$S = 2.00$
836 reflections
181 parameters
Only coordinates of H atoms refined
Weighting scheme based on measured e.s.d.'s
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.2B and 2.3.1)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å²)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
S	0.1966 (3)	0.0689 (1)	0.0378 (2)	0.061 (1)
O1	1.3034 (7)	0.0599 (3)	0.4097 (5)	0.070 (3)
O2	1.0605 (7)	0.1469 (3)	0.3333 (5)	0.074 (3)
O3	0.2025 (7)	0.2401 (2)	0.0240 (5)	0.057 (3)
N1	0.8059 (8)	0.0044 (3)	0.2996 (6)	0.046 (4)
N2	0.9002 (9)	−0.1333 (3)	0.3333 (6)	0.051 (4)
N3	1.1283 (9)	0.0741 (4)	0.3630 (6)	0.056 (4)
N4	0.4889 (8)	0.1828 (3)	0.1085 (7)	0.062 (4)
C1	0.750 (1)	−0.0794 (5)	0.2944 (7)	0.047 (4)
C2	1.060 (1)	−0.0827 (4)	0.3622 (7)	0.047 (5)
C3	1.004 (1)	0.0018 (5)	0.3417 (7)	0.041 (4)
C4	0.667 (1)	0.0822 (5)	0.2745 (8)	0.059 (6)
C5	0.640 (1)	0.1112 (5)	0.1395 (9)	0.064 (5)
C6	0.300 (1)	0.1652 (4)	0.0588 (7)	0.049 (4)
C7	−0.004 (1)	0.2372 (6)	−0.0363 (9)	0.069 (7)
C8	0.545 (1)	−0.1082 (6)	0.250 (1)	0.070 (6)

Table 2. *Selected geometric parameters* (Å, °)

S—C6	1.649 (7)	N2—C1	1.329 (9)
O1—N3	1.228 (7)	N2—C2	1.35 (1)
O2—N3	1.226 (8)	N3—C3	1.41 (1)
O3—C6	1.343 (7)	N4—C5	1.520 (9)
O3—C7	1.43 (1)	N4—C6	1.332 (9)
N1—C1	1.352 (9)	C1—C8	1.48 (1)
N1—C3	1.364 (9)	C2—C3	1.36 (1)
N1—C4	1.54 (1)	C4—C5	1.46 (1)
C6—O3—C7	118.6 (6)	N2—C2—C3	109.1 (6)
C1—N1—C3	105.0 (6)	N1—C3—C2	108.0 (6)
C1—N1—C4	124.4 (6)	N1—C4—C5	105.9 (7)
C3—N1—C4	130.3 (6)	N4—C5—C4	106.6 (8)
C1—N2—C2	105.7 (5)	S—C6—O3	124.3 (5)
C5—N4—C6	121.4 (5)	S—C6—N4	127.2 (5)
N1—C1—N2	112.3 (6)	O3—C6—N4	108.5 (5)
C7—O3—C6—S	0 (1)	O2—N3—C3—N1	−6 (1)
C7—O3—C6—N4	177.9 (7)	C5—N4—C6—S	7 (1)
C1—N1—C4—C5	−101.8 (8)	C5—N4—C6—O3	−171.5 (7)
C3—N1—C4—C5	84.7 (9)	N1—C4—C5—N4	175.0 (5)
O1—N3—C3—C2	−3 (1)		

