY, E. M., KINI, G. D. & ROBINS, R. K. (1990). *Acta Cryst.* **C46**, 1138–1141.
- LARSON, S. B., KINI, G. D. & ROBINS, R. K. (1990). *Acta Cryst.* **C46**, 1135–1138.
- PRUSINER, P. & SUNDARALINGAM, M. (1976). *Acta Cryst.* **B32**, 419–426.
- SANGHVI, Y. S., HANNA, N. B., LARSON, S. B., FUJITAKI, J. M., WILLIS, R. C., SMITH, R. A., ROBINS, R. K. & REVANKAR, G. R. (1988). *J. Med. Chem.* **31**, 330–335.
- SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
- SHELDRICK, G. M. (1986). *SHELXS86*. Program for crystal structure solution. Univ. of Göttingen, Federal Republic of Germany.
- STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Acta Cryst. (1990). **C46**, 1143–1145

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

BY HIROSHI NAKAI

Shionogi Research Laboratories, Shionogi & Co. Ltd, Fukushima-Ku, Osaka 553, Japan

(Received 13 November 1989; accepted 2 January 1990)

Abstract. C₁₅H₁₁N₃OS, *M_r* = 281.33, monoclinic, *P*2₁/*c*, *a* = 9.972 (7), *b* = 9.650 (5), *c* = 14.000 (3) Å, β = 101.28 (3)°, *V* = 1321 (1) Å³, *Z* = 4, *D_x* = 1.414 Mg m⁻³, λ(Cu Kα) = 1.54178 Å, μ = 0.108–2701/90/061143-03\$03.00

2.11 mm⁻¹, *F*(000) = 584, *T* = 295 K, *R* = 0.038 for 1897 observed reflections [*F_o* > 3σ(*F_o*)]. The molecule is almost planar except for H atoms of the methyl group, but torsion angles N(11)—N(12)—C(15)—

© 1990 International Union of Crystallography

Table 1. Atomic coordinates and equivalent isotropic temperature factors (\AA^2) with e.s.d.'s in parentheses

	$B_{\text{eq}} = (4/3)\sum_i \sum_j \beta_{ij} a_i \cdot a_j$			
	x	y	z	B_{eq}
C(1)	0.3131 (2)	0.4245 (2)	0.5460 (1)	3.47 (5)
C(2)	0.2180 (2)	0.5300 (2)	0.5345 (1)	3.92 (5)
C(3)	0.1780 (2)	0.5941 (2)	0.4443 (1)	4.02 (5)
C(4)	0.2304 (2)	0.5522 (2)	0.3657 (1)	3.79 (5)
N(5)	0.3790 (1)	0.4042 (2)	0.2956 (1)	3.41 (4)
C(6)	0.4698 (2)	0.3028 (2)	0.2987 (1)	3.32 (4)
C(7)	0.5166 (2)	0.2356 (2)	0.3848 (1)	2.88 (4)
C(8)	0.4716 (2)	0.2731 (2)	0.4718 (1)	2.72 (4)
C(9)	0.3694 (2)	0.3798 (2)	0.4673 (1)	2.79 (4)
C(10)	0.3263 (2)	0.4455 (2)	0.3765 (1)	3.10 (4)
N(11)	0.5343 (1)	0.2037 (1)	0.5488 (1)	2.83 (3)
N(12)	0.6253 (1)	0.1177 (1)	0.5110 (1)	2.84 (3)
C(13)	0.6191 (2)	0.1314 (2)	0.4121 (1)	2.95 (4)
O(14)	0.6925 (1)	0.0662 (1)	0.3651 (1)	3.71 (3)
C(15)	0.7165 (2)	0.0344 (2)	0.5747 (1)	2.87 (4)
S(16)	0.71803 (5)	-0.04525 (5)	0.69764 (3)	3.74 (1)
C(17)	0.8439 (2)	-0.0799 (2)	0.7208 (1)	4.25 (5)
C(18)	0.8822 (2)	-0.1254 (2)	0.6388 (1)	3.64 (5)
C(19)	0.8084 (2)	-0.0582 (2)	0.5539 (1)	3.53 (5)
C(20)	0.9897 (2)	-0.2334 (2)	0.6356 (2)	5.14 (6)