Data collection: Philips PW1100 software. Cell refinement: *Xtal LATCON* (Hall, Flack & Stewart, 1992). Data reduction: *Xtal REFCAL STARTX SORTRF*; *LSABS* (Blanc, Schwarzenbach & Flack, 1991). Program(s) used to solve structure: *MULTAN*87 (Main *et al.*, 1987). Program(s) used to refine structure: *Xtal CRYLSQ*. Molecular graphics: *Xtal ORTEP*. Software used to prepare material for publication: *Xtal CIFIO*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, including H-atom geometry, and torsion angles have been deposited with the IUCr (Reference: PA1156). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Berclaz, T., Bernardinelli, G., Geoffroy, M. & Rajalakshmi, N. (1992). *Chimia*, **46**, 130–132.
Blanc, E., Schwarzenbach, D. & Flack, H. D. (1991). *J. Appl. Cryst.* **24**, 1035–1041.
Blaton, N. M., Peeters, O. M. & De Ranter, C. J. (1979). *Acta Cryst.* B**35**, 753–755.
Hall, S. R., Flack, H. D. & Stewart, J. M. (1992). Editors. *Xtal*3.2 *Reference Manual*. Univs. of Western Australia, Australia, and Maryland, USA.
Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1987). *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
Olszak, T. A., Peeters, O. M., Blaton, N. M. & De Ranter, C. J. (1994). *Acta Cryst.* C**50**, 558–559.

Acta Cryst. (1995). C**51**, 1392–1394

6-Methylamino-4-methylthio-5-nitro-2-phenylpyrimidine

R. POMÉS HERNÁNDEZ,* J. DUQUE RODRÍGUEZ AND M. I. GARCÍA TRIMIÑO

National Centre for Scientific Research, PO Box 6990, Havana, Cuba

H. NOVOA DE ARMAS

Pharmaceutical Chemistry Centre, PO Box 16042, Havana, Cuba

R. ALFREDO TOSCANO

Institute of Chemistry, UNAM, 04510 Mexico DF

(*Received* 28 *September* 1993; *accepted* 28 *November* 1994)

Abstract

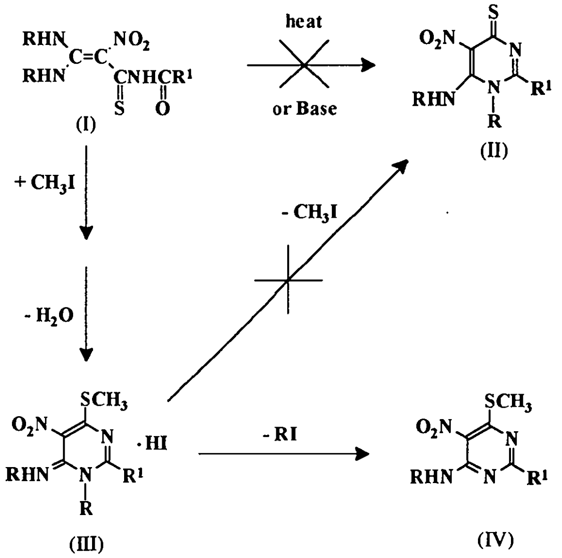
The title compound, $C_{12}H_{12}N_4O_2S$, is essentially planar, the dihedral angle between the pyrimidine and phenyl rings being 11.4 (3)°. There are no unusual intra- or intermolecular distances or angles. The molecules are packed with normal van der Waals distances.

Comment

Pyrimidine derivatives are constituents of naturally occurring molecules or, in synthetic form, useful as drugs or agricultural chemicals (Brown, 1984). Nucleosides and nucleotides and some alkaloids bear a pyrimidine ring as a structural element and many functionalized derivatives obtained by synthetic procedures show antibiotic, antimicrobial (*e.g.* pyrimidine sulfonamides), sedative (*e.g.* barbiturates) and antitumor activity. Cheng & Roth (1971) have reviewed the chemotherapeutic action of 5-nitropyrimidines.

The direct introduction of substituents into the pyrimidine ring is not always easy; strategies for the construction of the ring from appropriate functionalized synthons are desired. β-Aminocrotonate–acyl isothiocyanate adducts cyclize readily by heating or base treatment to pyrimidine derivatives bearing COO*R* moieties at the 5-position of the pyrimidine ring (Goerdeler & Pohland, 1963; Goerdeler & Gnad, 1965; Goerdeler & Wieland, 1967). However, analogous attempts to cyclize nitroketenaminal–acyl isothiocyanate adducts (*N*-acyl-3,3-diamino-2-nitrothioacrylamides), (I), failed.

A synthetic route to obtain 4-thioxo-5-nitropyrimidines (II) from the former adducts based on the activation of the thiocarbonyl C atom toward the nucleophilic attack of the amino group in position 3 in (I) was designed (García Trimiño, Macías Cabrera & Vélez Castro, 1992). The *S*-methylation of (I) should enhance the electrophilic activity of the thiocarbonyl C atom leading to the 6-amino-4-methylthio-5-nitropyrimidinium salt (III). Nucleophilic attack by the soft iodide ion on the methyl group attached to the S atom or on the N atom of the pyrimidine ring should lead to compounds (II) and (IV), respectively.



^{13}C NMR and X-ray structural determination studies of the synthesized products showed that the derivatives (IV) were obtained and not the previously reported (II). In this paper we report the structure of 6-methylamino-4-methylthio-5-nitro-2-phenylpyrimidine [(IV*a*), $R = CH_3$, R^1 = Ph, $C_{12}H_{12}N_4O_2S$].

There are no unusual intermolecular or intramolecular distances or angles. There is an intramolecular hydrogen bond between the amine H atom and the nearest O atom of the nitro group, the H(1)···O(1) contact distance being 1.90 (3) Å. Both rings in the molecule are essentially planar. The pyrimidine ring and the phenyl substituent are coplanar, which indicates an extension of the delocalized π-bonding system across both rings. A least-squares-planes calculation involving the atoms of both rings plus the atoms S(1), N(2) and N(4) showed that the mean deviation from planarity was 0.092 Å. The dihedral angle between the pyrimidine and phenyl rings is 11.4 (3)°.

Fig. 1 (Granovsky, 1991) shows a view of a molecule with the atomic numbering.

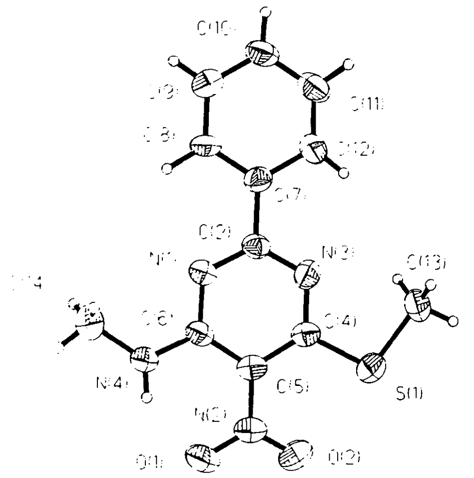


Fig. 1. View of the molecule of (IV*a*) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by spheres of arbitrary size.

Experimental

6-Methylamino-4-methylthio-5-nitro-2-phenylpyrimidine was obtained in 65% yield by methylation of 3,3-bis(methylamino)-*N*-benzoyl-2-nitrothioacrylamide with CH_3I in ethanol (García Trimiño, Macías Cabrera & Vélez Castro, 1992). Recrystallization from dimethylformamide (DMF) gave thin yellow plates (m.p. 488–489 K). The density D_m was measured by picnometry.

Crystal data

$C_{12}H_{12}N_4O_2S$
$M_r = 276.32$
Triclinic
$P\bar{1}$
$a = 5.335\ (2)$ Å
$b = 10.625\ (2)$ Å
$c = 11.886\ (2)$ Å
$\alpha = 109.36\ (2)°$
$\beta = 96.47\ (2)°$
$\gamma = 90.56\ (2)°$
$V = 630.81$ Å^3
$Z = 2$
$D_x = 1.45$ Mg m^{-3}
$D_m = 1.43$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 35 reflections
$\theta = 15–21°$
$\mu = 0.26$ mm^{-1}
$T = 298$ K
Thin rectangular plate
0.6 × 0.3 × 0.1 mm
Yellow