Table 2. Bond lengths (\AA) and angles ($^\circ$) with e.s.d.'s in parentheses

C(1)—C(2)	1.379 (3)	C(9)—C(10)	1.410 (3)
C(1)—C(9)	1.399 (3)	N(11)—N(12)	1.407 (2)
C(2)—C(3)	1.393 (3)	N(12)—C(13)	1.380 (2)
C(3)—C(4)	1.368 (3)	N(12)—C(15)	1.397 (2)
C(4)—C(10)	1.393 (3)	C(13)—O(14)	1.246 (2)
N(5)—C(6)	1.328 (3)	C(15)—S(16)	1.721 (2)
N(5)—C(10)	1.397 (3)	C(15)—C(19)	1.352 (3)
C(6)—C(7)	1.368 (3)	S(16)—C(17)	1.726 (2)
C(7)—C(8)	1.425 (3)	C(17)—C(18)	1.352 (3)
C(7)—C(13)	1.431 (3)	C(18)—C(19)	1.426 (3)
C(8)—C(9)	1.441 (3)	C(18)—C(20)	1.502 (3)
C(8)—N(11)	1.318 (2)		
C(2)—C(1)—C(9)	120.6 (2)	N(5)—C(10)—C(9)	120.4 (2)
C(1)—C(2)—C(3)	120.1 (2)	C(8)—N(11)—N(12)	103.3 (1)
C(2)—C(3)—C(4)	120.7 (2)	N(11)—N(12)—C(13)	114.5 (1)
C(3)—C(4)—C(10)	119.5 (2)	N(11)—N(12)—C(15)	119.1 (1)
C(6)—N(5)—C(10)	123.1 (2)	C(13)—N(12)—C(15)	126.3 (2)
N(5)—C(6)—C(7)	119.4 (2)	C(7)—C(13)—N(12)	103.0 (2)
C(6)—C(7)—C(8)	121.5 (2)	C(7)—C(13)—O(14)	132.5 (2)
C(6)—C(7)—C(13)	132.1 (2)	N(12)—C(13)—O(14)	124.4 (2)
C(8)—C(7)—C(13)	106.1 (2)	N(12)—C(15)—S(16)	118.7 (1)
C(7)—C(8)—C(9)	118.8 (2)	N(12)—C(15)—C(19)	128.8 (2)
C(7)—C(8)—N(11)	113.1 (2)	S(16)—C(15)—C(19)	112.4 (2)
C(9)—C(8)—N(11)	128.1 (2)	C(15)—S(16)—C(17)	90.5 (1)
C(1)—C(9)—C(8)	125.1 (2)	S(16)—C(17)—C(18)	112.6 (2)
C(1)—C(9)—C(10)	118.2 (2)	C(17)—C(18)—C(19)	112.0 (2)
C(8)—C(9)—C(10)	116.7 (2)	C(17)—C(18)—C(20)	124.9 (2)
C(4)—C(10)—N(5)	118.8 (2)	C(19)—C(18)—C(20)	123.1 (2)
C(4)—C(10)—C(9)	120.8 (2)	C(15)—C(19)—C(18)	112.5 (2)

S(16) = $-2.3 (2)^\circ$ and C(13)—N(12)—C(15)—C(19) = $-6.3 (3)^\circ$ show about 4.3° rotation of the thiophene ring around the N(12)—C(15) bond. The molecules form an intermolecular hydrogen bond between NH and O($1-x, \frac{1}{2}+y, \frac{1}{2}-z$), N \cdots O = $2.718 (2) \text{\AA}$ [$1.82 (3) \text{\AA}$ for H \cdots O].

Experimental. Needle-like pale brown crystals obtained from ethanol. Crystal of dimensions $0.1 \times$

$0.1 \times 0.3 \text{ mm}$. Rigaku AFC-5R diffractometer, graphite-monochromatized Cu $K\alpha$. Cell dimensions determined from 2θ angles for 25 reflections in the range $15 < 2\theta < 39^\circ$. Intensities measured up to $\theta = 65^\circ$ in $h0/11, k0/11$ and $l-15/15, \omega-2\theta$ scans, ω -scan width $(1.3 + 0.2 \tan \theta)^\circ$, three standard reflections monitored every 100 measurements showed no significant change. 2171 unique reflections measured, 1897 intensities observed [$F_o \leq 3\sigma(F_o)$] and three very strong reflections rejected, no absorption corrections. Structures solved by MULTAN84 (Main, Germain & Woolfson, 1984). H atoms located on a difference density map. Positional parameters of all atoms and anisotropic thermal parameters of non-H atoms refined by block-diagonal least squares. Temperature factor of each H atom equal to B_{eq} of the bonded atom. $\sum(w|\Delta F|^2)$ minimized, $w = 1/[\sigma^2(F_o) + 0.00091|F_o|^2]$, $w = 0$ for nine reflections with $w^{1/2}|\Delta F| \geq 3$. The final $R = 0.038$, $wR = 0.046$, $S = 1.1446$. The highest and lowest peaks in the final difference map are 0.5 and -0.3 e \AA^{-3} . Max. Δ/σ in the final cycle 0.1 . Atomic scattering factors calculated by $\sum[a_i \exp(-b_i \lambda^{-2} \sin^2 \theta)] + c (i = 1, \dots, 4)$ (International Tables for X-ray Crystallography, 1974).

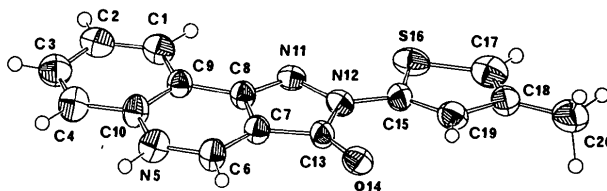


Fig. 1. ORTEP (Davenport & Hall, 1987) drawing. Non-H atoms are represented by thermal ellipsoids with 50% probability.