Data collection

Siemens *P*3/PC diffractometer
2*θ*/*θ* scans
Absorption correction: none
2429 measured reflections
2173 independent reflections
1072 observed reflections [$I > 3\sigma(I)$]

$R_{\text{int}} = 0.027$
$\theta_{\text{max}} = 25°$
$h = 0 \rightarrow 6$
$k = -11 \rightarrow 11$
$l = -14 \rightarrow 14$
3 standard reflections monitored every 100 reflections
intensity decay: <3%

Refinement

Refinement on *F*
$R = 0.061$
$wR = 0.068$
$S = 1.11$
1072 reflections
210 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F) + 0.001481F^2]$

$(\Delta/\sigma)_{\text{max}} = 0.069$
$\Delta\rho_{\text{max}} = 0.11$ e Å^{-3}
$\Delta\rho_{\text{min}} = -0.12$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{\text{eq}} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	*y*	*z*	U_{eq}
S(1)	0.3012 (4)	0.8984 (2)	0.5726 (2)	0.052 (1)
O(1)	0.8712 (10)	0.9989 (5)	0.8839 (5)	0.072 (2)
O(2)	0.7038 (9)	1.0157 (5)	0.7169 (4)	0.068 (2)
N(1)	0.3120 (10)	0.7212 (5)	0.8686 (5)	0.040 (2)
N(2)	0.7037 (11)	0.9672 (5)	0.7970 (5)	0.049 (3)
N(3)	0.1334 (10)	0.7424 (5)	0.6822 (4)	0.042 (2)
N(4)	0.6675 (10)	0.8399 (5)	0.9783 (5)	0.046 (2)
C(2)	0.1416 (11)	0.6904 (6)	0.7719 (6)	0.037 (3)
C(4)	0.3136 (12)	0.8340 (6)	0.6909 (6)	0.039 (3)
C(5)	0.5085 (12)	0.8732 (6)	0.7903 (6)	0.040 (3)
C(6)	0.4960 (11)	0.8126 (6)	0.8806 (6)	0.037 (3)
C(7)	−0.0534 (12)	0.5836 (6)	0.7564 (6)	0.041 (3)
C(8)	−0.0352 (13)	0.5062 (7)	0.8307 (6)	0.049 (3)
C(9)	−0.2156 (14)	0.4068 (8)	0.8157 (7)	0.059 (3)
C(10)	−0.4204 (14)	0.3835 (8)	0.7281 (6)	0.057 (3)
C(11)	−0.4390 (13)	0.4595 (8)	0.6545 (6)	0.053 (3)
C(12)	−0.2630 (13)	0.5587 (7)	0.6670 (6)	0.047 (3)
C(13)	0.0232 (14)	0.8108 (9)	0.4806 (7)	0.057 (3)
C(14)	0.6569 (16)	0.7707 (10)	1.0641 (8)	0.061 (4)

Table 2. *Selected geometric parameters* (Å, °)