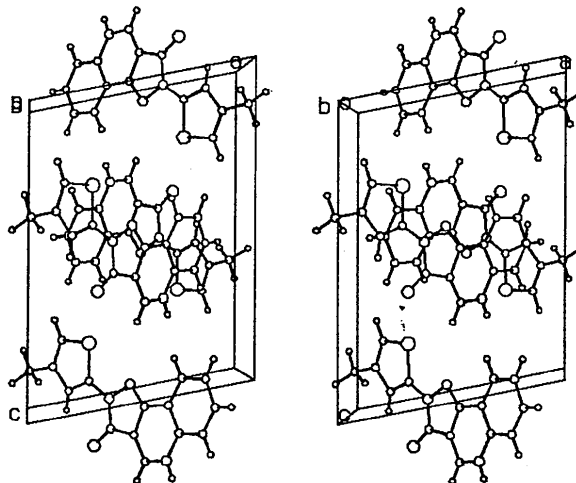


Fig. 2. Stereoview of the unit-cell packing drawn by PLUTO (Motherwell & Clegg, 1978).

Calculations performed on FACOM M340R computer at Shionogi Research Laboratories. The 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 atomic numbering system and a stereoview of the crystal packing are presented in Figs. 1 and 2, respectively.

Related literature. Structure-activity relationships of the title compound have been referred to 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 52570 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The author thanks Dr Takada for the supply of the crystals.

References

- DAVENPORT, G. & HALL, S. R. (1987). *ORTEP*. Version of *XTAL2.2 User's Manual*, edited by S. R. HALL & J. M. STEWART. Univ. of Western Australia, Australia, and Maryland, USA.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- MAIN, P., GERMAIN, G. & WOOLFSON, M. M. (1984). *MULTAN84. A Computer Program for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univ. of York, England, and Louvain, Belgium.
- MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.
- SHINDO, H., TAKADA, S., MURATA, S., EIGYO, M. & MATSUSHITA, A. (1989). *J. Med. Chem.* **32**, 1213–1217.

Acta Cryst. (1990). **C46**, 1145–1146

3-Diethylamino-2,4-bis(phenylthio)-2,4-pentadienenitrile

BY B. TINANT AND J.-P. DECLERCQ

Laboratoire de chimie physique et de cristallographie, Université Catholique de Louvain, 1 place Louis Pasteur, 1348 Louvain la Neuve, Belgium

AND M. VERMANDER, Z. JANOUSEK AND H. G. VIEHE

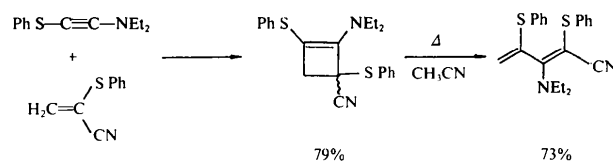
Laboratoire de chimie organique, Université Catholique de Louvain, 1 place Louis Pasteur, 1348 Louvain la Neuve, Belgium

(Received 10 November 1989; accepted 2 January 1990)

Abstract. $C_{21}H_{22}N_2S_2$, $M_r = 366.55$, monoclinic, $P2_1/a$, $a = 16.446$ (6), $b = 12.013$ (6), $c = 10.649$ (3) Å, $\beta = 110.21$ (2)°, $V = 1974$ (1) Å³, $Z = 4$, $D_x = 1.23$ g cm⁻³, $Mo K\alpha$, $\lambda = 0.71069$ Å, $\mu = 2.70$ cm⁻¹, $F(000) = 776$, $T = 291$ K, $R = 0.039$ for 3081 observed reflections. The configuration at the C2=C3 bond is *E*. Strong conjugation along N=C—C=C—NEt₂ is indicated by very short C1—C2 [1.408 (3) Å] and C3—N21 [1.333 (2) Å] distances, a very long C2=C3 bond [1.391 (3) Å] and by a small twist angle C1—C2=C3—N21 = 4.5 (6)°. The methylene group adopts a position nearly perpendicular to the C2=C3 bond plane [C2=C3—C4=C5 = 96.8 (6)°] and shows no evidence of conjugation [C4=C5 = 1.322 (3) Å].

Experimental. 2-Phenylthioacrylonitrile reacts smoothly with *N,N*-diethyl-2-(phenylthio)ethynylamine to give the corresponding cyclobutene

(Vermander, 1989). This compound undergoes cyclo reversion in refluxing acetonitrile thereby forming the title compound.



Parallelepiped crystal with dimensions 0.40 × 0.32 × 0.24 mm. Lattice parameters refined using 15 reflections in the range $5 \leq 2\theta \leq 30^\circ$. Syntex $P2_1$ diffractometer, graphite-monochromatized $Mo K\alpha$ radiation. 3877 $h,k,\pm l$ independent reflections with $(\sin\theta)/\lambda \leq 0.62$ Å⁻¹; $0 \leq h \leq 20$, $0 \leq k \leq 14$, $-12 \leq l \leq 12$, 3081 with $I \geq 2.5\sigma(I)$. Standard reflection (004) checked every 50 reflections: no significant