S(1)—C(4)	1.751 (8)	S(1)—C(13)	1.788 (7)
O(1)—N(2)	1.238 (7)	O(2)—N(2)	1.224 (9)
N(1)—C(2)	1.328 (8)	N(1)—C(6)	1.336 (8)
N(2)—C(5)	1.412 (9)	N(3)—C(2)	1.35 (1)
N(3)—C(4)	1.332 (8)	N(4)—C(6)	1.342 (8)
N(4)—C(14)	1.45 (1)	C(2)—C(7)	1.483 (9)
C(4)—C(5)	1.428 (8)	C(5)—C(6)	1.43 (1)
C(7)—C(8)	1.39 (1)	C(7)—C(12)	1.410 (9)
C(8)—C(9)	1.37 (1)	C(9)—C(10)	1.38 (1)
C(10)—C(11)	1.37 (1)	C(11)—C(12)	1.36 (1)
C(4)—S(1)—C(13)	100.7 (4)	C(2)—N(1)—C(6)	118.1 (6)
O(1)—N(2)—O(2)	121.3 (6)	O(1)—N(2)—C(5)	119.4 (7)
O(2)—N(2)—C(5)	119.3 (5)	C(2)—N(3)—C(4)	117.1 (5)
C(6)—N(4)—C(14)	121.5 (6)	N(1)—C(2)—N(3)	126.6 (6)
N(1)—C(2)—C(7)	117.5 (6)	N(3)—C(2)—C(7)	115.8 (5)
S(1)—C(4)—N(3)	116.3 (4)	S(1)—C(4)—C(5)	122.7 (5)
N(3)—C(4)—C(5)	121.0 (6)	N(2)—C(5)—C(4)	120.7 (7)
N(2)—C(5)—C(6)	122.4 (5)	C(4)—C(5)—C(6)	116.9 (6)
N(1)—C(6)—N(4)	116.6 (7)	N(1)—C(6)—C(5)	120.2 (5)
N(4)—C(6)—C(5)	123.1 (6)	C(2)—C(7)—C(8)	120.6 (6)
C(2)—C(7)—C(12)	121.1 (7)	C(8)—C(7)—C(12)	118.3 (6)
C(7)—C(8)—C(9)	120.5 (6)	C(8)—C(9)—C(10)	120.8 (8)
C(9)—C(10)—C(11)	118.9 (7)	C(10)—C(11)—C(12)	121.7 (7)
C(7)—C(12)—C(11)	119.8 (8)		

Data collection: *XSCANS* (Siemens, 1992). Cell refinement: *SHELXTL-Plus* (Sheldrick, 1990). Data reduction: *SHELXTL-Plus*. Program(s) used to solve structure: *SHELXTL-Plus*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1125). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Brown, D. J. (1984). *Comprehensive Heterocyclic Chemistry*, Vol. 3, edited by A. R. Katritzky & Ch. W. Rees, pp. 142–155. Oxford: Pergamon Press.

Cheng, C. C. & Roth, B. (1971). *Prog. Med. Chem.* **8**, 98–100.

García Trimiño, M. I., Macías Cabrera, A. & Vélez Castro, H. (1992). *Synth. Commun.* **22**, 1319–1331.

Goerdeler, J. & Gnad, J. (1965). *Chem. Ber.* **98**, 1531–1543.

Goerdeler, J. & Pohland, H. W. (1963). *Chem. Ber.* **95**, 526–531.

Goerdeler, J. & Wieland, D. (1967). *Chem. Ber.* **100**, 47–59.

Granovsky, G. I. (1991). *Molgraph Interactive Molecular Graphics*. Neosoft-GG, USA.

Sheldrick, G. M. (1990). *SHELXTL-Plus*. PC Version. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Siemens (1992). *XSCANS*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Acta Cryst. (1995). C**51**, 1394–1397

3,3′-Bis(octyloxy)-2,2′-bithiophene at 195 K

MICHEL PELLETIER, FRANÇOIS BRISSE, RENÉE CLOUTIER AND MARIO LECLERC

Département de chimie, Université de Montréal CP 6128, Succursale Centre-ville, Montréal H3C 3J7, Canada

(*Received* 9 *May* 1994; *accepted* 24 *January* 1995)

Abstract

In the solid state, the title molecule, $C_{24}H_{38}O_2S_2$, possesses a crystallographic centre of symmetry. The bithiophene ring system is planar and the central C2—C2′ bond length is 1.445 (2) Å. The O atom is within the thiophene mean plane and the torsion angles in the octyloxy chains are all *trans*, except for the angle around the first C—C bond which is *gauche*. The octyloxy chains are all parallel to one another and are aligned along the bisector of the *ac* face of the unit cell.